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Decomposition of Hydrogen Peroxide using Chemical and Catalytic Methods: A Reactor-based Approach

MD SHAHADAT HUSSAIN CHOWDHURY[1], MOHEBUL AHSAN[1], M. HUMAYUN KABIR[1,2],
SYED S. ALAM[1] and MOHAMMAD A. HASNAT[1,3,*]

[1]Electrochemistry & Catalysis Research Laboratory (ECRL), Department of Chemistry, School of Physical Sciences, Shahjalal University of Science and Technology, Sylhet-3100, Bangladesh
[2]Department of Chemistry, Noorjahan Memorial Women's Degree College, Bangladesh
[3]Bangladesh Academy of Sciences, Agargaon, Dhaka-1207, Bangladesh

*Corresponding author: E-mail: mah-che@sust.edu

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A feasible catalytic reactor for oxygen evolution has been proposed, which could complement the current methods of O_2 evolution efficiently and ensure the availability of pure oxygen on a wider scale. Several chemicals, such as NaI, MnO_2, $NiSO_4$, *etc.* can generally decompose hydrogen peroxide by producing oxygen as a product *via* chemical reactions. Meanwhile, the Pt-Pd catalyst efficiently catalyzes hydrogen peroxide decomposition reactions and generates pure oxygen without any unfriendly species. The excellence of the bi-metallic catalyst has been validated by the evaluation of turnover number (TON) and turnover frequency (TOF). Implementing the knowledge of chemical reactions and heterogenous catalysis, the proposed catalytic reactor could be helpful for generating a continuous flow of oxygen. Surely, the oxygen produced in this process is higher in amount compared to the oxygen present in the atmosphere at ambient conditions which might facilitate the breathing support for critically ill patients.

Keywords: Hydrogen peroxide, Oxygen evolution, Decomposition, turnover frequency, turnover number, Reactor.

INTRODUCTION

The supply of medical oxygen for the critically ill COVID-19 patients has become one of the major challenges for Bangladesh as well as other developing countries around the world [1]. Typically, industrial methods for the production of oxygen include purification of atmospheric air, electrolysis of water, artificial photosynthesis, cryogenic distillation, *etc.* [2-5]. These methods are not only complex but also considered time consuming in the requirement of pure oxygen supply. In this case, transportation, refill time and logistic support regarding the oxygen cylinder remain addressable challenges. Several barriers have to tackle from the threshold of production and delivery to hospitals or individual patients in the case of oxygen supply. The WHO suggests that around 20% of patients may need medical oxygen support to recover from the acute respiratory problems [6]. In such a case, managing this amount seems a herculean task, keeping in mind the manpower along with administrative support to make oxygen supply widely available. On top of that, the price of the oxygen cylinders has been sky rocketing due to the supply and demand, which is almost unbearable for most of the people in Bangladesh who are living below the poverty line [7,8]. Moreover, sometimes patients in remote villages may need urgent external oxygen aid. According to recent reports [9-12], due to the scarcity of commercial cylindrical oxygen, the lives of patients are often jeopardized. In these circumstances, an alternative way of generating pure oxygen *via* a chemical or catalytic process may be able to support the demand at a reasonable cost.

Hydrogen peroxide can be easily decomposed to water generating oxygen under suitable environment [13]. Since hydrogen peroxide is a benign, eco-friendly and easily portable chemical reagent used for synthesis purposes, cleansing of equipment, aerospace oxidizers, medical and cosmetic applications, wastewater treatment plants and most notably known as bleaching and Fenton reagents [14], this chemical can also be a complimentary source of oxygen for urgent usage despite having some explosive nature regarding synthesis and handling [15,16].

Concerning the hydrogen peroxide decomposition reaction, several studies have been conducted using different materials [17-20]. These are often expensive and require complex procedures for the synthesis purpose. However, in this study, the applicability of several ubiquitously available materials has been checked for decomposing hydrogen peroxide. It is noticed that sodium iodide (under basic conditions), several oxides and sulfates are efficient in decomposing hydrogen peroxide. Meanwhile, a bimetallic Pt-Pd catalyst was synthesized [21], which exhibited the utmost decomposition efficiency with reasonable durability for extensive use. Taking this into consideration, we have aimed at the design of a cost-effective, efficient and easily operable reactor where a common chemical reagent, hydrogen peroxide could be decomposed to produce oxygen *via* a chemical or catalytic process. Additionally, chemical or catalytic activity has been determined for batchwise reactors that can be extended to continuous flow reactors on a wider scale. It is believed that people having a minimum level of technical knowledge can fabricate the reactors for oxygen production in times of need.

EXPERIMENTAL

Materials used for this experimental purpose were of analytical grade, so no extra purification steps were performed. A PET (polyethylene terapathalate) container of 1.0 L, an inlet pipe, an outlet pipe, a flow controller was used to fabricate the batch reactor. For the catalyst preparation, palladium(II) chloride ($PdCl_2$), sodium borohydride ($NaBH_4$), chloroplatinic acid (H_2PtCl_6) and Nafion-117 membrane were supplied by Wako Chemicals (USA). Hydrogen peroxide, manganese dioxide and sodium iodide were supplied by Merck, Germany. Millipore Milli-Q water (resistivity more than 18 MΩ cm and micro-organic concentration ≥ 3 ppb) was used to make all the required solutions.

Chemical method: In order to check the oxygen evolution reaction, 250 mL of 15% hydrogen peroxide was taken in a glass bottle and then the outlet port was connected to a reservoir using a normal saline IV set pipe. Approximately 0.5 g of MnO_2 or NaI was added to the glass bottle just before the reaction was started. The pH of NaI solution was maintained at a slightly basic level between 7.0 and 9.0. In other cases, the pH was neutral. As soon as MnO_2 or NaI was added to hydrogen peroxide, the spontaneous chemical reaction generates oxygen as a product. The evolved oxygen was collected in the reservoir for the analysis.

Catalytic method: The catalytic process is considered one of the most reliable, efficient and long-serving processes that is essentially non-destructive. This process ensures the selective production of molecular oxygen along with high purity and incessant supply if the catalyst is not destroyed physically or contaminated in any manner [22-26]. To decompose H_2O_2 catalytically, a composite of Pt-Pd film was fabricated by a piece of Nafion-117 membrane (size: 6 cm^2), which was first sand blasted to make the surface notched [21,27]. Afterwards, this blasted polymer was washed properly and dried at 80 ºC for 1 h. Meanwhile, 0.1 g of a mixture of H_2PtCl_6 and $PdCl_2$ (1:1) was added to 200 mL of water, which was vigorously stirred. To ensure the complete dissolution, several drops of concentrated ammonia were added. Later, the dried polymer was placed in the reaction mixture by glass rods such that none of the surfaces of the polymer adhered to the beaker. After that, a mixture of 3 M NaOH and 2 M $NaBH_4$ solution was added dropwise to the beaker at a rate of 2 mL h^{-1}. At the same time, the mixture was stirred at a rate of 200 rpm with a magnetic bar and the temperature was raised gradually from 30-65 ºC at a rate of 5 ºC h^{-1}. In this way, within 12 h, a film of mixed Pt and Pd metals was fabricated on both surfaces of the Nafion polymer. The thickness of the metallic film on either side was approximately 2.0 µm. Later, the prepared metal-polymer assembly was washed with dil. HCl to remove unreacted NaOH and subsequently sonicated. The assembly was next washed with an ample of distilled water and dried at 110 ºC prior to applied for catalytic reaction. The catalyst, once ready, was placed in a 1.0 L glass bottle containing 250 mL of 15% H_2O_2. As soon as the catalyst comes into contact with H_2O_2, the decomposition reaction was initiated and the evolution of oxygen commences. The evolved oxygen was collected at normal temperature and pressure for the analysis as mentioned in the previous section. From the thorough investigations, it was ascertained that no hazardous substances were produced throughout this process, as this is a simple catalytic decomposition where the product is only oxygen and water. So, the catalytic method mentioned here can be termed a "complete green process".

RESULTS AND DISCUSSION

Hydrogen peroxide decomposition: Fig. 1 represents the time dependent volume of the oxygen evolution generated due to the chemical method and the catalytic decomposition of the hydrogen peroxide at room temperature and atmospheric pressure.

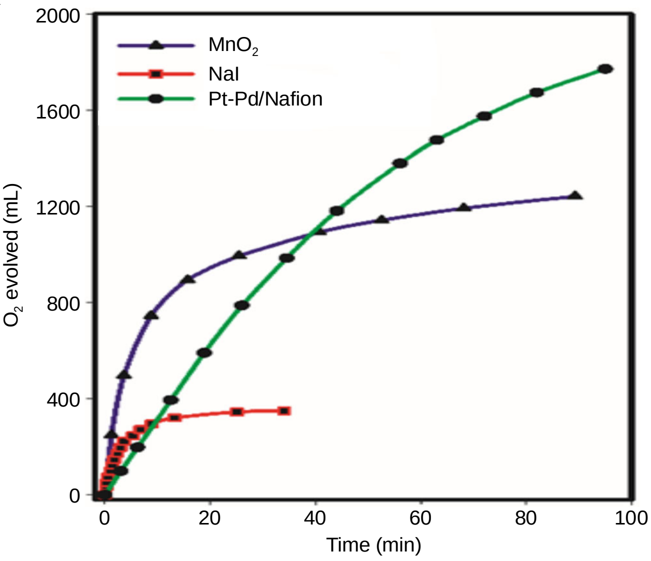


Fig. 1. Volume of the O_2 evolution by chemical and catalytic methods at standard temperature and pressure

Chemical decomposition: It is apparent from Fig. 1 that oxygen evolution is very rapid while NaI is being used as a decomposing agent for hydrogen peroxide. However, within 20 min, the oxygen evolution becomes limited. This is because the yield of a reaction is proportional to the reagent introduced to the reaction vessel. As soon as iodide ions come into contact with hydrogen peroxide, the conversion of H_2O_2 to O_2 is achieved in multi-steps as reported earlier [28-30]. Since in an acidic medium, iodine is the major product, a basic or neutral medium is highly recommended for the decomposition. Afterwards, by varying the concentration of NaI from (0-8.0) mM, the decomposition of H_2O_2 was examined. It was found that with the increment in the concentration of NaI, the decomposition rate was increased up to 3.7 mM of NaI (Fig. 2a). Understandably, exceeding the concentration level, the decomposition rate was declined in a notable manner.

Since the rate of oxygen evolution was found to rely on the concentration of hydrogen peroxide [21] hence, a first order kinetic pathway was involved during the decomposition reaction as per the following equation:

$$\nu_{O_2} = k[H_2O_2] \quad (1)$$

Thus, as long as iodide and hydrogen peroxide remain in the reactor, oxygen evolution continues. Herein, iodide was used as a limiting reagent, so within 20 min, all of the iodide ions were perhaps consumed and the extent of oxygen evolution became limited. By contrast, it is seen from Fig. 1 that MnO_2 efficiently catalyzed the decomposition reaction in optimized conditions where O_2 evolution is five times greater than NaI. But after a passage of time, it reached a plateau with the maximum evolution of oxygen. The reaction pathway of the chemical decomposition has already been reported in the literature [31,32]. To a certain extent, MnO_2 catalyzed the decomposition reaction, so it was necessary to optimize the reaction conditions. *Ipso facto*, the influence of MnO_2 dosage on hydrogen peroxide was also checked and optimized. By optimizing the concentration as shown in Fig. 2b, it is clear that a suitable catalytic dosage of (0-2.3) mM was maintained to obtain the best decomposition rate of H_2O_2. However, above this dosage, the rate of reaction reaches a maximum peak.

Catalytic decomposition: It is widely acknowledged that noble metals possess unique catalytic properties regarding hydrogen peroxide decomposition reactions [33-39]. In this connection, a bimetallic Pt-Pd/Nafion catalyst was fabricated [21] to decompose hydrogen peroxide in an effective manner. Fig. 1 reveals that while the Pt-Pd/Nafion catalyst was employed, oxygen evolution increased steadily and showed a maximum magnitude. This observation suggests that the Pt-Pd film efficiently catalyzed the hydrogen peroxide decomposition without acting as a limiting reagent. The reaction was repeated several times and similar observations were noticed. However, the Pt-Pd film was not involved in any chemical reaction and the catalytic surface was not passivated, which was assured in this observation. The mechanism of the catalytic decomposition of the hydrogen peroxide proposed in the literature as follows [40,41]:

$$\text{Cat} + H_2O_2 \longrightarrow \text{Cat-O} + H_2O \quad (2)$$

$$\text{Cat-O} + H_2O_2 \longrightarrow \text{Cat} + O_2 + H_2O \quad (3)$$

Since the reaction is dependent on the concentration of the hydrogen peroxide [41], hence the rate of oxygen evolution is best explained by the first order kinetics which is analogous to the eqn. 3. However, if a Pt or Pd catalyst is used alone, the reaction rate is not so strong to be used for applied purposes. While Pt and Pd nanoparticles are aggregated together, these metallic sites become negatively and positively charged in a partial manner, respectively. It is noted that the stability of the hydrogen peroxide deteriorates in contact with the negatively charged sites, which favours the rapid decomposition of the hydrogen peroxide [42,43]. Substantially, it can be stated that while a catalytic surface contains a considerable amount of Pt and Pd particles, the rate of hydrogen peroxide decomposition revs up and this type of catalyst may be conveniently helpful for the practical applications of oxygen production on a wider scale.

Chemical and catalytic activities: At this stage, it is established that catalytic decomposition has supremacy in stability and efficacy concerns over chemical methods comparatively. As the catalyst will not evolve O_2 for an unlimited amount of time, it is imperative to calculate the amount of O_2 produced

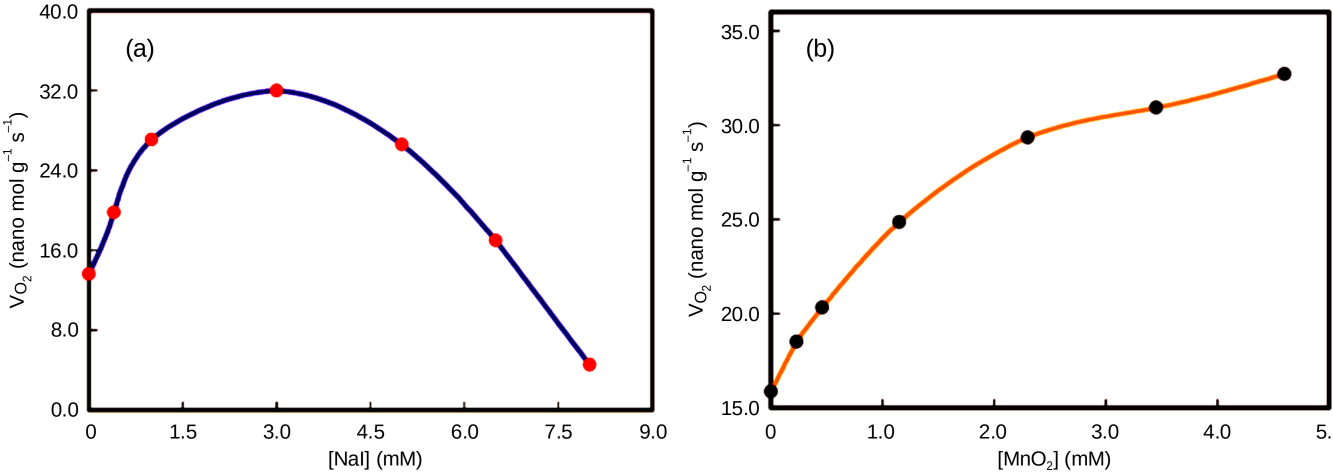


Fig. 2. Effect of (a) NaI and (b) MnO_2 concentration on H_2O_2 decomposition. {$[H_2O_2]$ = 1.0 M; volume of the reactor: 250 mL}

against the amount of catalyst or chemical introduced. At this succession, the total number of turnovers the catalyst can achieve until it's total decay regardless of time was calculated. Basically, the maximum usage of the catalyst under experimental conditions is determined by the number of reaction cycles, termed as the turnover number (TON) [44,45]. For further validation of the excellence of the bimetallic catalyst, TON was calculated as per eqn. 4:

$$\text{TON} = \frac{\text{Amount of } O_2 \text{ produced}}{\text{Amount of chemical or catalyst introduced}} \quad (4)$$

On further note, to assume the instantaneous efficiency of the catalyst, turn over frequency (TOF) [44,45] was calculated (Table-1) using TON against the time of chemical or catalytic decomposition of H_2O_2 reaction following eqn. 5:

$$\text{TOF} = \frac{\text{TON}}{\text{Time of the decomposition}} \quad (5)$$

It is obvious from the comparative tabulated data in Table-1 that the Pt-Pd/Nafion catalyst has 40 times more catalytic cycle involvement than the MnO_2 and almost 176 times more catalytic cycle involvement than NaI. From the above comparison, it can be stated that the bimetallic catalyst has the highest number of catalytic cycles involved during the decomposition of the H_2O_2 pertaining to O_2 evolution, which clearly confirms its superiority.

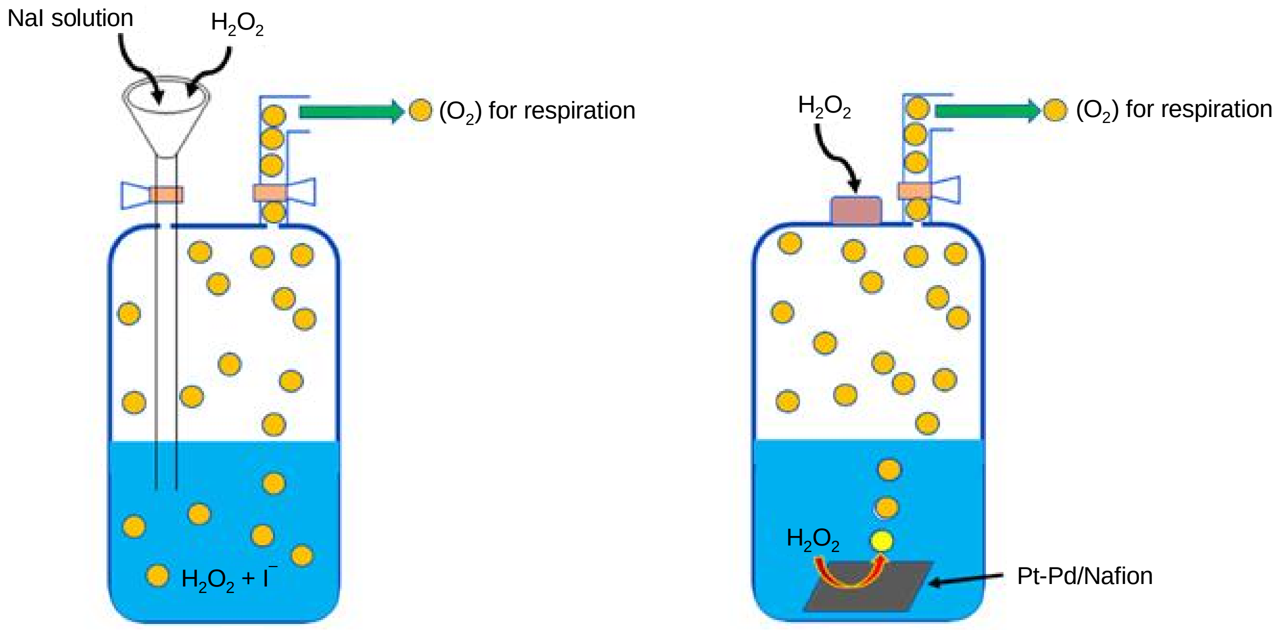
TABLE-1
EVALUATION OF THE TURNOVER NUMBER AND TURNOVER FREQUENCY OF THE H_2O_2 DECOMPOSITION USING CHEMICAL AND BI-METALLIC CATALYST

Chemical/catalyst	O_2 evolved (mL)	TON	TOF (min^{-1})
NaI	365	2684	72.54
MnO_2	1180	11920	132.54
Pt-Pd/Nafion	1750	472973	4978.66

Note: NaI and MnO_2 were taken 0.5 g, each in 250 mL of H_2O_2 solution. The time needed for the completion of the decomposition for NaI, MnO_2 and Pt-Pd/Nafion was 37, 90 and 95 min, respectively. The total catalytic (Pt-Pd/Nafion) weight was 2.1 mg, having 76 % of Pt and 24% of Pt with a geometric surface area of 12 cm^2. The time needed for the complete decomposition of the chemical was obtained from Fig. 1.

Design of flow reactor: Based on the above discussion, it is highly likely possible that flow type reactors can be fabricated such that continuous O_2 supply can be ensured for the breathing support of critically-ill patients. In this connection, the simple designs of flow type reactors are illustrated in Fig. 3. For generating O_2 *via* chemical reactions, a 1.0 L PET reaction vessel having an inlet funnel, outlet pipe and flow controller was used. Here, it should be ensured that the end of the inlet funnel sufficiently remains in contact with hydrogen peroxide in the vessel such that no gas can escape through the inlet channel. It is observed that the container should have a minimum space to accumulate oxygen gas as evolved by the decomposition reaction where the gas pressure can be regulated using a flow controller. Therefore, since alkaline NaI instantly and rapidly decomposes hydrogen peroxide, hydrogen peroxide and alkaline NaI should be inserted into the vessel gradually through the funnel to ensure the continuous oxygen flow. For alkalinity, a few drops of NaOH could be poured into the vessel before adding NaI and H_2O_2. The reaction rate is unimolecular, so the time of flow of oxygen could be regulated by controlling the concentration of H_2O_2.

Meanwhile, in case of the catalytic decomposition of H_2O_2, a similar reactor vessel can be used. In this case, catalyst (Pt-Pd/Nafion) needs to be placed inside the reactor first, then H_2O_2 is required to pour on the reactor gradually using the funnel. Later, the oxygen flow could be controlled by the size of the catalytic surface and the concentration of H_2O_2. This idea easily aligns with the fabrication of a batch reactor. Here, the concentrated H_2O_2 and catalyst are required to be placed in the vessel together, where oxygen is expected to be generated



(a) (b)

Fig. 3. Flow diagram of the evolution of O_2 from H_2O_2 in the presence of (a) NaI and (b) Pt-Pd surface in the batch type reactor

as long as H_2O_2 remains unreacted. In this case, the reactor should have some space above the solution level such that evolved gas could be accumulated prior to passing through the outlet channel. To get the maximum output pertaining to O_2 evolution, the P_{O_2} should be properly maintained such that it exceeds the atmospheric pressure and the P_{H_2O} remains below the both pressures. So, it is recommended to use a vacuum system to sort out the hassle. However, the above-mentioned mechanical designs were only attributed to check the efficiency of oxygen generation in a laboratory environment using chemicals and catalysts. Further industrial production or practical multiplication and the maintenance of such reactors require sufficient knowledge of chemical engineering and other mechanical insights.

Conclusion

Although hydrogen peroxide may be converted to different products, iodide ions can selectively decompose hydrogen peroxide to produce oxygen. Most importantly, this reaction must be carried out in a neutral or basic medium, otherwise molecular iodine will be generated. The reaction can alternatively be carried out using materials like K_2SO_4, MnO_2, $NiSO_4$, $CoSO_4{\cdot}7H_2O$ and $CuSO_4$. By contrast, the Pt-Pd bimetallic catalyst deposited on a Nafion surface catalyzes hydrogen peroxide decomposition in more efficient manner, which was further justified by calculating TON and TOF. Moreover, such a reactor based on the catalytic process converts hydrogen peroxide to pure oxygen through a decomposition process, which is seemingly more convenient and cost-effective than contemporary methods as it has the perks of repeatable usage.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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