



R&D activities of fuel cell Research at KFUPM

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Abstract

Development of systems with reduced emission of pollutants is one of the major challenges of this century. Fuel cells promise to provide clean and renewable source of energy, which can operate on many fuels. They are promising candidates for transportation and portable power source applications. These applications include battery replacement for portable telephones and computers, power sources in remote areas, etc. Fuel cells can also work using other fuels such as hydrocarbons including methanol either directly or indirectly. Since there is plenty of hydrocarbons resources available in the Gulf region such as gasoline, naphtha, and methane/natural gas etc., they can be utilized in the fuel cell to produce clean power without combustion. There has been tremendous research effort in other parts of the world, especially the Western world for the development of fuel cells. Research at KFUPM is focused on two different aspects, fuel for fuel cells (reformat feed) and PEM fuel cell system. Our research group at KFUPM is actively involved in fuel cell research since 1980s. Current focus is to develop PEM fuel cell system emphasizing three different aspects: (A) developing novel low cost proton conducting membranes, (B) developing multifunctional catalyst system and (C) development of hydrocarbon based fuel processing systems. In this communication research activities and/ongoing projects undertaken at KFUPM for fuel cell development will be presented.

Keywords: PEM fuel cell; Membranes; Electrochemical filter; Reformate

1. Introduction

Fuel cells do not need an introduction now. They are being considered as savior of environment without compromising society's energy addiction. They have gone through a long journey from their humble invention in 19th century

to potential widespread automotive application. Fuel cells are now being seen as alternative to internal combustion engines in automobiles and batteries in so many portable applications.

The electrochemical research group at KFUPM has been actively engaged in fuel-cell research and development activities since early 1980s. Earlier work started with the development of alkaline fuel cells (AFC). The main

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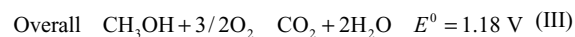
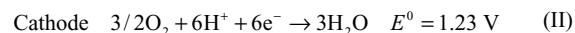
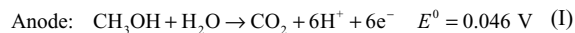
contributions were basically in three areas, namely, electrode development [1–3], mathematical modeling of the electrode performance [4,5] and electrode performance testing [6–13]. In the recent years, the focus shifted from AFC to polymer electrolyte membrane fuel cell (PEMFC) and fuel processing system. Low cost membranes operable at relatively higher temperature are much needed. The efforts in this direction exploit the use of less expensive polymers to prepare membranes after their modification and also by blending them with inorganic proton conducting solid materials [14–23]. For direct methanol fuel cells (DMFC) we are working to develop a multifunctional catalyst which will be more efficient in the presence of higher local carbon monoxide concentration.

It has been observed that the practical fuel-cell automobile will run on hydrogen produced from fossil fuel because of two reasons: (a) alternative options to produce hydrogen are not commercially developed at this time and (b) there exists an efficient distribution infrastructure for fossil fuel. However the fuel processing for producing hydrogen suitable for fuel cells needs lots of effort. A group at KFUPM is engaged in the development of efficient catalyst to do the job. In addition, the group is also working on the development of low-cost polymer membranes, which is expected to reduce the permeation of methanol, a major problem in the DMFC, in addition to providing high conductance. Also, KFUPM is working on a novel concept of electrochemical carbon monoxide filters to process the output of a catalytic reformer. In this article all of these research and development activities are described.

2. Development of proton conducting membranes

The direct methanol fuel cell works by oxidizing the liquid methanol to CO_2 and water. This eliminates the need for an external hydrogen

supply. A solid proton conducting membrane, used both as an electrolyte and separator between anode and cathode, is sandwiched between porous carbon structures. The relevant electrode reactions are



The maximum voltage attainable from the overall reaction in the methanol–air fuel cell is 1.18 V with a theoretical efficiency of 96.5%, but in practice it is not achieved due to the poor electrode kinetics and ohmic losses through the electrolyte [24,25].

Presently, perfluorosulfonic acid membranes, trade named as Nafion produced by Du Pont and Dow companies are widely used as solid polymer electrolyte. The membrane provides ionic communication between the electrodes as well as acts as a separator for the reactants.

Currently there are some obstacles [26,27] which need to be overcome before large scale commercialization of DMFC: (a) low activity and high cost of anode electrocatalyst, the anode reaction has poor electrode kinetics, particularly at lower temperatures, making it highly desirable to identify improved catalysts and to work at as high a temperature as possible; (b) the reduction of oxygen on cathode is also low though the problems are not so serious as with aqueous mineral acid electrolytes; (c) the permeability of the current perfluorosulfonic acid membranes, Nafion to methanol, which allow considerable crossover of methanol from anode region to cathode region. And the last, but not less significant is the high cost of Nafion membrane in the range of US\$ 800–2000/m². The high price of Nafion provides incentive for the development of less expensive proton conducting membranes. A number of studies have

been carried out for the development of less expensive and methanol resistant membranes, but still Nafion remains the material of choice.

In search of low cost and thermally stable proton conducting membranes Zaidi et al. [15–17] developed a series of composite proton conducting membranes by incorporation of solid proton conductors such as powdered boron phosphate, heteropolyacids and their compounds into sulfonated polyether–ether ketone (SPEEK) matrix. These membranes showed a high conductivity of the order of 0.2 S/cm, which is higher than exhibited by Nafion 117. They have been studied for their electrochemical properties such as conductivity and morphology and were subjected to other membrane characterizations. However, these membranes have not been tested in an actual fuel cell with methanol or hydrogen. These membranes need to be tested in a direct methanol fuel cell for their performance evaluation and longevity tests. At KFUPM, we are working for the performance testing and development of novel low cost membranes for fuel cell aimed at reducing the methanol permeation as well as increasing conductivity. Also, we are actively engaged for the development of new membranes based on the tungstophosphoric acid supported mesoporous materials and molecular sieve zeolites, aimed at reducing the methanol crossover. The outcome of this research study will result in the low cost membranes with improved properties. This project will also help develop an expertise in this field in the Gulf region.

3. Development of multifunctional electro-catalyst for direct methanol PEM fuel cell

The electro-oxidation of methanol requires the presence of platinum based catalyst as it is involved in two key steps occurring during oxidation route. One is the dehydrogenation step and the second is the chemisorption of CO. The methanol

electro-oxidation reaction is a slow process and it involves the transfer of six electrons to the electrode for complete oxidation to carbon dioxide. Various reaction intermediates may be formed during methanol oxidation [28]. Some of these (CO-like) species are irreversibly adsorbed on the surface of the electrocatalyst and severely poison Pt for the occurrence of the overall reaction, which has the effect of significantly reducing the fuel consumption efficiency and the power density of fuel cell. Thus it is very important to develop new electrocatalysts to inhibit the poisoning and significantly increase the rate of electro-oxidation by at least a factor of two to three times. Until now platinum is proved to be the only effective anode catalyst for DMFC. In a recent study [29] it has been shown that the electro-oxidation of methanol is promoted by silicotungstic ($H_4SiW_{12}O_{40}$) acid when heteropolyacid is used as an electrolyte. The promoting behavior of $H_4SiW_{12}O_{40}$ for methanol electro-oxidation is still present even when the heteropolyacid is dissolved in small volume of sulfuric acid. The kinetics of the electro-oxidation of carbon monoxide in phosphotungstic acid (PWA) electrolyte also showed some improvement [30–31].

The research in methanol electro-oxidation using Pt anode reached important breakthroughs during the last 15 years. The most significant issue in the development of methanol fuel cells for generating electric current is the poisoning of the platinum anode by carbon monoxide that is generated during the oxidation. Carbon monoxide molecules formed from the early steps of methanol oxidation adsorb on and block polycrystalline platinum electrode surfaces and are not oxidized away by the reaction with water to make carbon dioxide unless the anode potential is increased to about 0.6V(SHE). The net result of doing this is an unacceptable loss of cell voltage and efficiency. Many attempts have been made in order to increase the catalytic activity and to decrease the poisoning of the electrode by preparation of multiple metal anodes. Platinum

containing bimetallic alloy anodes was investigated for Pt–Ru, Pt–Sn, Pt–Pd, Pt–Rd, Pt–Pb, Pt–Ti, Pt–Re [32]. It was reported that the addition of Ru, Sn, Ti, and Pb yields a positive effect for the methanol electrooxidation. The function of Sn and Ti appears to promote the formation of active Pt–O groups capable of completing the oxidation reaction. It has been shown that alloying of Sn and Ru with Pt gives rise to electrocatalysts, which strongly promote the oxidation of methanol and CO. To date a Pt–Ru electrocatalyst (1:1) wt.% has shown the best results [33].

From the detailed literature review it can be seen that the electrocatalyst activity of the presently used catalyst is very low and the platinum consumption is also very high. There is a need to develop and/or modify the anode catalyst for the methanol fuel cell in order for the DMFC to show the performance comparable to the hydrogen fuel cell.

At KFUPM, we are in the process of developing new electrocatalyst for the methanol oxidation in fuel cell. The focus of the study will be on the development of novel electrocatalyst formulations using heteropolyacids and Pt–Ru catalyst supported on activated carbon for methanol oxidation for DMFC. Recently, it has been demonstrated that the electrooxidation of methanol improves when heteropolyacids are used

as electrolyte. Significant improvement in the activity was also reported when heteropolyacids were dissolved in sulphuric acid electrolyte. In another study, presence of heteropolyacids in electrolyte was observed to improve oxidation of carbon monoxide. These recent developments prompted us to suggest heteropolyacids as a component in electrocatalyst formulation.

4. Fuel processing systems

4.1. Reforming of hydrocarbon fuels

The production of hydrogen from hydrocarbons is a well-established process, with a combination of oxidation and steam reforming of methane forming the basis of an important industrial process. However, the constraints on the overall process in the context of supplying hydrogen for fuel cells introduce the possibility of new reactions and different catalysts. On-board reforming of hydrocarbons for automotive applications comprise three options namely, steam reforming, catalytic partial oxidation, and auto-thermal reforming. Table 1 presents the characteristics of these options. Nearly all processes considered for commercial exploitation are based on autothermal reforming or partial oxidation.

Table 1
Fuel reforming options [34]

Option and typical reaction	v
Steam reforming (SR) $C_8H_{18} + 8H_2O \rightarrow 8CO + 17H_2$ $\Delta H = +1273 \text{ kJ/mole}$	Highest H ₂ concentration Heat transfer limited Complicated design
Catalytic partial oxidation (CPOX) $C_8H_{18} + 4O_2(N_2) \rightarrow 8CO + 19H_2$ $\Delta H = -661 \text{ kJ/mole}$	Simple design, fast response Forms coke Contains N ₂
Auto-thermal reforming (ATT) $C_8H_{18} + 4O_2(N_2) + 8H_2O \rightarrow 8CO + 17H_2$ $\Delta H = -990 \text{ kJ/mole}$	Simple design, fast response Requires water of recycle Contains N ₂

For fuel-cell applications, a supply of pure hydrogen is desired and more selective processing is required. This usually involves a mixture of oxidation and steam reforming or partial oxidation. As a result, the production of hydrogen from natural gas, LPG (liquefied petroleum gas), methanol, gasoline, and diesel has received attention and considered by many researchers worldwide. The disadvantages of using methanol for the production of hydrogen are due to the following: toxicity of methanol, its low energy density, marginal production capacity for fuel use, as well as the non-existing infrastructure. On the other hand, the advantages of using methanol include: pure compound, liquid syngas, less nitrogen in process stream. Using gasoline offers some advantages such as non-toxicity, highest energy density, adequate production for fuel use, and global availability. Its disadvantages include complex mixture, and during partial oxidation 50% of nitrogen is produced in process stream.

Progress is being made by improving catalysts employed in many of these hydrogen-producing reactions. It is thought that continued tinkering with new catalysts can bring hydrogen power into the mainstream. Common catalysts have included precious metals like gold and platinum. Cheaper materials are just part of this cost equation. Finding catalysts that are either more active or will work at lower temperatures is another crucial step [35]. Most commercial hydrogen today is produced in natural-gas power plants by reacting steam and methane over a catalyst at high temperatures. But cooler reactions requiring less energy could make smaller-scale hydrogen production affordable.

The steam reforming of hydrocarbons to produce hydrogen is favored at higher temperatures, where coke formation can be a problem. Sufficient water must also be added to minimize coking, although a choice of catalyst may reduce the amount of coke to be gasified. With very heavy fuels, it may be preferable to move

to non-catalytic partial combustion. Methanol and lower-molecular-weight hydrocarbons can be steam reformed to produce hydrogen. Copper–zinc oxide or palladium–zinc oxide are the preferred catalysts for methanol, but careful control of temperature is necessary with copper-based catalysts. Nickel-based systems are more cost-effective for light hydrocarbons, although rhodium is an excellent catalyst. Coking is a potential problem which increases as the molecular weight and aromaticity/olefinicity of the fuel increases [36].

Steam reforming reactions are endothermic, and heat must be supplied either externally or by combustion of reactants and products. Autothermal operations are stand-alone systems in which fuel is partly oxidized and partly steam reformed to produce hydrogen. The system must start to operate at low temperatures, with the balance between oxidation and steam reforming depending on the heat demands of the particular fuel. Reactors based on methanol are widely available, but other fuels may be used if reactor operation is adjusted. The wide availability of LPG, gasoline, and diesel would make them ideal as fuels for hydrogen production. Regrettably, not enough attention has been focused on the catalytic conversion, particularly of LPG.

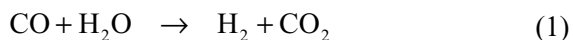
Currently, KFUPM is conducting a research project for the development of a new reforming catalyst for hydrogen production from liquid hydrocarbons. The project comprises catalyst preparation and performance evaluation in laboratory scale unit.

4.2. Removal of CO from reformat

Proton exchange membrane fuel cells (PEMFCs) operating on pure hydrogen exhibit excellent energy and power capabilities. Most CO tolerable electrocatalyst (Pt–Ru) would require H₂ with less than 100 ppm [37]. The poisoning of CO can reduce the performance of the PEM fuel cell by 40–60% depending upon the concentration of the

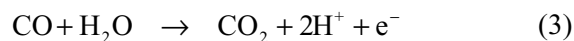
CO in the feed. Conventionally, reducing CO content of the reformat stream involves water gas shift reactor and preferential oxidation (Prox), which takes up a large volume of the fuel cell system and leave a stream that still contain about 100 ppm CO. Extensive efforts have been made to develop new effective and efficient technologies to remove even traces of CO present in the reformat gas. Some of the unconventional approaches of CO removal are as plasma reforming [38–41], methanation [42–44] adsorption [45–47], H₂ purification membrane [48,49], water-gas shift membrane reactor [50–52] and electrochemical CO filter [53–55].

Electrochemical CO filter (EF) is an electrochemical device that preferentially oxidizes CO over H₂ present in reformat by using current PEMFC technology. The proposed filter may be capable of replacing either low temperature shift (LTS) or PrOx reactor or both LTS and PrOx reactors used in a conventional fuel processing system. The electrochemical filter operated under a pulse potential mode to achieve high selectivity for CO oxidation over H₂ oxidation. During the off portion of the pulse, the cell is open circuited and CO adsorption is promoted. During the on portion, CO oxidation is preferred over H₂ oxidation while the surface is covered with CO. Repeated pulsing enables continuous removal of CO from the reformat gas. Hydrogen is produced on the cathode from H⁺ formed during the oxidation of CO and H₂, such that the overall reactions in the cell are given by reactions (1) and (2) below.

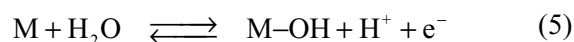
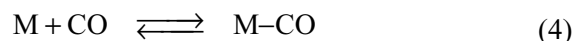


Reaction (2) is shown to remind us that all the H₂ oxidized at the anode (H₂ → 2H⁺ + 2e⁻) is formed back in the cathode (2H⁺ + e⁻ → H₂) resulting in no net consumption of H₂. However, this reaction does represent, net power consumption in

the device thus impacting the overall electrical efficiency of the fuel cell system. Electro-oxidation of CO occurs through the following reaction (3).

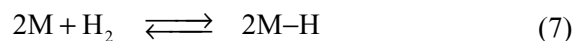


The mechanistic steps of the above reaction are given as follows:



where, M refers to either Pt or Ru metal catalysts sites.

Reaction (4) corresponds to the adsorption of CO onto the Pt–Ru catalyst (i.e., CO poisoning). Reaction (5) corresponds to the formation of hydroxyl ions via oxidation of water, and reaction (6) corresponds to the oxidation of adsorbed CO by reaction with the hydroxyl species [56]. The competitive adsorption and oxidation of H₂ are shown in reactions (7) and (8), respectively.



The preliminary results show that by varying the pulse-potential profile (e.g., on time, off time, and pulse potential) the CO and H₂ oxidation currents can be varied independently. CO adsorption is promoted during the off portion of the pulse (i.e., open circuit). During the on-portion of the pulse, CO oxidation is preferred while the surface is covered with CO. It is hypothesized that an optimized ex situ electrochemical filter could replace both LTS and PrOx units. This would reduce the size of the fuel processing system by a factor of three. Others have used pulse-potential control to oxidize the

adsorbed CO in PEMFC and direct methanol fuel cell, however they have pulsed the fuel cell rather than an external unit. The advantage of the present approach is that the catalyst and operating conditions can be optimized for each device independently, and the pulse profile does not interfere with the operation of the fuel cell.

Our group at KFUPM is actively engaged in furthering this research. We are currently interested in finding out the best potential profile and potential values. We intend to make a scale up unit for demonstration.

5. Conclusions

Fuel cells have the potential of providing cleaner source of energy and hence will result in pollution free environment. They are now considered at the threshold of commercialization. The commercialization of fuel cells especially direct methanol fuel cell is in the strategic interest of the region and methanol producing companies like Sabic will be the direct beneficiaries. The research in the fuel cell area will provide futuristic benefits to the Kingdom as this technology is now being commercialized in many advanced countries. The market penetration of this technology will depend on the rate of technological advancements. At KFUPM we are actively engaged in the fuel cell research aimed at the development of fuel cell systems.

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