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ADVANCEMENT IN APPLICABILITY OF CARBON NANOTUBES IN PROGRESSIVE FUEL CELLS

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Abstract. This paper focuses on proton exchange membrane fuel cells (PEMFC) and usage of carbon nanotubes for increased efficiency. The paper also discusses a possible material that may find potential application in the fuel cell as an alternative to the carbon nanotubes existing so far.

Keywords: carbon nanotubes, catalyst support, proton exchange membrane fuel cells, azo-benzene carbon nanotubes.

1. Introduction

Carbon nanotubes (CNTs) have played an important role in ensuring the economic feasibility of fuel cells due to their application as a catalyst support. However, research on further improving upon these nanostructures still continues. This paper explores the possibility of using azo-benzene based carbon nanotubes as a catalyst support in proton exchange membrane fuel cells. These special types of functionalized carbon nanotubes were originally developed as a means to convert and store solar energy to chemical energy. The objective of this review is to put forward the idea of combining this particular nanostructure's applications to harness two forms of renewable energy-solar energy and hydrogen energy. It also discusses the possibility of broadening the prospects of PEMFC in the global market.

Carbon nanotubes were first observed by Japanese researcher Sumio Ijima, in 1991 in the carbon soot of graphite electrodes during an arc discharge, by using a current of 100 amps, that was intended to produce fullerenes. A carbon nanotube is essentially an allotrope of carbon, comprised of graphite sheets rolled up into hollow cylinders. These structures can be classified as separate molecules due to their dimensions. Their diameters range in the order of nanometres, and their length in approximately micrometres. The folding of graphite sheets can occur in numerous ways, resulting in different diameters and microscopic structure. The resulting geometric structures can be used to explain various properties of the nanotubes.

The synthesis processes adapted to manufacture single walled (SWNT) or multi-walled (MWNT) nanotubes depending on the applications [1].

The applications decide the properties of the CNTs to be used. Hence, the structure, morphology, properties *etc.*, all depend on the manufacturing technique used. Carbon nanotubes are commonly produced by the following techniques: [1]

- Arc discharge
- Laser ablation
- Chemical vapor deposition

The primary importance of the carbon nanotube lies in its nanostructure. Due to its dimensions, it will tend to exhibit properties not observed in most of the bulk materials in use today. Some of the properties (Table 1, Fig. 1) that make this material lucrative include increased surface area, high electrical and thermal conductivity, high tensile strength (~100 times than that of steel), flexibility and ease of manipulation without causing significant change in the properties.

Table 1

Comparison of different materials properties

Fibre material	Specific density	Strength, GPa	Strain at break, %
Carbon nanotube	1.3–2	10–60	10
HS Steel	7.8	4.1	<10
Kevlar 49	1.4	3.6–4.1	2.8
Nanocellulose	1.45	0.5	12

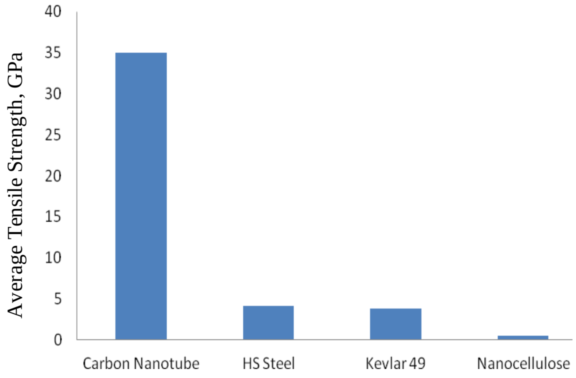

Fig. 1. Average tensile strength of different materials compared to CNT [1]

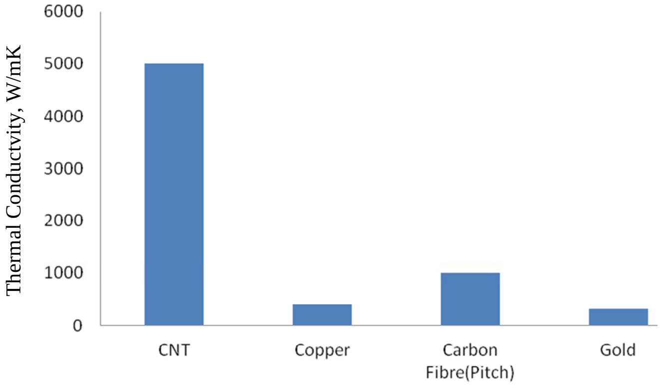

Fig. 2. Thermal conductivity of different materials compared to CNT [1]

Table 2

Electrical conductivity of different materials compared to CNT [1, 3]

Material	Electrical conductivity, S/m
Graphite	$3{\cdot}10^5$
CNT	$6.7{\cdot}10^6$
Copper	$6{\cdot}10^7$
Mercury	$1.02{\cdot}10^6$

Compared to other conductive materials, the thermal and electrical conductivity of carbon nanotubes are also very high (Fig. 2, Table 2).

As is evident from the above data, carbon nanotubes have much higher electrical conductivity compared to any other form of carbon. This makes them useful in the field of energy as effective electrodes (when grown on graphene sheets) and catalyst supports in fuel cells, due to their large surface area. A. Peigney *et al.* [4] carried out BET calculations to determine the surface area of carbon nanotubes. It was found to be 1315 m^2/g for SWNT and 50 m^2/g for MWNT with a diameter of 35nm and 40 walls. Carbon nanotubes are of primary importance as a catalyst support in proton exchange membrane fuel cells. Though copper has a much higher electrical conductivity than carbon nanotubes, it does not have high surface area, which is also an important criterion for a catalyst support in such fuel cells.

1.1. Fuel Cell

A fuel cell (Fig. 3) is like a battery that generates electricity from an electrochemical reaction. Unlike a battery, the fuel cell does not store energy within it for use until it is depleted. A fuel cell can run indefinitely as long as it supplied with an external source of chemical energy.

This source of chemical energy is hydrogen. Hydrogen can be directly supplied from hydrogen storage devices or indirectly. Indirect hydrogen supply is provided in the form of hydrocarbon fuels like methane, coal based gases, *etc*. Despite being labeled as a "fuel", hydrogen does not undergo combustion but simply oxidation.

A fuel cell usually consists of anode (positive), cathode (negative), an electrolyte to carry charged particles between the electrodes (must be selective in nature) and a catalyst [2].The basic working of a typical fuel cell can be summarized as follows:

- Hydrogen enters the cells at the anode to get oxidized to H^+ ions. Electrons generated pass through external circuit.
- Protons pass through the electrolyte towards the cathode.
- Oxygen enters the cathode and combines with the electrons and protons to release water as the final by-product.

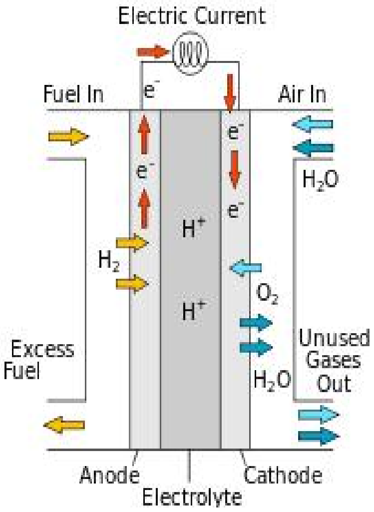

Fig. 3. A typical fuel cell [2]

Some of the most commonly used varieties of fuel cells are solid oxide fuel cells, molten carbonate fuel cells and PEMFC. These cells differ from one another based on electrode material, nature of the electrolyte, working principle of the fuel cell and original fuel supplied. However, our focus will be on the proton exchange membrane fuel cells.

1.2. Proton Exchange Membrane Fuel Cells

These are low temperature fuel cells operating at the temperature ranges within 353–373 K (low temperature PEMFC) or 373–473 K (high temperature PEMFC). High temperature PEMFCs have a higher platinum loading and resistance to impurities, but a lower power density than low temperature PEMFC [2]. The average current density in these fuel cells should ideally be 3321 A/m^2 for commercial viability [3].

These cells are comprised of a solid electrolyte in the form of water based acidic thin membrane sheet, usually made from polymers, *e.g.* Nafion. The electrolyte consists of platinum catalyst on either side. Thus we have platinum based electrodes in this cell. Platinum speeds up the reactions at both the electrodes.

Platinum is present as small particles on larger carbon particles that act as a catalyst support (Fig. 4).

Due to the use of carbon particles as the catalyst support, there remains scope for further improvement in the cell performance in the form of carbon nanotubes. Pt catalyst used to constitute 40 % of the cost of PEMFCs.Their use was therefore limited to military and spacecraft applications, but now with the advent of CNTs, the scope of their application has extended to simple, everyday electronic devices like laptops, phones, cars, buses, *etc*.

Fig. 4. Pt catalyst on C

Commonly used catalyst supports are platinum black, carbon black, gold, activated carbon and more recently, carbon nanotubes. Apart from gold, all of these have been used as catalyst supports in PEMFCs. Platinum black was initially used, but was found unsuitable [6]. One of the reasons was its low current density of 5 mA/cm^2.

The electrical conductivity of these catalyst supports are illustrated in Table 3.

Therefore, as can be concluded from the above data, carbon nanotubes are the best choice for catalyst supports in the fuel cells. Their high electrical conductivity and surface area ensures a much lower resistance to flow of electrons.

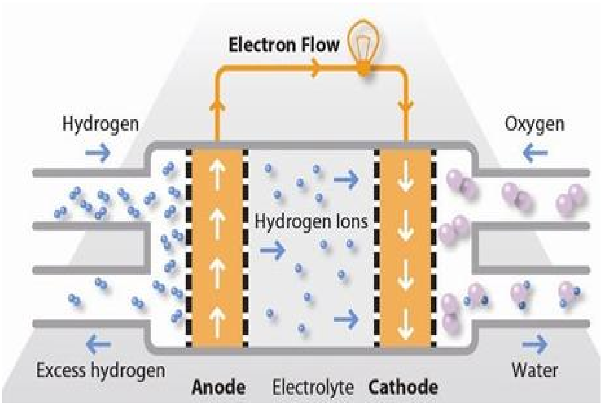

Fig. 5. Proton exchange membrane fuel cell

Principle of exchange membrane fuel cell working [2] is illustrated in Fig. 5.

- Hydrogen is oxidized at the anode to produce hydrogen ions.
- The hydrogen ions migrate across the Nafion polymer membrane (electrolyte) towards the cathode where they combine with oxygen and electrons to form water as the final product.
- The catalyst is coated onto the electrolyte by rolling, spraying or hot pressing.

Table 3

Electrical conductivity of different catalyst supports compared to carbon nanotubes [5]

Material	Electrical conductivity, S/m
Platinum black	$1.05 \cdot 10^{-7}$
Gold	$2.214 \cdot 10^{-8}$
Carbon black	0.024
Activated carbon	60
Carbon nanotubes	$67 \cdot 10^{5}$

These fuel cells have the following advantages:

- Compact, quick start up (due to low operating temperature), light weight.
- No hazard of corrosive fluid storage.
- More efficient compared to other fuel cells (40 % efficiency).

Limitations of described fuel cells and the "carbon nanotube solution"

Despite their advantages, these cells originally had certain limitations that have been more or less successfully overcome by the use of carbon nanotubes.

1. Improvement of platinum loading at the electrodes, especially the cathode.

Originally, platinum black was used as the catalyst for PEMFCs. Platinum black as the catalyst involved coating black platinum powders as a thin film on a platinum surface. However due to the presence of less active sites and higher platinum loading (~25–50 mg/cm^2), it was replaced by carbon supported platinum catalysts which have a higher surface area [4]. With the introduction of more active sites, a smaller platinum loading is feasible as it is distributed over a larger area. Platinum loading is reduced to 0.4–0.5 mg/cm^2, thus making the fuel cell a more economically more viable option.

Alternative catalyst support combinations can also be considered to improve platinum utilization, *e.g.* Pt/X (X is Co, Ni or Fe).

Carbon nanotube solution: Carbon nanotubes have also gained popularity in the role of catalyst support because of their high surface area thereby leading to greater number of active sites and smaller catalyst particles.

Vertically aligned CNTs can be a promising catalyst support compared to other CNTs due to their highly ordered structure. This ensures larger surface area, good electrical conductivity and more active sites for Pt utilization.

2. Requirement of an efficient water management system for adequate membrane hydration.

Water accumulates at the cathode and must be effectively managed to ensure that the cell membrane electrolyte does not dry up (in case of too much water removal), or the cell does not flood (accumulation of too much water). Cell flooding blocks access of reacting gases to platinum. A dry membrane will not be able to conduct hydrogen ions from anode to the cathode. A well chosen catalyst support will have the required hydrophyllic and hydrophobic properties to ensure an optimum water balance [6].

Carbon nanotube solution: Once again, carbon nanotubes can be considered for this particular function due to their high graphite content which makes the membrane hydrophobic [7]. CNTs or any other carbon material can be modified to have the desirable hydrophobicity.

3. Complex oxygen reduction reaction at the cathode.

Unlike the oxidation reaction at the anode, oxygen reduction reaction at the cathode has a slow and complex mechanism. Thus it requires more platinum loading to speed up the reaction. Increased Pt loading requires higher number of active sites, and hence a greater surface area catalyst support. The catalyst support can be modified with additional functional groups that can act as sites for Pt.

Carbon nanotube solution: CNTs with their higher surface area can ensure availability of more active sites and higher Pt loading. However, it is important not to exceed a certain limit for Pt loading in the catalyst support. Too many Pt particles usually include larger particles which effectively reduce the availability of active surface areas. Thus, even with carbon nanotubes as a catalyst support, the cell performance is adversely affected.

4. Degradation of membrane by peroxide.

The membrane used as electrolyte in proton exchange membrane fuel cells is made of a polymer called Nafion. Membranes used ideally must possess the following properties:

- Proton conductivity
- Electron insulation to prevent short circuit
- Low permeability to feed fuels (hydrogen, methanol)
- Mechanical and chemical stability

The advantages of using Nafion membranes include high proton conductivity, electron insulation and reasonable chemical stability. However, they do have their limitations such as moderate mechanical strength at high temperature which can cause breakdown of these membranes during fabrication and operation (swelling and contraction of the membrane due to differing water content) and degradation of Nafion in the presence of peroxide radicals that are occasionally formed at the cathode.

Oxygen reduction at the cathode can take two pathways [7]:

(i) $O^* + H^+ + e^- \rightarrow OH^*$

$OH^* + H^+ + e^- \rightarrow H_2O^{+}*$

(ii) $O_2 + * \rightarrow O_2^*$

$O_2^* + H^+ + e^- \rightarrow HO_2^*$

$HO_2^* + H^+ + e^- \rightarrow H_2O_2^*$

* denotes a vacant site

The second pathway is a highly undesirable one due to the formation of peroxide which on desorption can degrade the membrane. An important relation has been obtained between the number of electrons transferred and the pathway to be followed: the number of electrons transferred (n) denotes which mechanism is to be followed.

- n = 2 implies poor oxygen dissociation at the cathode, leading to H_2O_2 formation.
- n = 4 implies the normal pathway.

Carbon nanotube solution: Nitrogen doped vertically aligned carbon nanotubes can be used as metal free catalysts in PEMFCs. Their main advantage is that they ensure the 4 electron pathway required to avoid formation of H_2O_2. Unfortunately, they fail to demonstrate the same level of activity as Pt catalyst.

5. Loss of stability of the catalyst.

Sintering of Pt particles reduces the number of active sites. The Pt particles dissolve in the acidic environment and precipitate into larger particles which further reduce the availability of active sites. The main aim in this case would be to strengthen the bond between the metal catalyst and support so as to decrease the mobility of Pt particles.

Corrosion of the carbon catalyst support is another major disadvantage. This process occurs by oxidation of the carbon followed by hydrolysis and gasification to carbon dioxide [7]:

$$C_s \rightarrow C_s^+ + e^-$$

$$C_s^+ + \frac{1}{2}H_2O \rightarrow C_sO + H^+$$

$$C_sO + H_2O \rightarrow CO_2\,(g) + 2H^+ + 2e$$

Carbon nanotube solution: A thin Pt film can be coated on the electrode surface using techniques like sputter deposition. The resulting film completely covers the CNT, reducing carbon corrosion. The use of carbon nanotubes implies increased graphitization, thereby reducing the number of oxidization sites for carbon corrosion to take place in. Also, the strong connection between the Pt surface and the CNT wall hinders detachment of Pt molecules, ensuring stability of the catalyst.

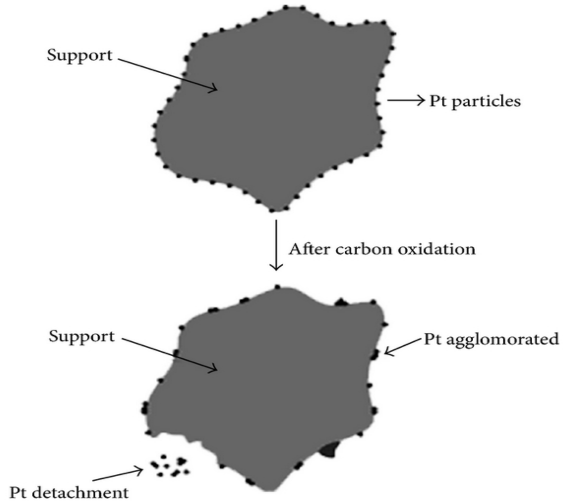

Fig. 6. Pt detachment from the catalyst support

1.3. Types of Carbon Nanotubes Can be Used in Fuel Cells

As discussed previously, the primary role of carbon nanotubes in a proton exchange membrane fuel cell is that of a catalyst support. A catalyst support is a material to which a catalyst is affixed, for the purpose of increasing the overall surface distribution of the catalyst. Carbon nanotubes have several advantageous properties that make them very attractive options as catalyst supports [7].

Firstly, their hollow cylindrical geometry guarantees a high surface area for anchoring the catalyst particles. Another advantage of carbon nanotubes is their chemical inertness. Other important properties include good electron conductivity, mechanical strength and thermal conductivity, which are important factors in the consideration of choosing a good catalyst support. Electronic properties of carbon nanotubes are also important in their role as a catalyst support. They can be either metallic or semiconductive, depending on their helicity and diameter, and this property can be used to manipulate charge transfer processes. Carbon nanotubes also possess a very large Young's modulus of 1000 GPa and great tensile strength of 63 GPa. Their good thermal conductivity helps to prevent the agglomeration and growth of small nanoparticles during postannealing treatments.

We have therefore seen, how carbon nanotubes, with the help of their unique properties have helped counter the various disadvantages of a proton exchange membrane fuel cell. These properties also give them an edge over other previously used material in the proton exchange membrane fuel cells (Table 4).

Table 4

Comparison of the different materials used at the electrode for PEMFC

Material	Particle size, nm	Pt content, wt %	ECS, m^2/g
Pt-black	10–15	98	16–20
Pt/ Carbon support	5–6	51	0.46 + (or –) 0.07
Pt/ CNT	1–2 3–4	20	0.47 + (or –) 0.07

Table 5

Comparison of Pt/VACNT and Pt/commercial CNT in terms of power density [11]

Property/CNT used	Pt/VACNT	Pt/commercial CNT
Power density, mW/cm^2	697	525
Pt utilization, g/kW	0.30	0.38

However, they still have their limitations, and hence further exploration of carbon nanotube materials and their variations is required to suggest improved catalyst supports.

Various types of carbon nanotubes have found application in these fuel cells. These nanotubes range from single-walled to multi-walled, doped to functionalized and differently aligned (*e.g.* vertically aligned carbon nanotubes).

A. Mehdi *et al.* [8] did a comparative study between the use of single walled and multi-walled carbon nanotubes in fuel cells. They first modified the surfaces of commercial CNTs by functionalizing them with carboxyl functional groups. This was done by refluxing the carbon nanotubes with concetrated sulphuric acid at 413 K for 10 h, followed by cooling, centrifuging, washing with deionized water and drying. Platinum catalyst particles were then supported on both types of nanotubes. Accelerated durability tests (ADT) were then carried out to compare the durability of both the catalyst support systems. For this purpose, the assembled electrode systems were placed in ADT cells with liquid sulphuric acid as the electrolyte instead of solid Nafion polymeric membrane. Since the entire catalyst surface is in contact with the electrolyte, the entire surface is active. In the case of Nafion electrolyte, only the part of the catalyst in contact with Nafion is active. Thus accelerated degradation of the electrode assembly takes place. The results of the tests indicated that the rate of platinum detachment from the catalyst support was greater for single-walled nanotubes. Potential cycling beyond a certain limit revealed that single-walled nanotubes experienced 19 % greater reduction in electrochemical surface area than multi-walled nanotubes. Since a larger electrochemical surface area is always desirable, it can be concluded that multi-walled nanotubes should be preferred to single-walled ones for use in fuel cells.

Another interesting observation is that functionalized carbon nanotubes are better catalyst supports compared to the commercially used ones. The attached functional groups provide a larger active surface area for the platinum particles to anchor themselves to [8].

Further improvement of the properties of multi-walled carbon nanotubes is of primary importance to produce better quality catalyst supports for proton exchange membrance fuel cells. M. Shichun *et al.* [9] synthesized heat treated multi-walled carbon nanotubes as catalyst support. The nanotubes were heat treated in ammonia atmosphere up to temperatures as high as 1273 K. This treatment improved their resistance to electrochemical oxidation, thereby reducing the occurrence of carbon corrosion. The dispersion of platinum is also improved, leading to less agglomeration as compared to that on commercial multi-walled nanotubes. It is believed that the reduction of carbon corrosion is due to incorporation of nitrogen atoms on the nanotube walls at high temperatures, thereby reducing the occurrence of surface oxides on the wall surfaces. Thus, heat treated multi-walled nanotubes can also be considered as an improvement on the current CNT catalyst supports.

It can be also noted that nitrogen doped nanotubes can entirely replace the catalyst support system, *i.e.* they can eliminate the use of platinum catalyst entirely. This would cut down the cost of these fuel cells.

Now let us consider another carbon nanotube that has great potential as a catalyst support. One of the main objectives of the research on CNTs as catalyst support is to reduce the platinum loading. Vertically aligned carbon nanotubes should also be considered as a candidate for catalyst support. Their highly ordered structure results in the reduction of transport length, thereby improving the proton transfer capacity and electron conductivity between the electrodes. Platinum nanoparticles can be coated on the nanotubes by various means of sputtering deposition. N. Soin *et al.* [10], as well as Yue Shen *et al.* carried this out using DC sputtering under Ar at the base pressure of around $9 \cdot 10^{-7}$ mbar.

In the resulting catalyst support system platinum dissolution is reduced due to the strong bonding between platinum film and the nanotube wall. Moreover, while comparing the performance of Pt/VACNT with Pt/commercial CNT (Table 5), it was found that the former had a higher power density for less platinum utilization. Vertically aligned CNTs as the catalyst support resulted in a power density of 697 mW/cm^2 for Pt utilisation of 0.30 g/kW. The maximum power density and the corresponding Pt utilization of the cell with commercial catalyst are 525 mW/cm^2 and 0.38 g/kW, respectively.

The nitrogen doped carbon nanotubes mentioned earlier can also be vertically aligned, forming an excellent metal-free catalyst system on their own.

2. Experimental

From our overview on the carbon nanotubes commonly used, we can conclude that multi-walled, vertically aligned and preferentially functionalized carbon nanotubes have greater potential for use in fuel cells. However, it would be an interesting challenge to counter the limitations of single-walled nanotubes, and one such technique is their functionalization. Single-walled carbon nanotubes can be functionalized covalently as well as non-covalently.

Common functional groups attached to CNTs used in proton exchange membrane fuel cells include carboxyl groups. However, based on our findings regarding carbon nanotube functionalization, we would like to suggest a type of functionalized CNT that has recently gained attention for solar energy storage. These are azo-based carbon nanotube systems [12].

Azobenzene (2 phenyl rings linked by N–N double bond) or their derivatives can be chemisorbed to the CNT substrates with the help of amide linkers. The nanotubes help create a close-packed and ordered crystalline structure. However, these structures require the use of a solvent where the Azo/CNT structure can remain dissolved for greater stability.

3. Results and Discussion

The azobenzene structures, at exposure to sunlight get excited from *trans* to *cis* state. The resulting difference in energies between the ground state and excited state gets stored as chemical energy in the bonds (Fig. 7).

From the overview of the study, the advantages of the suggested CNT based catalyst support are as follows:

- Azo/CNT systems have an energy density of 690 wh/l (each CNT can support 4–8 azobenzene molecules around its circumference).
- Additional energy is stored in the form solar energy, and can be released by applying heat or using a catalyst [12]. Since one CNT can support 4–8 azobenzene molecules, total heat application for the *cis* to *trans* reaction would be within 373–473 K. Since proton exchange membrane fuel cells can operate at the temperatures up to 473 K, the resulting heat (producing the temperature of 620 K [12]) can be used by the compound to return to the ground state, releasing the stored solar energy which can be utilized for various purposes. Moreover, the excitation of the compound results in the release of electrons that contribute to the circuit flow outside.
- An ordered structure also leads to ease of manipulation of inter and intra molecular interactions, which in turn increases energy storage. Manipulations of molecular interactions include addition of polar functional groups to those portions of the molecules exposed to the solvent. This may have negligible effect on the ΔH and E_a value, but it increases the solubility of the nanostructures, ensuring greater stability of the crystalline structure [12]. For example, to increase the fuel efficiency, it is desirable to have both ΔH and E_a reasonably high. Substitution of hydroxyl groups for one or more hydrogen in the phenyl rings can ensure this due to the addition of more hydrogen bonds. The more the number of hydrogen bonds, the greater the stability of the molecule.

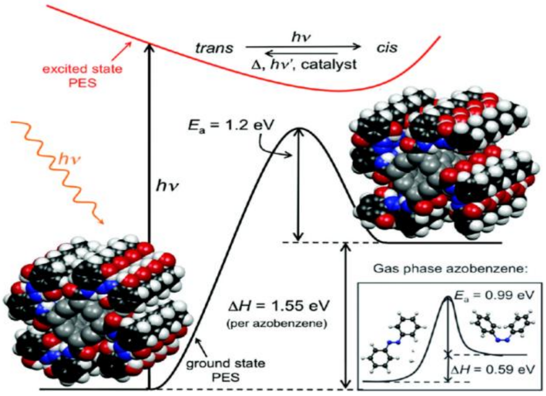

Fig. 7. Exciation of azobenzene molecules attached to CNT and the resulting energy stored compared with unattach azobenzene (bottom right corner) [12]

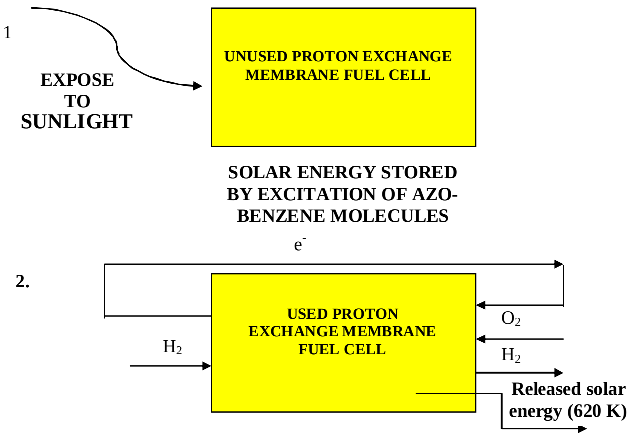

Fig. 8. Proposed mechanism of modified PEMFC

The disadvantages of these azo-based systems are the followings [2, 12]:

Requirement of a suitable solvent to maintain the CNT/Azo structures in proton exchange membrane fuel cells require the use of solid polymeric Nafion electrolyte. However, instead of Nafion, a liquid electrolyte can be used. Liquid electrolytes are not completely unheard of in these fuel cells. Many such liquids are used as electrolytes instead of Nafion for accelerated durability tests of these fuel cells. Less severe and corrosive liquids could be utilized. A solvent may be better but not entirely compulsory. If, however, it is used, the requirement of platinum as catalyst can be eliminated. Doping these structures with nitrogen may serve as a better option as a catalyst support system.

4. Conclusions

The use of azobenzene based carbon nanotubes for chemical storage is still in its incipient stages, but they can be considered as a possible alternative catalyst support, which utilizes two green energy sources, namely hydrogen and solar energy. Its application in proton exchange membrane fuel cells may result in a slight modification of their normal functioning, but the advantageous properties of this special type of carbon nanotube that allow it to store and convert solar energy to chemical energy makes it a strong candidate as a multi-tasking catalyst support in PEMFCs.

Not only has this material proven to be feasible for energy storage by utilizing two forms of clean energy, azobenzene based carbon nanotubes display immense possibilities of increasing the impact of fuel cells in the energy market.

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ПОСТУП У ЗАСТОСУВАННІ ВУГЛЕЦЕВИХ НАНОТРУБОК В ПРОГРЕСИВНХ ПАЛИВНИХ ЕЛЕМЕНТАХ

Анотація. *В роботі показано застосування вуглецевих нанотрубок для підвищення ефективності паливних елементів з протонно-обмінною мембраною. Проведено дослідження матеріалу, який може знайти потенційне застосування в паливному елементі як альтернатива існуючим на сьогодні вуглецевим нанотрубкам.*

Ключові слова: *вуглецеві нанотрубки, носій каталізатора, паливні елементи з протонно-обмінною мембраною, азо-бензол вуглецеві нанотрубки.*