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Research Article

Experimental Validation of Borohydride Electro-oxidation in Membraneless Borohydride Fuel Cell

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Abstract

A membraneless sodium borohydride fuel cell has been fabricated using sodium borohydride as the fuel and sodium percarbonate as the oxidant in an alkaline and acid media configuration. Different operating conditions and different cell dimensions were used to assess the performance of this fuel cell. At room temperature, the laminar flow-based membraneless fuel cell was found to reach a maximum power density of 23.61 mW cm^{-2} using 0.15 M sodium borate in 3 M sodium hydroxide solution as the fuel and 0.15 M percarbonate in 1.5 M sulphuric acid solution as the oxidant.

Keywords: Sodium borohydride; Power density; Membraneless borohydride fuel cell; Portable power applications.

Introduction

Demands for new sources of portable power have given rise to current work on miniaturized fuel cell technology. A hopeful direction is the development of fuel cells that use laminar flow to separate the fuel and oxidant streams. These eliminate the need for a membrane because the anode and cathode reactions are separated by laminar flow region [1-4]. A membraneless fuel cell with a planar structure has a high design flexibility, which enables its easy integration into definite microfluidic systems and portable electronic devices.

Recently, Sodium borohydride ($NaBH_4$) is gaining increasing attention of researchers in energy systems, due to their following advantages: it is nontoxic, easily stored, large amounts of hydrogen (10.6 wt. %) and specific energy density. Besides, its oxidation product, metaborate, can be recycled to produce borohydride [5-7]. In addition, it has a higher volumetric hydrogen capacity than compressed and liquefied hydrogen and higher gravimetric hydrogen capacity compared to metal hydrides.

The membraneless borohydride fuel cell (MLBFC) developed in our study employs an alkaline solution of sodium borohydride as a fuel and an acidic solution of sodium percarbonate ($2Na_2CO_3.3H_2O_2$) as an oxidant. A number of drawbacks associated with membrane cells that were discussed earlier can be avoided in the MLBFCs. Assessing the performance of the fuel cell in alkaline and acid media (one electrode is acidic and the other one is alkaline) is the focus of this study.

Sodium percarbonate is nontoxic, cost-effective, environmentally friendly, large-scale industrial chemical used primarily in detergents and as a mild oxidant. It is a true peroxo salt and is a readily available source of hydrogen peroxide [8,9]

$$2Na_2CO_3.3H_2O_2 \rightarrow 2Na_2CO_3 + 3H_2O_2 \quad (1)$$

A unique feature of sodium percarbonate is that it can be used not only as an oxidant but also as a reductant [10,11], which is an advantage compared to fuel cells that use H_2O_2. On the performance side, the MLBFC generates electric power comparable to a typical air-breathing direct methanol fuel cell (DMFC) operating in a microchemical channel at room temperature. These advantages make MLBFCs a suitable alternative for portable power applications. We have developed a new simplified architecture, which is unique from those that have been reported in the literature, by eliminating and

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integrating the key components of a conventional MEA in this study.

The objective of this study is to develop a membraneless microfluidic fuel cell operating in alkaline and acid media. Experimental methods are applied to a planar microfluidic fuel cell design employing platinum-coated carbon electrode as the anode as well as the cathode. The sodium percarbonate fuel cell in our study is tested in all-acidic, all-alkaline, and alkaline-acid media to ascertain the best media configuration for the further fuel cell experiments. We also investigate the effect of variation of fuel and oxidant concentrations, flow rate variations, electrolyte variations, and electrode distance effect to improve performance of the fuel cell.

Experimental

Chemical and materials

The chemicals used in the planar microfuel cell design were $NaBH_4$ (98%, Merck), NaOH (98%, Merck), $2Na_2CO_3.3H_2O_2$ (99%, Riedel), and H_2SO_4 (98%, Merck). All the solutions were prepared in deionized water. The materials used in fuel cells were poly(dimethylsiloxane) (PDMS; 99.9%, Chemsworth), and poly(methylmethacrylate) (PMMA; 92%, G. Khanna & Co), graphite plates (Kriti Graphite), and silicon tubes (Shree Gaurav Rubber Products).

Catalyst deposition

In the MLBFCs, graphite plates served as current collectors and catalyst structures.
For all the experiments of fuel cells, the sides of the graphite plates are brushed by unsupported platinum black nanoparticles so that they act as the cathode and as the anode that line the microfluidic channel. For both electrodes, the catalyst suspensions were prepared by mixing Pt black nanoparticles (Alpha Aesar) at a concentration of 6.0 mg ml^{-1} in a 10 wt.% Nafion solution (Nafion stock solution: Dupont, 5% (w/w) solution). This mixture was sonicated and hand-painted on to the side faces of the graphite plates at a loading of 2 mg cm^{-2}. Then solvent was evaporated by the use of a heat lamp for uniform loading [10].

Fabrication of fuel cell

The E-channel structure required for co-laminar flow was moulded with poly(dimethylsiloxane) (PDMS), typically 1–10 mm in thickness, after coating it with 1-mm thick graphite plate. To provide rigidity and robustness to the layered system, more rigid top and bottom capping layers were formed, such as 2 mm poly(methylmethacrylate) (PMMA). To guide the fuel and the oxidant into the E-shaped channel systems and to let the waste stream out of the channel, fluidic tubing (silicon tubes, Shree Gaurav Rubber Products) was attached to the slabs of the material. Typically holes are punched exactly at the three ends of the E-shaped channel design. Silicon tubing was glued to the inlets and outlet by epoxy (Figure 1).

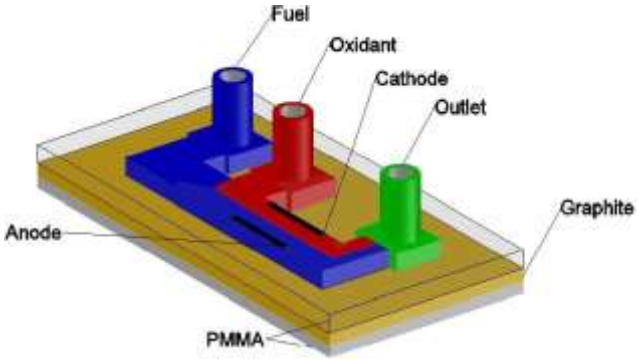

Figure 1. Schematic of the E-shaped membraneless laminar flow-based fuel cell with graphite plates molded with poly(dimethylsiloxane) and sealed with poly(methyl methacrylte)

Testing of the fuel cell

The assembled fuel cells were tested in a configuration of alkaline and acid media (acidic oxidant and alkaline fuel). When the reactants were injected through the inlets, the fuel and the oxidant solutions merge at the E-junction and continue to flow in a laminar fashion in parallel over the anode and the cathode where both the fuel and the oxidant, respectively, are oxidized and reduced. Polarization curves were obtained at different cell potentials using CS310 computer-controlled potentiostat (Zhengzhou Triangle Instrument Co. Ltd.) with the associated Thales Z software package.

Potentiostat leads were attached to the anodic and cathodic graphite current collectors using copper alligator clips. The lead of the working electrode was attached to the anode while the reference and counter electrode leads were combined and attached to the cathode. The potentiostat was used to produce an applied potential and a multimeter (Fluke), with its leads attached to the anodic and cathode graphite

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current collectors, was used to determine the actual cell potential.

The fuel and oxidant stream flow rates were varied between 0.1 and 1.0 ml min^{-1} (per stream) using a syringe pump. Upon exiting each fuel cell, the streams travel through the silicon tubing and get collected in a beaker. All the experiments were performed at room temperature.

Results and discussions

Scheme of the various cell reactions in MLBFC

In this study, we treated the MLBFC in all-acidic media, all-alkaline media, alkaline–acid media 1 (alkaline cathode and acidic anode), as well as alkaline–acid media 2 (alkaline anode and acidic cathode). Their cell reactions and theoretical potential are illustrated in the following. For MLBFCs, the anode stream had 0.15 M sodium borohydride ($NaBH_4$) in 3.0 M sodium hydroxide (NaOH) and the cathode stream had 0.15 M percarbonate in 1.5 M H_2SO_4.

Performance of MLBFC in all-acidic media

Equations (2) and (3) show the half-cell reactions and standard electrode potentials of sodium borohydride oxidation and percarbonate reduction in acidic media. Eq. (4) represents the overall cell reaction [12]. The acidic–acidic (both anode and cathode are acidic media) configuration provides a maximum theoretical open circuit potential (OCP) of 2.251 V. But we achieved the maximum potential 0.69 V at a current density of 14.01 mA cm^{-2} using our membraneless sodium percarbonate fuel cell:

Anode: $NaBH_4 + 2H_2O \rightarrow NaBO_2 + 8H^+ + 8e^-$

$E^\circ = -0.471$ V (2)

Cathode: $4H_2O_2 + 8H^+ + 8e^- \rightarrow 8H_2O$

$E^\circ = 1.78$ V (3)

Overall: $NaBH_4 + 4H_2O_2 \rightarrow NaBO_2 + 6H_2O$

$\Delta E = 2.251$ V (4)

Performance of MLBFC in all-alkaline media

The redox reactions and standard electrode potentials of BH_4^- oxidation and percarbonate reduction in the alkaline media are shown in Eqs. (5) and (6). Eq. (7) represents the net cell reaction [13]. The all-alkaline configuration gives a maximum theoretical OCP of 2.226 V.

Anode: $NaBH_4 + 8OH^- \rightarrow NaBO_2 + 6H_2O + 8e^-$

$E^\circ = -1.24$ V (5)

Cathode: $4H_2O_2 + 8e^- \rightarrow 8OH^-$

$E^\circ = 0.986$ V (6)

Overall: $NaBH_4 + 4H_2O_2 \rightarrow NaBO_2 + 6H_2O$

$\Delta E = 2.226$ V (7)

The mass transport limitations region, however, is reached at 1.09 V when 25.03 mA cm^{-2} of current density is achieved in the MLBFC operating in an all-alkaline media.

Performance of MLBFC in alkaline–acid media 1 (alkaline cathode, acidic anode)

In alkaline–acid media system 1, one stream is acidic while the other stream is alkaline. In this configuration, the overall cell reaction, Eq. (8), can be obtained from Eqs. (2) and (6). The maximum theoretical OCP obtained is 1.457 V in alkaline cathode and acidic anode configuration. An OCP of 0.417 V is observed as a result of over potentials on the cathode and the anode [6]. This MLBFC run in the acidic anode and alkaline cathode configuration is still limited by borohydride hydrolysis in the presence of an acid, which causes a drop in the anode potential [14,15].

Anode: $NaBH_4 + 2H_2O \rightarrow NaBO_2 + 8H^+ + 8e^-$

$E^\circ = -0.471$ V (2)

Cathode: $4H_2O_2 + 8e^- \rightarrow 8OH^-$

$E^\circ = 0.986$ V (6)

Overall: $NaBH_4 + 4H_2O_2 + 2H_2O \rightarrow NaBO_2 + 8H^+ + 8OH^-$ $\Delta E = 1.457$ V (8)

Performance of MLBFC in alkaline–acid media 2 (acidic cathode, alkaline anode)

In contrast, in alkaline anode and acidic cathode configuration, use of an alkaline fuel stream (Eq. (5)) and an acidic oxidant stream (Eq. (3)) allows energy to be obtained both from the borohydride oxidation and percarbonate reduction reactions, as evident from the overall cell reaction (Eq. (9)). The coupling of two galvanic reactions in this configuration yields a desirable high theoretical OCP of 3.02 V compared to H_2/O_2 and CH_3OH/O_2 fuel cells that provide equilibrium voltages of 1.24 and 1.21 V, respectively [16].

Anode: $NaBH_4 + 8OH^- \rightarrow NaBO_2 + 6H_2O + 8e^-$

$E^\circ = -1.24$ V (5)

Cathode: $4H_2O_2 + 8H^+ + 8e^- \rightarrow 8H_2O$

$E^\circ = 1.78$ V (3)

Overall: $NaBH_4 + 4H_2O_2 + 8H^+ + 8OH^- \rightarrow NaBO_2 + 14H_2O$ $\Delta E = 3.02$ V (9)

The use of alkaline anode and acidic cathode (alkaline–acid media 2) resulted in a higher overall cell potential than those obtained for the all-alkaline, all acidic, alkaline–acid media 1 experiments in MLBFC. For example,

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the acid–alkaline media 2 yields a potential of 1.89 V, whereas both the all-acidic, alkaline–acid media 1 cells already start from a lower OCP and thus have a current density that are significantly lower than those of the alkaline–acid media 2.

At first look, the higher power densities of the alkaline and acid media configuration 2 fuel cells may look very promising. Due to the higher power densities and higher cell potential, the MLBFC in alkaline–acid configuration 2 is subjected to further tests, such as ascertaining the effect of varying fuel and oxidant concentrations, flow rate variations, electrode distance effect, and cell stability test.

Influence of fuel concentration

On increasing the concentration of $NaBH_4$, fuel diffusion and the oxidation kinetics are observed to be improved, which leads to higher power density. However, borohydride hydrolysis also increases, resulting in a decrease of open circuit voltage in turn. Therefore the concentration of $NaBH_4$ has to be optimized for better cell performance. The effect of sodium borohydride concentration on the performance of MLBFC is studied by varying the $NaBH_4$ concentration between 0.05 and 0.3 M in 3 M NaOH.

The cell polarization and power density curves are shown in Figure. 2. As the $NaBH_4$ concentration changes, the cell OCP remains practically in the range 1.38–1.89 V and the current density varies between 67.35 and 56.55 mA cm^{-2} for 0.15 M $NaBH_4$. From the cost perspective, the$NaBH_4$ concentration of 0.15 M seems to be the best composition for the fuel.

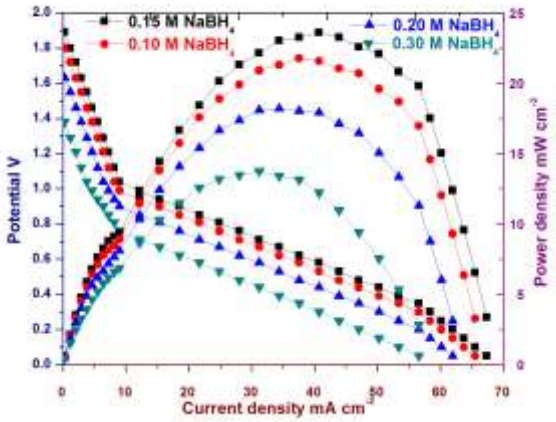

Figure 2. Curves of cell polarization and power density for MLBFC at different fuel concentrations: Fuel: X M in 3 M NaOH. Oxidant: 0.15 M percarbonate in 1.5 M H_2SO_4 solution. Flow rate of the reactants: 0.3ml min^{-1}

Effect of oxidant composition

With liquid cathode reactant, increasing percarbonate concentration leads to an increase in potential according to the electrode reaction. Figure 3 shows the influence of percarbonate concentration on the performance of MLBFC. The cell performance improves on increasing percarbonate concentration from 0.050 to 0.15 M in 1.5 M H_2SO_4. However, further increase in percarbonate concentration leads to no change in cell performance. The peak power density is as high as 23.61 mW cm^{-2} at 0.58 V and 40.71 mA cm^{-2} for 0.15 M percarbonate, which is comparable to the air-breathing direct methanol fuel cell and much higher compared to membraneless microfuel cells of other designs [17-19].

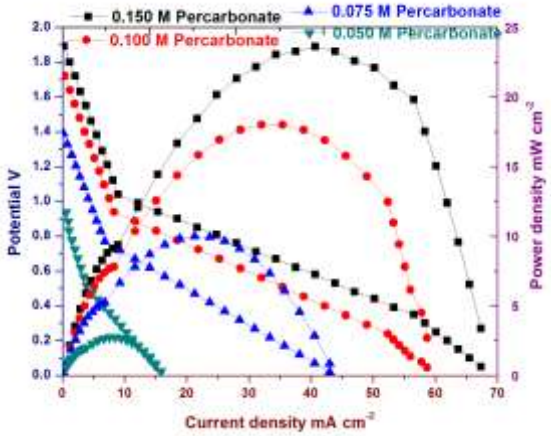

Figure 3. Curves of cell polarization and power density of MLBFC at different oxidant concentrations. Oxidant: X M percarbonate in 1.5 M H2SO4. Fuel: 0.15 M NaBH4 in 3 M NaOH solutions. Flow rate of the reactants: 0.3ml min−1

Variation of electrolyte concentrations

The performance of the oxidant is dependent on the electrolyte concentration. Figure 4 show that the power density of the fuel cell increases when the H_2SO_4 concentration increases from 0.10 to 1.5 M. When sulphuric acid concentration is increased further, the cell performance decreases. Therefore, 1.5 M sulphuric acid is fixed as the ideal oxidant solution to be used as the electrolyte.

The concentration of alkaline anolyte also affects the fuel cell performance. Increasing NaOH concentration up to 3 M has been found to be beneficial for MLBFC performance in

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terms of enhanced OCP, power density, and sustained current density. Beyond the NaOH concentration of 3 M, a negative effect is observed. Increasing NaOH concentration manifests in negative effects of improving anode reaction, increasing the conductivity of the sodium hydroxide solution, and reducing possible borohydride hydrolysis [20,21]. Yet, increased NaOH concentration provides a negative effect to the cathode reaction and also leads to an increase in the solution viscosity, which decreases the movement of borohydride ions and sodium ions in the catalyst layer [22]. In addition, at a higher concentration, sodium borohydride is converted to sodium carbonate in the presence of atmospheric CO_2. Therefore, we optimized the sodium hydroxide concentration for better cell performance.

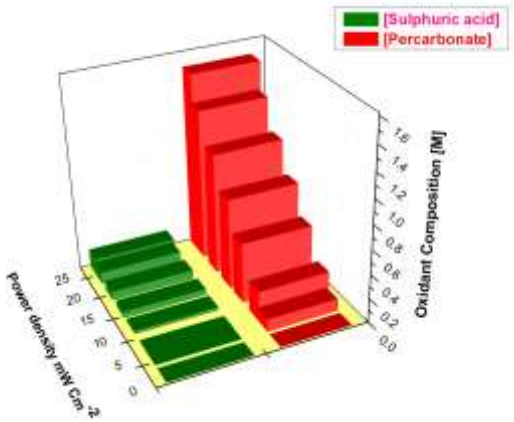

Figure 4. Effect of various combinations of percarbonate and sulphuric acid concentrations on the maximum power density (23.61 mW cm^{-2}) of the MLBFC at room temperature. The fuel mixture for variation of oxidant is ([fuel]: 0.15 M sodium borohydride + 3 M NaOH, [oxidant]: X M percarbonate + 1.5 M H_2SO_4) and the fuel mixture for variation of sulphuric acid is ([fuel]: 0.15 M sodium borohydride + 3 M NaOH, [oxidant]: 0.15 M percarbonate + x M H_2SO_4). Stream flow rates: 0.3 mL min^{-1}

Effect of flow rate

The effect of fuel mixture flow plays an important role in the performance of MLBFCs and provides control over the transport time of the reacting species moving between the anode and the cathode. In this experiment, fuel mixture flow rates of 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 0.9, and 1.0 ml/min were tested. The cell potential and current density were measured with different external loads as a function of the flow velocity of the fuel mixture. Using the flow rate applied and the cross-sectional area of the channel, flow velocity can be calculated. The higher potential and current density was found to be achieved at a flow rate of 0.3 ml flow rate, beyond which the cell performance is found to decrease, as shown in Figure. 5.

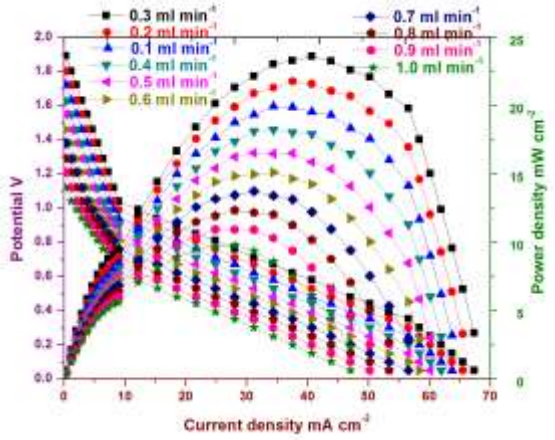

Figure 5. Curves of cell polarization and power density for MLBFC at different flow rates. Fuel: 0.15 M $NaBH_4$ in 3 M NaOH solutions. Oxidant: 0.15 M percarbonate in 1.5 M H_2SO_4 solutions

Effect of distance between the anode and the cathode

The influence of distance between the anode and the cathode on the fuel cell output was experimentally studied, as different inter-electrode distances can significantly alter cell resistance and thus cell performance. The distance between the anode and the cathode was varied from 1 to 100 mm. As the distance between the anode and the cathode is decreased, the maximum output power increased due to the reduced cell resistance. Figure 6 shows that the maximum power density is achieved at an inter-electrode distance of 2 mm. Considering the role of a charge carrier, a shorter diffusion length is believed to lead to a faster electrochemical reaction because the diffusion time of reacting species would be shorter. Therefore, more reactions can take place at a given time, which increases the total number of charges involving the electrochemical reactions at the anode and the cathode. This observation provides a clear evidence for the presence of a charge carrier moving between the anode and the cathode in the fuel mixture to complete the redox reactions of the fuel cell [22].

Cell stability study

The stability of the fuel cell was tested by applying a constant current density (30 mA cm^{-2}) in the absence of current flow. Short-term

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stability of MLBFC was tested by monitoring the cell voltage change during the galvanostatic discharge of the MLBFC for a period of about 100 h, an example of which is shown in Figure. 7. The fluctuation in the cell voltage seen in the figure is due to addition of the solutions or restarting the experiments after an overnight break. The MLBFC was found to maintain a relatively stable performance with little decay of the cell voltage over the entire test period. The cause for deterioration is presumably changes in the catalyst surface area, supplies of the fuel and the oxidant as well as the removal of the product, among others.

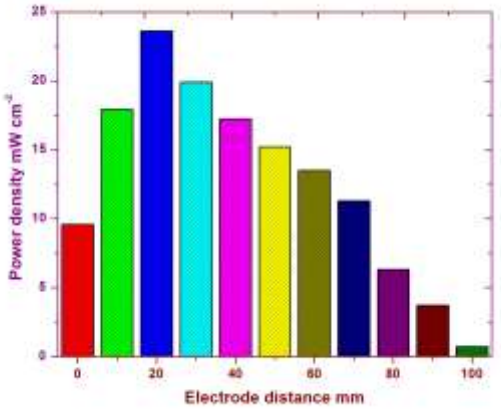

Figure 6. Effect of distance between the anode and the cathode on the maximum power density of the MLBFC at room temperature. [Fuel]: 0.15 M sodium borohydride + 3 M NaOH. [Oxidant]: 0.15 M percarbonate + 1.5 M H_2SO_4. Stream flow rates: 0.3 mL min^{-1}

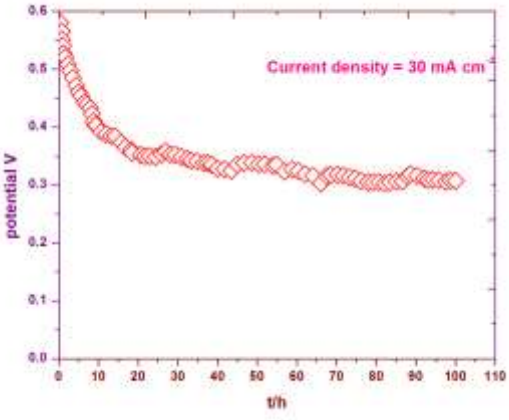

Figure 7. Performance stability of the MLBFC operating at a current density of 30 mA cm^{-2} at room temperature. [Fuel]: 0.15 M sodium borohydride + 3 M NaOH. [Oxidant]: 0.15 M percarbonate + 1.5 M H_2SO_4. Stream flow rates: 0.3 mL min^{-1}

Conclusions

A small-scale membraneless sodium percarbonate fuel cell was fabricated on poly(dimethylsiloxane) and its performance was evaluated under different operating conditions. Standard microfabrication techniques were used to develop the fuel cell. In this membraneless fuel cell, sodium borohydride was used as a fuel at the anode and percarbonate was used as an oxidant at the cathode. This is first time a combination of alkaline anode and acidic cathode is used, to the best of our knowledge, in a membraneless fuel cell. The experiments described in this study indicate clearly that membraneless sodium percarbonate fuel cells are media flexible; they can operate in all-acidic, all-alkaline, or even combined alkaline and acid media configurations. At room temperature, the laminar flow-based microfluidic fuel cell produced a maximum power density of 23.61 mW cm^{-2}. In the fuel cell, power density was found to increase with an increase in percarbonate concentration till 0.15 M and above this concentration, a decrease in cell performance was noted. The variation of sodium borohydride concentration at the anode produced was found to have slight influence on the cell performance. Thus, the present experimental results have confirmed that this membraneless microfuel cell is cathode-limited, and indicate that a crucial factor for improving cell performance is increasing the concentration of the oxidant in the cathode stream. The membraneless microfuel cell system investigated in this study seems to be a good candidate for feasible application in portable power electronics, because its performance is comparable to an air-breathing DMFC without CO_2 emission. In addition, on varying flow rates, studying the effect of electrode distance, and performing a durability test in our study, it is found that a flow rate of 0.3 ml and a distance of 20 mm distance result in good cell performance. The results demonstrated that the performance of the developed membraneless fuel cell is enhanced significantly when the concentration of the oxidant in the cathodic stream is 10 times larger, and the current density is also increased approximately 10 times. The MLSPCFC provides advantages of miniature size, simplicity of fabrication, use of aqueous fuel, and cost efficiency. Furthermore, percarbonate is a cost-effective, nontoxic, stable, easily handled,

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environmental friendly, large-scale industrial chemical and is a convenient source of hydrogen peroxide. We hope that the membraneless sodium percarbonate fuel cell may be a promising candidate for use as practical fuel cells to provide a clean and sustainable energy in future.

Conflict of interest

Authors declare there are no conflicts of interest.

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