ACTIVATED CARBON AS AN INEXPENSIVE ALTERNATIVE FOR COUNTER ELECTRODE IN DYE SENSITIZED SOLAR CELLS

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The following faculty members have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirement for the degree of Master of Science, with a major in Mechanical Engineering.

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Wei Wei, Committee Chair

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Yang-seon Kim, Committee Member

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Wujun Si, Committee Member

DEDICATION

To my parents, friends and family

and

To my advisor, Dr. Wei Wei

ACKNOWLEDGMENTS

I would like to express my gratitude for Dr. Wei Wei for being my advisor and for her constant support, encouragement and ingenuity. This research work wouldn't have been possible without her apportioning of knowledge on the topic. I would also like to sincerely thank Dr. Yang-seon Kim and Dr. Wujun Si for agreeing to serve on my examination committee and providing valuable insights.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES (continued)

Figures Page

LIST OF FIGURES (continued)

Figures Page

LIST OF FIGURES (continued)

Figures Page

ABSTRACT

Dye Sensitized Solar Cells (DSSCs) have attracted a lot of attention from solar industries because of the low cost and high efficiency to convert energy from photo-to-electric. DSSCs mainly consist three parts, photoelectrodes (a dye-sensitized nanocrystalline semiconductor), electrolyte with redox couple and a counter-electrode. In this thesis report, a literature review was first provided to establish the background of my research. Then in the experimental section, activated carbons with KOH and microwave post-treatments were employed as counter electrodes for DSSCs. Finally, in the results and discussion section, the I-V, CV, and EIS measurement results confirm that the activated carbon counter electrode with KOH post-treatment showed higher power conversion efficiency than Pt counter electrode. This would provide a promising approach to lower the fabrication cost of DSSCs.

CHAPTER 1

INTRODUCTION

With the increasing demand of the energy and depleting fossil fuels developing a new renewable energy source is the most important challenge we all are facing today. Solar is one of those renewable energy sources which is widely available almost everywhere in the world. DSSC provides an alternative to current p-n junction photovoltaic cells in a more affordable and more reliable way[1, 2]. Biggest hinderance for widespread use of solar is the cost of electricity produce from it. Present day solar cells use ultra-pure silicon for their working which requires large amount of energy for its production making it costly to afford[3]. DSSC is a photovoltaic cell whose functioning is completely different than solid state solar cells. In the photovoltaic cell positive charge generate at the semiconductor surface of conductive photoelectrode due to sunlight absorption which then transferred through ionically conducting electrolyte to suitable counter electrode which will efficiently transfer the electrons from external circuit to electrolyte and work as a good catalyst for the redox reaction of the electrolyte[4]. Theoretically, maximum potential difference can be obtained by difference between the redox potential of counter electrode and fermi level of the semiconductor on a photoelectrode at open circuit[4]. However, the CE potential is always lower than theoretical value because of different losses like mass transfer, current transfer through electrodes to electrolyte interface[4]. Pt has always considered as standard material for using counter electrode because of its good electrocatalytic activity, high stability and good conductivity [4-6]. For a photoelectrode, choice of semiconductor oxide material is TiO_2 and also ZnO, Nb_2O_5 are explored[1]. We can further reduce the cost of the DSSC by using material cheaper than Pt and more superior in properties like catalytic activity and conductivity. Different attempts have been made to reduce the cost through using materials like Graphene[7-9], Tungsten[10-17]

and Molybdenum sulfide[9, 18-25]. Among these all other low-cost materials mesoporous material got much attention because of its very large surface area and narrow pore size diameter. Carbon was not only tested in different forms but also tested in a composite form with other materials like Pt[26], Tungsten[27, 28] and MoS_2[29, 30] to increase the performance. Michael Grätzel is the considered as a father of this technology[2]. For the photo-electrode in standard we use a TiO_2 material because it is non-toxic, low cost and biocompatible. It is so widely available that we can find it in a domestic application like paint pigment. Although the certified efficiency so far achieved is 10% it is subjective to the solar illumination, roughness of counter electrode, photophysics and many more[1]. Increased efficiency of cell using AC as counter electrode is due to the fact of large increment in area which provides huge carbon conductive matrix results in high charge storage and high electron transfer[31].

1.1 Photo Electrode

The photoelectrode is a mesoporous oxide layer composed of nanometer-sized particles with a monolayer of dye attached to the surface, which is responsible for light absorption. It is an important component in DSSC, as it converts photons into electrical energy. It can strongly influence the photovoltage, the fill factor, and the photon-current conversion efficiency (IPCE). The process can be described as follows: when exposed to sunlight, the electrons of the dye are first excited from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO). Then they are transferred to the semiconductor's conduction band and passed from the semiconductor network, being accumulated at the transparent conducting oxide. An electrolyte solution which typically consists of an iodide/triiodide redox couple donates electrons to regenerate the oxidized dye. At last, the electrons transport to the counter electrode through the external circuit and reduce the triiodide ions back to the iodide to complete the process.

A good photoelectrode is supposed to provide good electron injection, good electron collection, and good usage of light. Many different materials have been explored as photoelectrode in DSSCs. Among them, titanium dioxide (TiO_2) powder is most abundantly used because of the low recombination rate for the hole-electron pair and great absorption property. Moreover, ZnO which has a very high electron movability has also gathered so much attention to be employed as photoelectrode materials in DSSCs[32].

1.2 Counter Electrode

The counter electrode is a very important part of DSSC. Generally, counter electrodes are made of a material based on metals and alloys, transparency and flexibility, transition metal compounds, carbon materials, conductive polymers, and hybrids, respectively. Counter electrode has two very important roles which are transferring charge in open circuit and working as a good catalyst for redox reaction of electrolyte (I^-_3/I^-) in DSSC[33]. That's why counter electrodes required to have two properties, one it should have high conductivity for electron transfer, and another is an excellent catalytic activity for reduction of electrolyte (I^-_3/I^-). High exchange of current can be achieved by increasing surface area which increases by increasing porosity of CE material[34]. That's why the counter electrode helps in increasing efficiency if they have mesoporous structure[35]. Because of this reason carbon materials are attracting much attention in DSSCs nowadays. Pt CE does good in both qualities, but Pt is costly element because of its limited resources and also Pt shows very low chemical stability in corrosive redox I^-/I^-_3 environment[35]. So, as we want DSSCs to be cheaper using Pt as a counter element is not so advisable. It's not necessarily possible to have both qualities in one material so that's why many researchers have combined various materials with required properties and used them as counter electrode[8-10, 13, 17, 18, 21, 24, 26-30, 33, 34, 36-44]. For example, graphene has very less charge transfer resistance

so when it is used in Pt CE DSSCs, it works well to transfer electrons from FTO rod to Pt and improves the electrocatalytic activity when placed between Pt and electrolyte[45]. This is how we can add different materials and use their synergetic effect to increase the efficiency of DSSC exponentially. The whole point of making DSSC is reducing the cost of solar cells so obviously, the alternating materials for CE must be the cheapest and widely available like carbon. An extensive research is going on to find that alternative material for a counter electrode which has power conversion efficiency (PCE) equal to or more than PCE of Pt counter electrode and some of these materials have shown excellent efficiency. Various metal oxides, metal sulfides, carbon, nitrides, and polymers have been explored as a counter electrode. Graphene, MoS_2, and WS_2 are also explored as a counter electrode and showed really good results. Every material has unique properties and those can be efficiently used in DSSCs.

1.3 Electrolyte

Electrolyte is responsible for transferring the charge between electrodes inside the DSSC. For the successful redox reaction electrolyte needs to reduce the dye cations before they can recombine the photoelectrode electrons and while doing that electrolyte should not intercept photoelectrode electrons. So, this is kind of challenging requirement electrolyte has to fulfill. The iodine/triiodide is the most common electrolyte used in for all DSSC. Iodine/triiodide electrolyte can go deep inside of the semiconductor layers and keep serving the charge to reduce the dye solution. However, iodine/triiodide is not the best electrolyte available because it has some disadvantages which are still not been able overcome till now. Iodine/triiodide electrolyte tend to form other polyiodides such as I_5^-, I_7^-, etc. These polyiodides absorb lot of visible light which reduces the efficiency of cell. There are some other electrolytes available which have been explored little bit such as Transition metal mediators of copper complexes, ferrocene/ferrocene[46].

CHAPTER 2

LITERATURE REVIEW

There are so many researchers working on DSSC to bring down the cost and increase the efficiency. They had prepared different compositions and methods to manufacture the carbon induced counter electrode. Other than Activated carbon various forms of carbon have also been explored such, Graphene, Carbon nanotubes, Mesoporous carbon and many more. Carbon materials have many advantages over other materials because of its structure. Graphitic carbon has 2 different structural regions one is called basal plane and the other one is edge plane[47]. Basal plane has a high conductivity which helps in charge transfer and edge plane has more defect sites, so it helps in high catalytic activity. That's why carbon materials have excellent potential to be a good CE. Also, manufacturing process for carbon usually involves the low temperature processes therefore cost of manufacturing carbon material is lower than the Pt.

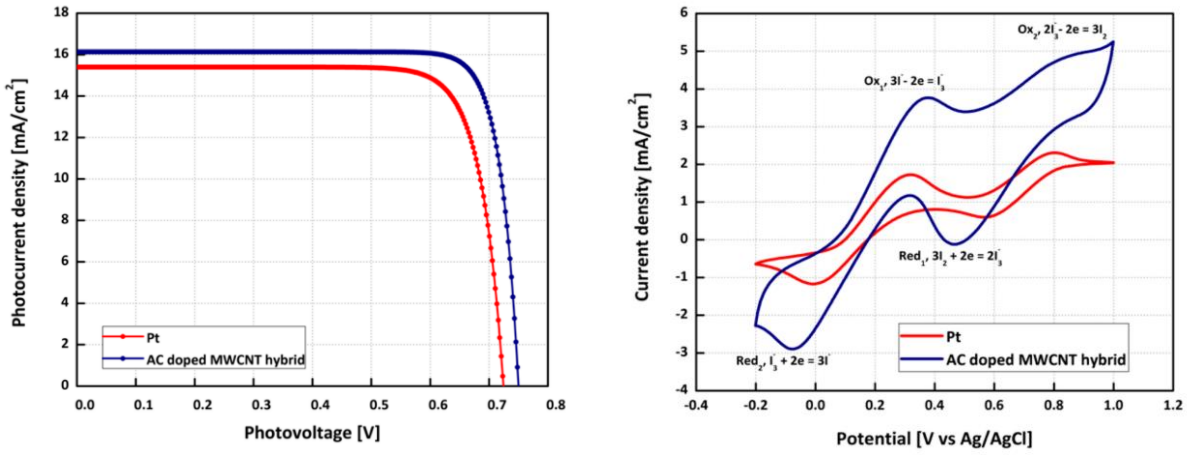
Some of the examples for graphitic carbon is Carbon nanoparticles and carbon nanopowder. They have a spherical shape and high surface area. Usual size of carbon nanoparticles is 10 to 45 nm and the area ranges from 30 to 50 m^2g^{-1} whereas for nanopowders size is 75 to 100 nm and the area is 2 to 10 m^2g^{-1}[47]. For activated carbon surface adsorption is high because of its high micro-porosity. It has rough surface consisting small pores distributed providing high surface area for the gas adsorption. Because of high number of pored and rough surface it also have high number edge defects which increases catalytic activity of the CE[47].

Graphene is a semi-metal material which has zero band gap [48]. It had been unintentionally produced in small quantities for centuries, through the use of pencils and other similar graphite applications. It was originally observed in the electron microscope in 1962. Scattering graphite in

a fluid medium can create graphene by sonication took after by centrifugation. Graphite particles can be eroded in liquid salts to frame an assortment of carbon nanostructures including graphene. Graphene is highlighted for its two-dimensional (2D) planar structure, it has atomically thick sp2 carbon atoms which are arranged in honeycomb-like hexagonal lattice structure[43]. It exhibits the extraordinary properties of large specific surface area and high conductivity[33]. Graphene has so many unique physiochemical properties like high electron mobility, high mechanical strength. Another unique property graphene has is that it can repair holes in the sheet by itself when it is exposed to the molecules containing carbon atoms. Carbon atoms align themselves in a hexagonal lattice perfectly. The Graphene has lamellar structure and high conductivity which when introduced to the MoS2 can help to lower the charge-transfer resistance and increase the photovoltaic efficiency of DSSCs (5.98%)[29]. Graphite paper which is another form of graphene is very lightweight and it has a flexible structure which is good for mass production and to use in DSSCs[21]. Reduced graphene oxide (RGO) is an intermediate stage of graphene oxide and graphene and it has various type of oxygen-containing functional group like (−OH, =O, −COOH) and lattice surface defect which helps in the electrocatalytic site in metal nanoparticles. This why RGO are considered much better than the fully reduced defect-free graphene[8]. Apart from all this carbon-based counter electrode have some drawbacks like these materials require large dosage in order to attain the targeted catalytic activity[43]. Graphene has one environmental issue has which is its toxicity. The toxicity of graphene relies upon its shape, purity, size, after creation handling steps, oxidative state, functional groups, amalgamation techniques, dispersion state, course, measurements of organization and exposure times.

2.1 Activated Carbon as CE

Alvira Ayoub Arbab and his coworkers worked on the activated-charcoal-doped multiwalled carbon nanotube (MWCNT) material to use as CE[31]. MWCNT's have a unique characteristic such as high stability, high electrical conductivity but they lack in electrocatalytic activity because of their defect free surfaces. Alvira Ayoub Arbab with his team doped the MWCNT's with the Activated Carbon to create the defects on the surface of the MWCNT's. First, they prepare lipase enzymes in ethanol. 0.4 g of MWCNT was then mixed in lipase solution and stirred for 8 hours at room temperature. Well wrapped lipase solution around the CNT's reduce the distance between the CNT's considerably. Before adding Activated carbon to MWCNT's, it was mixed with 50 mL of ethanol and kept in ultrasonic for 3hours. After that it was added in MWCNT's and kept at stirring for 8 hours in ultrasonic at the room temperature. Concentrated carbon mixed ethanol then taken out using vacuum filter method. 0.5μm pore size PTFE filter was used then washed with deionized water twice. After washing of the sample 15 mL of the carboxymethyl cellulose polymeric binder solution was mixed with carbon cake. After that sample was stored overnight to get a good consistency. Paste was applied on the FTO glass through facile tape casting method under drying air. Thickness of the sample was maintained 3M. Then they dried carbon CE samples at 150^{O}C for 15 minutes and annealed for 300^{O}C for 30 minutes. Electrolyte used is iodine/iodide redox solution. TiO_2 semiconductor oxide photoelectrode is used. They got efficiency of 10.05% with AC doped MWCNT as CE while using Pt CE efficiency was only 9.3% as shown in Figure 1(a) below.



(a) (b)

Figure 1- (a) Shows the IV performance curve for the Pt CE DSSC and AC doped MWCNT CE DSSC. (b) Shows the CV curves for the Pt CE and AC doped MWCNT CE DSSC[31].

Vorrada Loryuenyong and Achanai Buasri worked on activated carbon CE as well[49]. They used coconut shell (CS) to prepare activated carbon. Using Perkin Elmer Series II 2400 they analyzed that CS contents 46.56% Carbon in it. Apart from the carbon they found out CS also contents hydrogen and nitrogen. So first they grinded CS with hammer and ball milling to convert it into powder form. Powder was filtered through a mesh of 75 μm. For chemical activation K_2CO_3 was first mixed with the CS powder in the weight ratio of 1:2 for 24 hours. Then this mixture of K_2CO_3 and CS was mixed with $Fe(NO_3)_3 \cdot 9H_2O$ in ethanol with various mixture ratios like 1:0, 1:4, 1:1, 4:1. Then using the pressure vacuum sintering they heated the samples at 900^{O}C with the ramp rate of 10 ^{O}C/min for 30 minutes. Samples were then washed with 20% wt. solution of HCl followed by deionized water to make pH of sample 7. Finally, samples were dried at 60 ^{O}C for 24 hours. When they used these materials as counter electrode they got a maximum efficiency with 4:1 ration sample. The maximum efficiency they achieved was 1.24%.

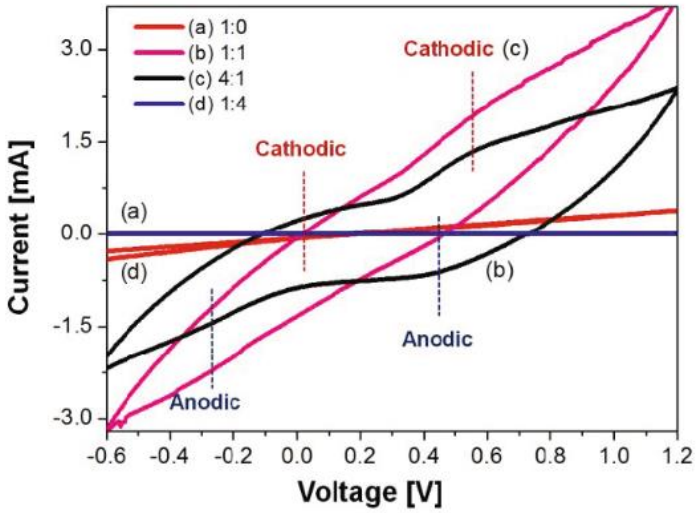
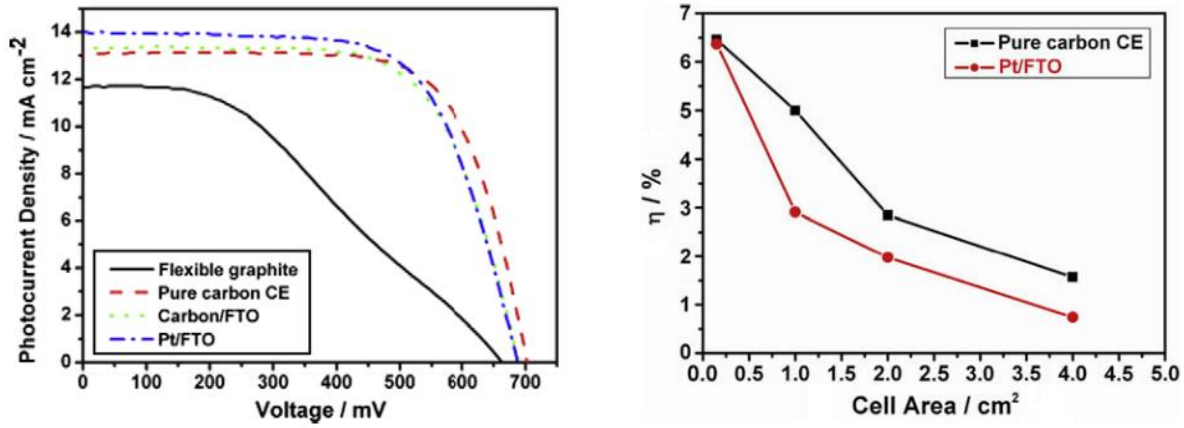


Figure 2- Cyclic Voltammetry Curve[49].

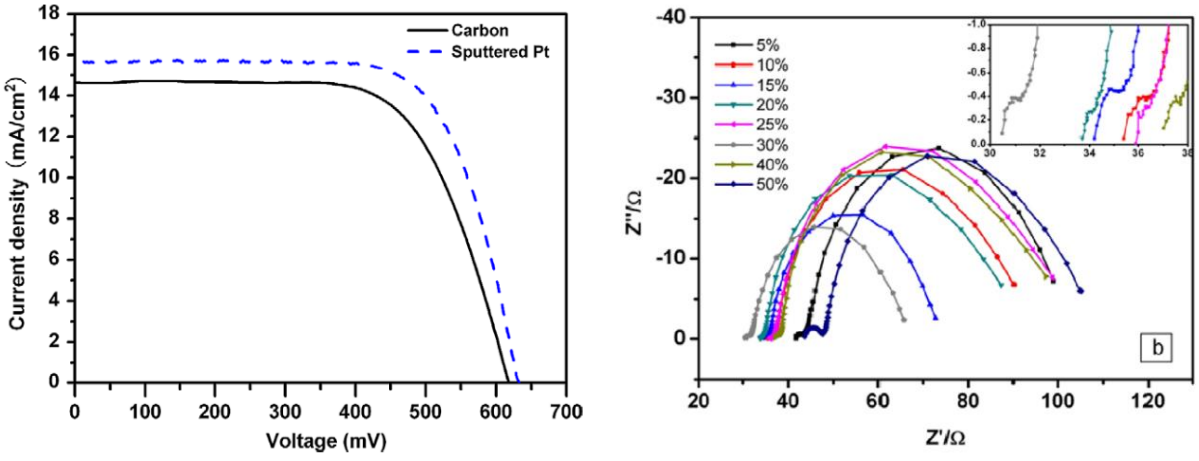
Jikun Chen with his research group worked on the similar research where they combined flexible graphene and activated carbon to make a CE[50]. For preparation of carbon CE they first cleaned graphite sheets with ethanol, water and air-dried. 0.4 g activated carbon (Brunauer–Emmett– Teller (BET) surface area 1958 m^2g^{-1}), 0.1 g carbon black (BET surface area 77 m^2g^{-1}), 4 g terpineol and 0.1 g SnO_2 nanoparticle were mixed together to make a CE material paste. Mixture was ball milled for 5 hours to mix it well. Then that paste was applied on cleaned graphite sheets and then on FTO glass by doctor blading to make a CE. For 60 minutes the layer was left for drying and sintering. Pt CE was also prepared for comparison the performances. H_2PtCl_6 was used to prepare the Pt CE in 30 mM isopropanol. Thermal decomposition was done at 385ºC for 30 min. Electrolyte and photoelectrode used were the same as used previously in other research, Iodine redox solution and TiO_2 metal oxide photoelectrode. After the test they found out performance of Carbon CE was comparatively little better than Pt CE. Efficiency of the DSSC with the Pt CE was 6.37% whereas efficiency with carbon CE was 6.46%.



(a) (b)

Figure 3- (a) Shows the IV performance curve for the Pt CE DSSC, Pure carbon CE DSSC, Carbon CE DSSC and flexible graphite. (b) the plots of n% against cell active area for DSCs with pure carbon CEs and Pt/FTO electrodes[50].

Kexin Li with his research group made a Carbon CE with low temperature fabrication technique[51]. They also prepared the CE material almost similarly as Jikun Chen's research. They mixed 0.4 g of activated carbon and 0.1 g of carbon black. They added 4 mL of 2M $SnCl_4$ to make a solution aqueous. The aqueous solution was then ball milled to mix well for 5 hours. The aqueous solution converts into gel form overnight. Then using doctor blade method gel is applied on to the FTO glass. To dry the gel layer, they were kept at 120ºC for 1 hour. Thickness of the layer was kept around 10 µm. Carbon particles size of 30 nm were added to into usual 5 µm particle size carbon material to increase the conductivity of the material. Electrolyte and photoelectrode used were the same as used previously in other research, Iodine redox solution and TiO_2 metal oxide photoelectrode. From the test they figure out that SnO_2 helps in forming uniform layer on FTO which results in high conductivity. Until the SnO_2 content goes up to 25% the conductivity increases but after that it start decreasing. They achieved maximum efficiency of 6.1% with this carbon CE which is comparable to the Pt CE with 7% efficiency.



(a) (b)

Figure 4- (a) Photocurrent–voltage characteristics of the cells using carbon counter electrode fabricated at 120 °C (black solid line) and sputtered Pt electrode. (b) Nyquist plots of DSCs using carbon counter elelctrodes with different SnO_2 content. The inset shows the Nyquist plots in high frequency region[51].

Zaky Mubarak in his research compare the performances of AC CE and Nanopowder CE in DSSC[52]. They used the monolithic design to prepare the DSSC. Monolithic structure DSSC is prepared layer by layer and it also eliminates one conductive substrate. Initially they made structural design of monolithic DSSC using CorelDraw X7 and printed on transparent plastic sheet. For making of the DSSC in monolithic way they prepared screen mesh. For printing a carbon layer, they prepared a screen mesh of the stainless steel and for printing a ZrO_2 and TiO_2 layers they prepared nylon-based screen mesh. Screen mesh was cleaned using stencil remover paste and fabric abrader & degreaser. One side of the screen mesh was then applied with a photographic film while other side was with mixture of screen emulsion and sensitizer. Then a design on the plastic sheet was transferred to photographic film. Plastic film was removed by exposing the screen to the UV beam for 5 minutes. After that FTO glasses were prepared for next process. FTO side of glasses were sanded first where the FTO layer was removed. FTO glasses washed nicely then with various chemicals such as acetone, soapy water, etc. in ultrasonic cleaner bath. For making the paste of

carbon, carbon nanopowder, graphite powder, ethyl-cellulose (Merck), TiO_2 powder (P25, Degussa, Germany) and α-terpineol (Aldrich) were grinded together. Similarly, for the paste of activated carbon, instead of carbon nanopowder they added activated carbon keeping all other materials similar and grinded them. Both of the pastes were grinded in mortar and pestle for 2 hours to make them smooth. 2 layers of TiO_2 paste was applied on FTO glass and dried at 120^OC for 10 minutes followed by sintering at 500^OC for 40 minutes. Then for annealing these layers were immersed in a $TiCl_4$ for 30 minutes at 70^OC. TiO_2 layers also helps in absorbing more light. After a TiO_2 layers Zr-Nanoxide paste was printed similarly as TiO_2 only except the sintering temperature for Zr is used as 400^OC. carbon paste was then printed on the ZrO_2 layer. For drying it was heated at 120^OC for 30 minutes and then sintered at 400^OC for 40 minutes. Then a complete sample was soaked in a dye solution of Z907 Ru-dye, Solaronix 0.25 mM of ethanol in dark place for 20 hours. Then sample was assembled with another glass with thermoplastic and pressed for 30s at 120^OC. Electrolyte was put inside after all this from the gap between two glasses. In the characterization they found out that activated carbon layer had smaller average thickness than the carbon nanopowder. They found out that resistance of the activated carbon layer was little less than the resistance of the carbon nanopowder. With the four points probe method the resistance of activated carbon layer and carbon nanopowder they obtained around 10.70 Ω/sq and 11.09 Ω/sq respectively.

Another group from IIT Kanpur, India worked on this topic was Rahul Gupta, Rudra Kumar, Ashutosh Sharma and Nishith Verma[53]. In their research they synthesis Carbon Nanofibres on Surface of Activated Carbon Fiber (ACF) and transition metal Cu. They used composite of the Cu and CNF as CE for DSSC. There were various steps involved in a making of Cu-PACF/CNF (shown in figure.) like first they did some pretreatment on the ACF for removing unwanted ions

from the samples. They washed ACF sample for couple of times using the deionized water and then dried it by static air for 6 hours at room temperature, at 120^{O}C for 12 hours and vacuum drying for 12 hours at 200^{O}C. After that ACF was ball milled for 30 minutes to gets 10μm length of the ACF particles at 20-Hz frequency. For adding Cu into a PACFs $Cu(NO_3)_2 \cdot 3H_2O$ precursor was used. Cu precursor was added using 50 ml of deionized water and mixing it with magnetic stirrer. Nitrate salt was added to blockage of the pores of the PACFs. Then solution was filtered using a 42 mesh ashless filter paper and then dried in oven for 2 hours at 50^{O}C. After that solution was separated from the filter paper and then dried in the oven at 120^{O}C for 12 hours. Through calcination Copper Nitrate was dispersed on the PACF's surface at 200^{O}C for 4 hours in constant N_2 flow. Then the reduction was performed at 330^{O}C for 2 hours again using N_2 flow which helped to convert back the oxide of metal to metallic state again. Finally, CNF was grown on this Cu dispersed PACF substrate through the CVD method. CVD was performed under C_2H_2 gas flow at 300^{O}C for 1 hour. Then the standard photo electrode was prepared using the TiO_2 material. Iodine/Triiodide solution was prepared by adding 0.3M LiI, 0.05M I_2, 0.6M 1,2-dimethyl-3-methylimidazolium iodide, and 0.5M tert-butylpyridine to use as an electrolyte solution. After performing the performance test, they achieved the conversion efficiency of the 4.36% whereas with pure PACF CE 1.75% and with Cu-PACF 3.20%. Cu NPs helps in catalyzing the redox reaction of the electrolyte.

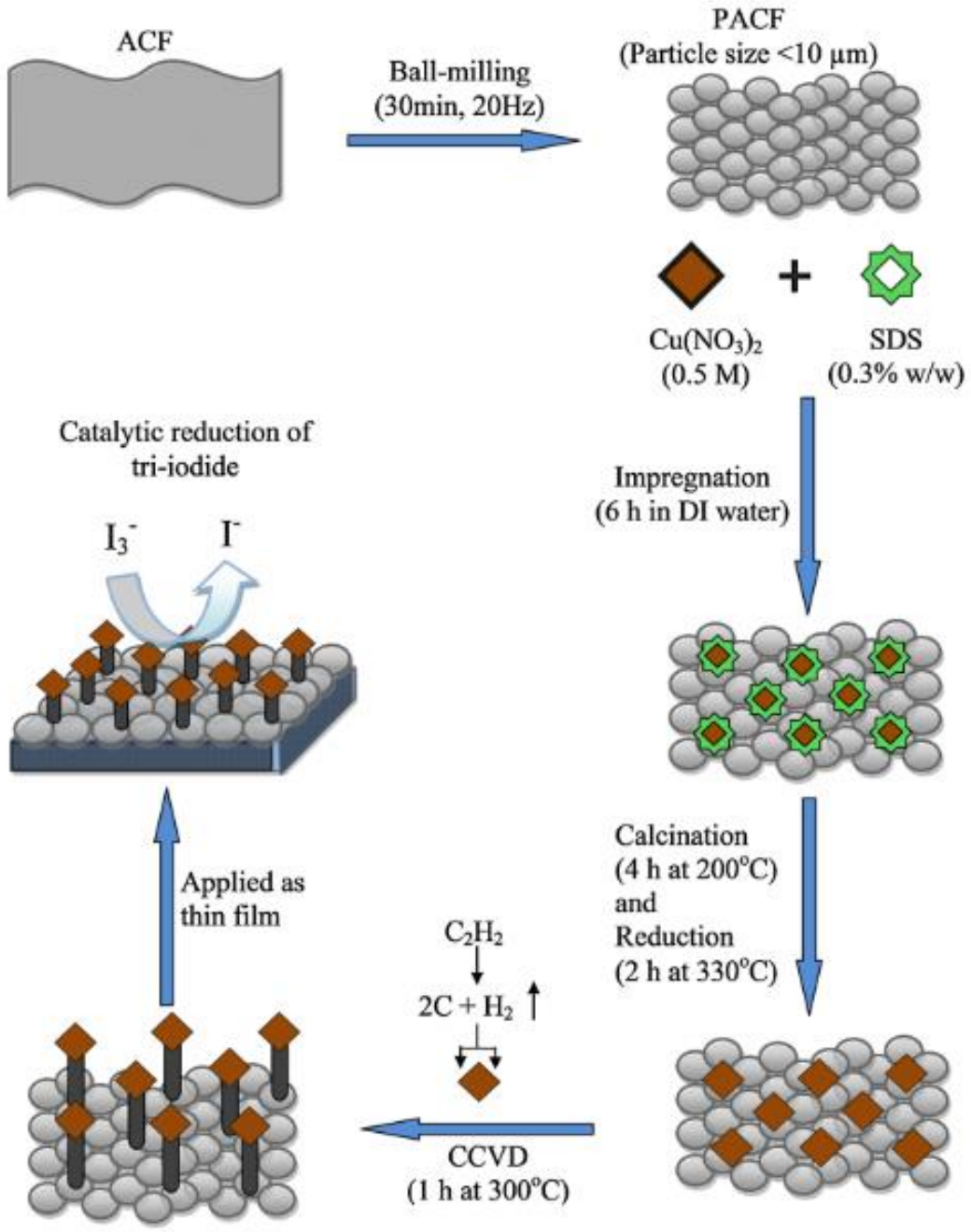
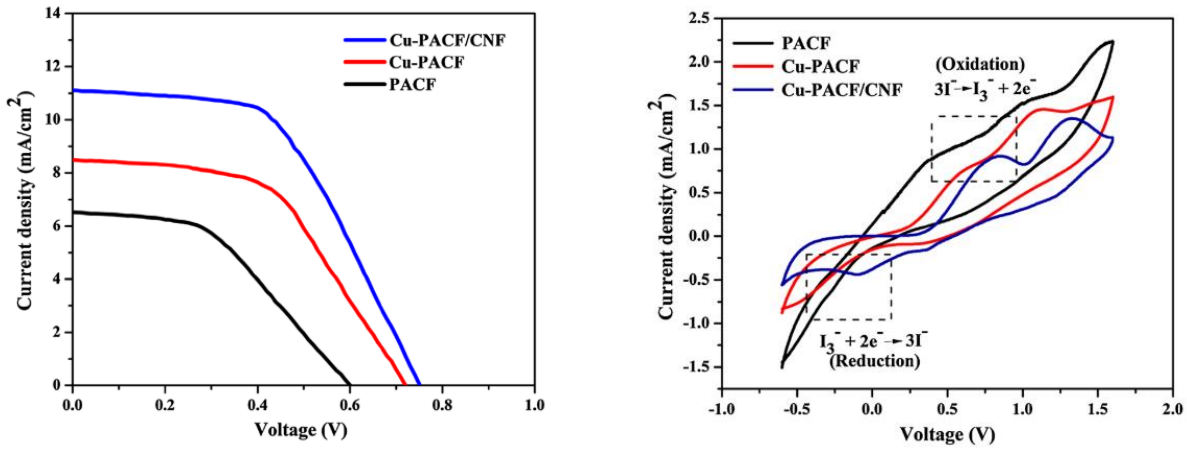


Figure 5: Schematic illustration showing the preparation of Cu-PACF/CNF and its catalytic activity[53].

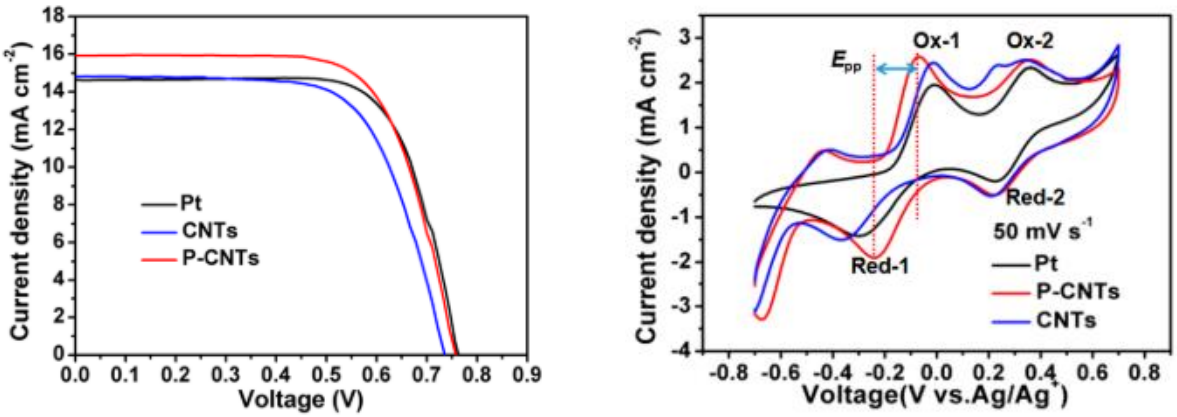


(a) (b)

Figure 6- (a) Current density–voltage characteristic of DSSCs with PACF, Cu-PACF, and Cu-PACF counter electrodes under illumination (I= 100mW cm^{-2}). (b) CVs of PACF, Cu-PACF, and Cu-PACF/CNF at a scan rate of 50 mV s^{-1}[53].

Activated Carbon nanotubes are also explored by Jiafu Hong and her research group as CE for DSSC[46]. They synthesis Heteroatom-doped micro-/nanostructured carbon materials which is activated carbon nanotubes (P-CNTs) and used it as CE for better performances of DSSC. For the preparation P-CNTs they used commercial CNTs (Shenzhen Nanotech Port Co.,Ltd., diameter: 40−60 nm, length: >5 μm). First, they treated that P-CNTs with using the plasma (commercial 13.56 MHz PCE-6 plasma system) with a power of 29.6 W and with a pressure of 50 Pa in oxygen atmosphere for various time slots (0, 1, 3, 5, and 30 min). P-CNTs also etched for 3 minutes. Carboxyethyl cellulose was mixed with this CNTs to make a paste with grinding. That slurry was then applied on the FTO glass using the doctor blade method and annealed for 500^{O}C for 30 minutes. Photoelectrode for this research used was TiO_2 photoelectrode immersed in N719 dye solution ethanol. Photo electrode and counter electrodes were hot pressed together and electrolyte was injected through predrilled holes of this cell. Various test including X-ray Diffraction, scanning electron microscopy and transmission electron microscopy, BET test, C-V test and I-V

test. In the performance test they find out that with P-CNTs as CE they achieved the efficiency of 8.35 which was even more than Pt as CE 8.04%. They also figure out that adding hydroxyl and carbonyl groups largely effect in reducing ionization energy for redox reaction of triiodide.



(a) (b)

Figure 7. (a) J−V curves (b) CV curves at a scanning rate of 50 mV s^{-1}[46]

Table 1-Photovoltaic Parameters of Various CEs[46].

samples	J_{sc} (mA cm^{-2})	V_{oc} (V)	FF (%)	η (%)
Pt	14.63 ± 0.16	0.76 ± 0.01	72.0 ± 0.8	8.04 ± 0.04
CNTs	14.80 ± 0.23	0.74 ± 0.01	66.9 ± 1.2	7.29 ± 0.06
Ar-P-CNTs	15.72 ± 0.19	0.74 ± 0.01	64.7 ± 0.8	7.52 ± 0.04
P-CNTs	15.92 ± 0.21	0.76 ± 0.02	69.2 ± 0.6	8.35 ± 0.03

2.2 Pure Graphene as CE

Yi Hu and his coworkers explored graphene in p-type DSSCs for the first time[54]. After illumination holes are injected from photoexcited dye to valence band of p-type semiconductor. NiO is used as photoelectrode in p-type DSSC. Electrons in dye are left to reduce the electrolyte. Iodide is used as electrolyte in this DSSCs. Valence band then migrating through external circuit comes graphene CE. Through CE then redox electrolyte is oxidized by valence band. This how

circuit completes in p-type DSSCs. GN films are coated on CE are about of 16 µm. They are stacked perpendicularly to form 3D network of graphene and giving more active site for reaction.

3D nano-foam of few-layer graphene(3D-NFG) has large surface area and high conductivity that's why it has a potential of being good CE for DSSC. Jung-Soo Lee and his coworkers explored 3D-NFG as CE for DSSC[55]. They found unique way to produce 3D-NFG on SiO_2/Si substrate by CVD without even using toxic carbonaceous gas. Source of carbon provided by carbonized-c which is created by pyrolysis in the environment of hydrogen gas. Through pyrolysis reduced nickel was also created which works as catalyst for graphene growth. First PVA-$NiCl_2$ solution with the thickness of 500 nm was spin-coated on SiO_2/Si substrate. There is electrostatic interaction between hydroxyl groups of PVA and nickel ions and that's why with the nickel precursor it forms homogeneous dispersion. SiO_2/Si substrate with that composite on it was then heated at 1000ºC in quartz tube. Composite was heated to induce carbonization of PVA which lead to formation of nano-foam because of removal of carbon materials. Not just for carbonization but also for triggering the reduction of Nickel ions (ı ı) into Nickel (0). After that for reinforcing the structure polymethyl methacrylate (PMMA) was spin coated on graphene/Ni nano-foam. In ammonium persulfate solution nickel was removed and PMMA was removed by dissolving composite in acetone for 4 hours. Finally, they got freestanding 3D-NFG CE. TiO_2 on FTO was used as photoanode. Solaronix Iodolyte AN-50 was injected into the cell as electrolyte. Efficiency shown by this DSSC is 5.2% which is very close to Pt CE DSSC.

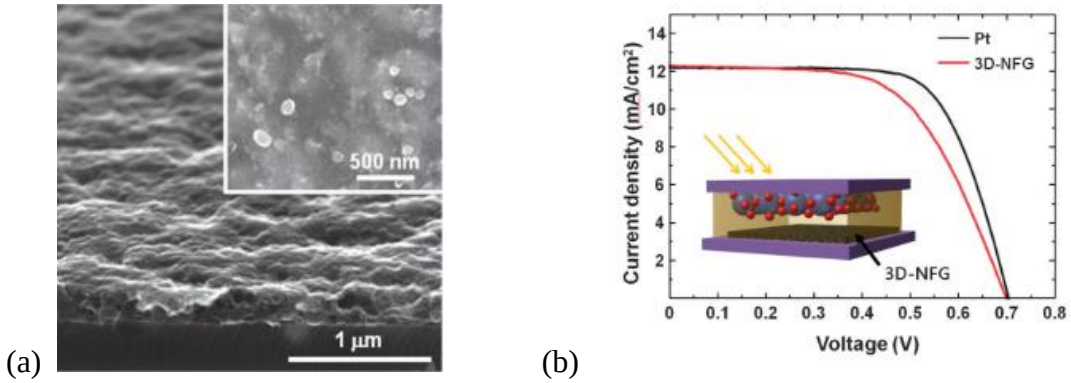


Figure 8-(a) SEM image of the 3D-NFG. (b) I–V curves for DSSCs using 3D-NFG CE and Pt CE[55].

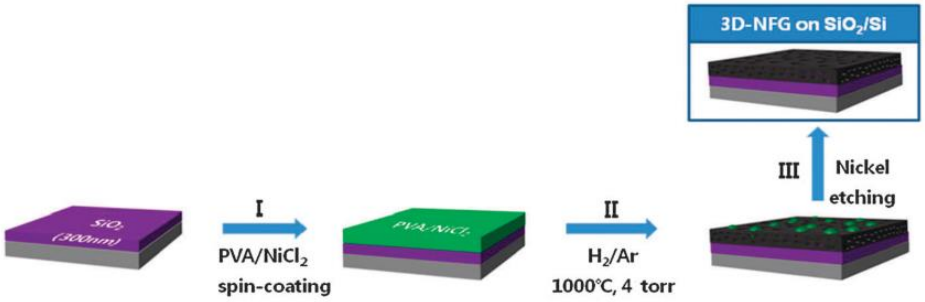


Figure 9- Schematic illustration of the fabrication process for 3D nano-foam of few-layer graphene (3D-NFG)[55].

Table 2- Photovoltaic characteristics of the DSSCs and EIS parameters of the symmetric cells with 3D-NFG CE or Pt CE[55].

	R_s/Ω	R_{ct}/Ω	V_{oc}/V	J_{sc}/mA cm^{-2}	FF (%)	η (%)
Pt	4.3	1.7	0.7	12.1	69.2	5.7
3D-NFG	5.0	11.47	0.71	12.2	60.0	5.2

Hui Wang with his team found novel method to synthesized 3D graphene which has structure like honeycomb[56]. They did a simple reaction between lithium oxide (Li_2O) and CO to form graphene.

$$Li_2O + 2CO \rightarrow C(\text{graphene}) + Li_2CO_3$$

In batch ceramic-tube reactor 1 mol of Li_2O from Aldrich was reacted with CO. Initial pressure maintained at 35 psi and at temperature of 550ºC for 12, 24 and 48 hours. The conversion of Li_2O for 12, 24 and 48 hours are 87, 90 and 92% respective. To remove Li_2O and Li_2CO_3 from products they were treated with hydrochloric acid and washed with H_2O then dried at 80ºC. This graphene has more conductivity than graphene synthesized by chemical exfoliation of graphite (CEG). Photoanode used for this DSSC is TiO_2 layer on FTO glass plate and I_3^-/I^- as electrolyte. Given DSSC with Pt CE gives result of 8% PCE whereas same DSSC with HSG-12h as CE shows PCE of 7.8%.

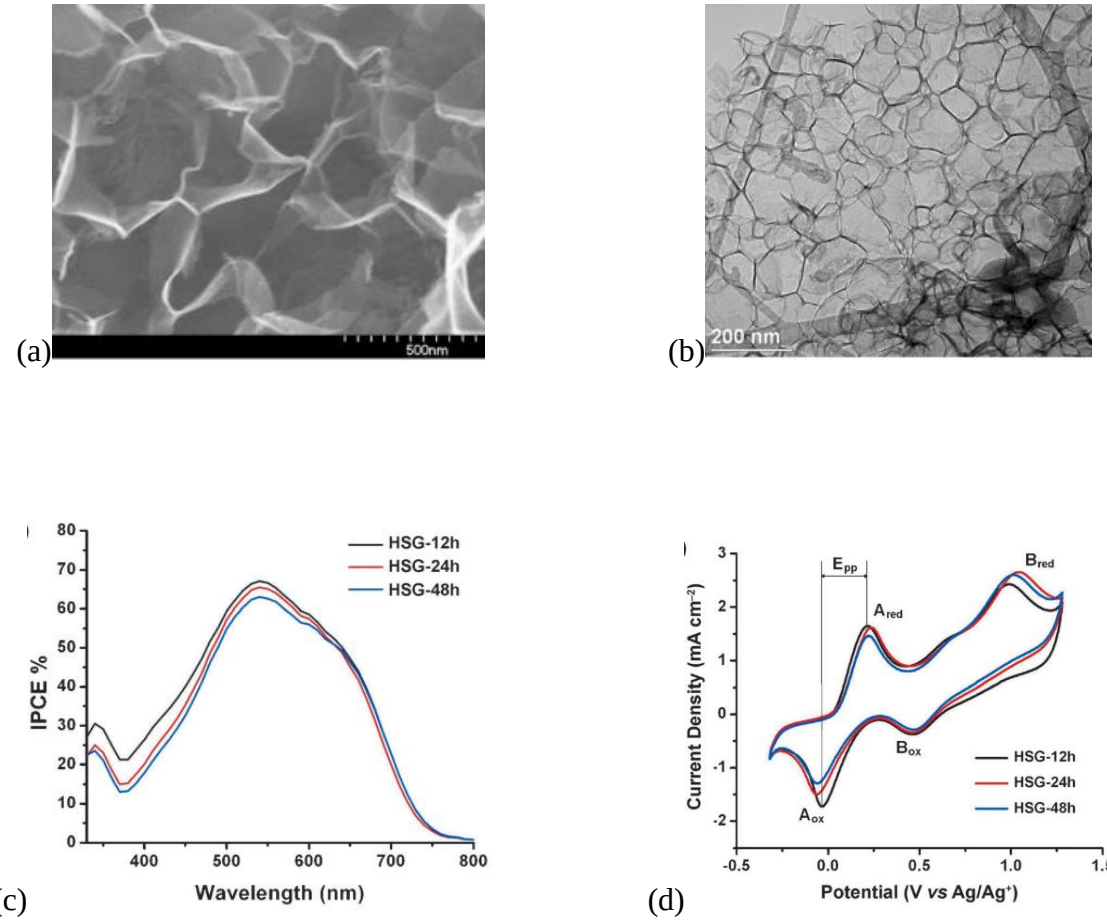


Figure 10. (a) FESEM image honeycomb-structured graphene (HFG). (b) TEM images of honeycomb-structured graphene (HSG). (c) IPCE of DSSCs with HSG counter electrodes. (d) CV curves of HSG electrodes[56].

Table 3- Photovoltaic performance and electrochemical characteristics of DSSCs[56].

CE[a]	J_{sc} [mA cm^2]	V_{oc} [V]	FF	η [%]	R_s [Ω]	R_{ct} [Ω]	Z_N [Ω]
HSG-12 h	27.2	0.773	0.371	7.80	24	20	220
HSG-24 h	26	0.774	0.325	6.53	25	35	265
HSG-48 h	26	0.773	0.314	6.30	24	45	310
CEG	6.48	0.785	0.127	0.64	27	2500	10^4

Similarly, Wei Wei also found another way to form 3D graphene reacting carbon dioxide (CO_2) with potassium (K)[57]. 3D graphene formed after this reaction is also honeycomb structure graphene (3DHG).

$$4K_{(l)} + 3CO_{2(g)} \rightarrow C(\text{graphene})_{(s)} + 2K_2CO_{3(s)}$$

They have further invented different 3D graphene structures like 3D cauliflower-fungus-like structured graphene and 3D crape myrtle flower-like structured graphene. Aldrich powder used as source potassium which was put into CO_2 environment at 50 psi pressure and room temperature into the ceramic tube. Later temperature is increase at the rate of 10ºC/min till 550ºC and remain same for selected time periods like 12, 24 and 48 hours. After that solid product is treated with 36.5% HCl and washed with DI for several times. For drying this solid product, it was put into the rotary water remover which rotates at 3600 rpm then kept at 80ºC overnight. 9 graphene layers synthesized through this method with one layer of thickness around 400 nm. To, make CE, this graphene powder was mixed up with alcohol to make paste and then deposited on FTO glass plate. TiO_2 photoelectrode and Iodide electrolyte was in this DSSC. All three samples of graphene showed good result. DSSC with 3DHG-24 as CE showed highest PCE which was 8.25%.

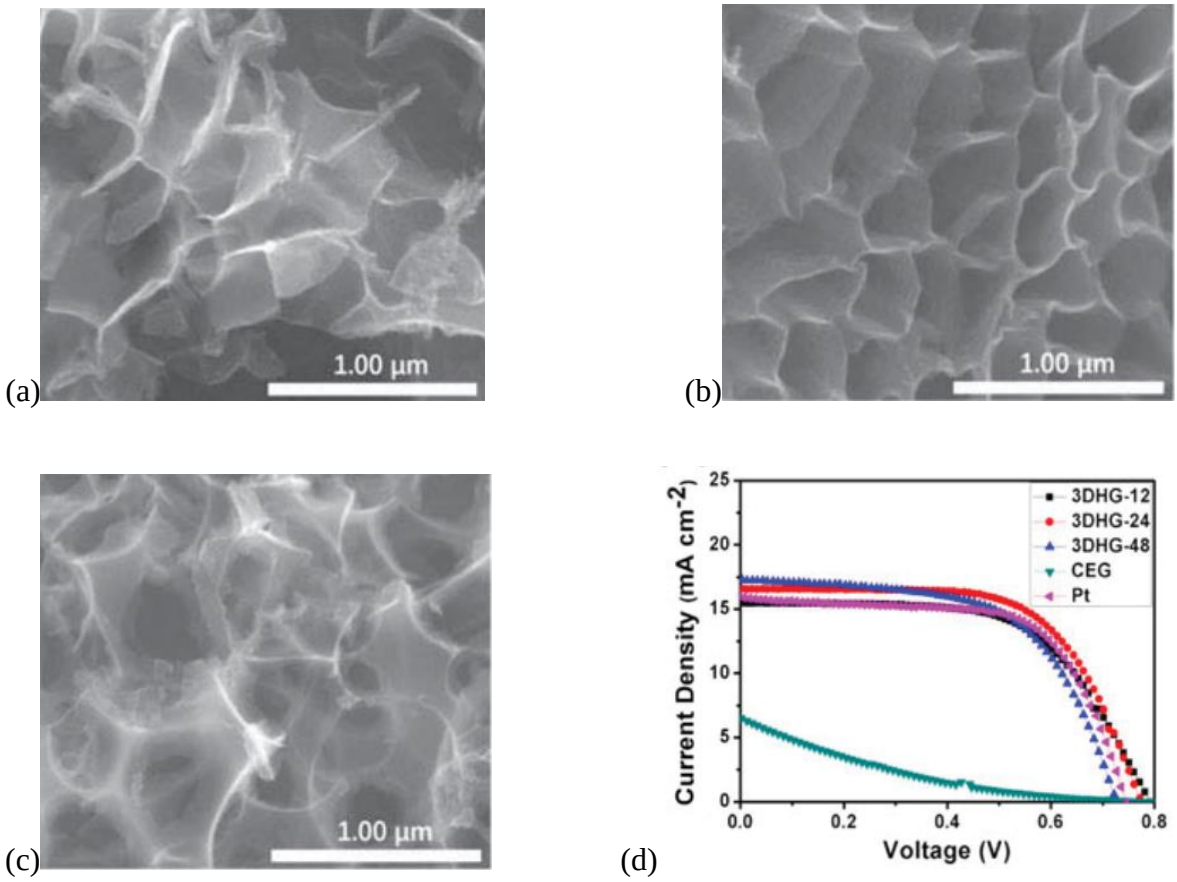


Figure 11. Image of graphene layer through field emission scanning electron microscope (a) 3DHG-12 (b) 3DHG-24 (c) 3DHG-48 (d) Characterization of dye-sensitized solar cells (DCCSs) I-V curves[57].

Table 4- Characteristics of dye-sensitized solar cells (DSSCs)[57].

Samples	V_{oc} (V)	I_{sc} (mA cm^{-2})	FF	Efficiency (%)	R_s (Ω)	R_{ct} (Ω)	Z_N (Ω)
3DHG-12	0.80	15.48	0.60	7.41	19	21	180
3DHG-24	0.78	16.60	0.64	8.25	19	17	150
3DHG-48	0.73	17.26	0.59	7.47	19	20	150

Wei Wei and her team also formed 3D cauliflower-fungus-like structured graphene (CFG) through somewhat similar reaction instead of using K they Li[58]. Graphite formation is avoided through simultaneous generation of Li_2CO_3.

$$4Li_{(l)} + 3CO_{2(g)} \rightarrow C(graphene)_{(s)} + 2Li_2CO_{3(s)}$$

Reaction has negative Gibbs energy making it thermodynamically favorable and exothermic as well. Reaction was carried out in ceramic tube at 500ºC to 650ºC. Products of reaction were confirmed by X-ray diffraction. Li_2CO_3 and Li_2O was removed by hydrochloric acid followed by DI wash and dried at 80ºC. From the observations of electron energy loss spectroscopy (EELS) structure of cauliflower-fungus-like graphene was confirmed. CFG samples prepared at 500, 550, 600, and 650ºC were denoted as CFG-500, CFG-550, CFG-600, and CFG-650, respectively. Heterogeneously distributed oxygen groups also observed in graphene structure. Oxygen group create defect site in graphene sheets because of which it reduces conductivity of graphene. That why less oxygen group in graphene is good for graphene to be CE. With increasing temperature of synthesis of graphene, oxygen content reduces. Efficiency of DSSC with CFG as CE is also very good. It is comparable to the PCE of DSSC with 3DHG as CE but little less than that.

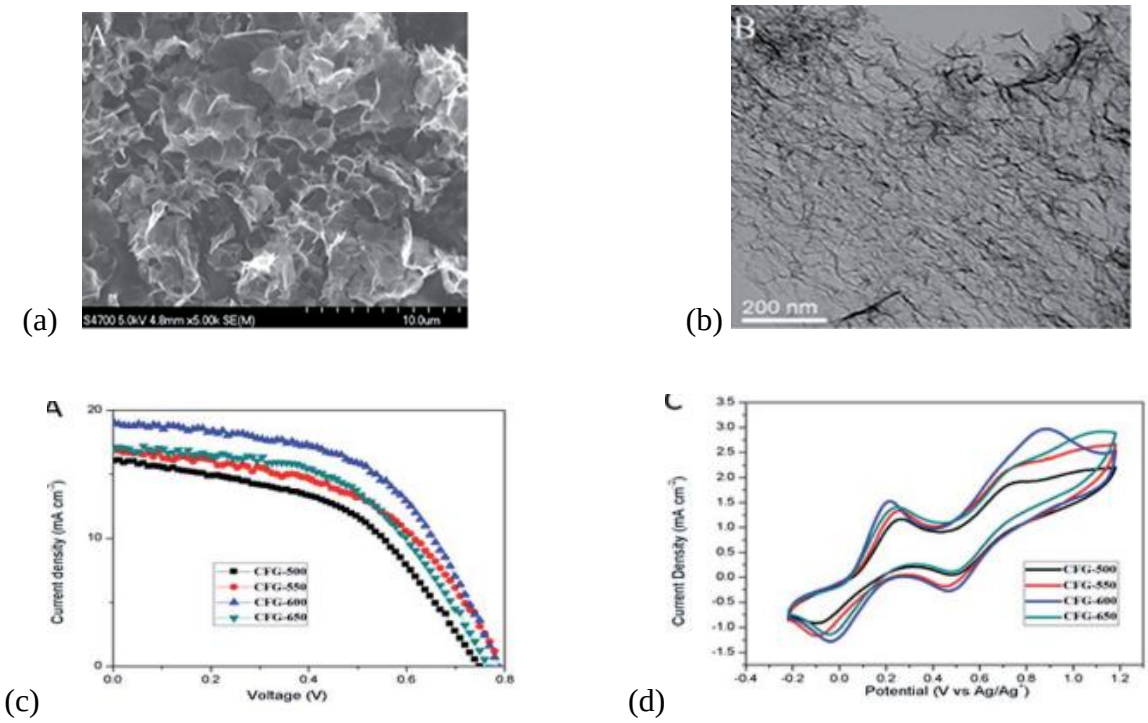


Figure 12. (a) FESEM image of CFG-600 (b) TEM image of CFG-600 (c) J–Vcurves of DSSCs with CFG counter electrodes (d) CV curves of CFG electrodes[58].

Table 5- Photovoltaic performance and electrochemical characteristics of DSSCs[58].

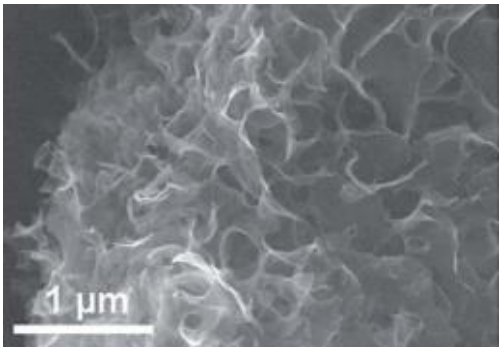
Counter electrode	J_{sc} (mA cm^{-2})	V_{oc} (V)	FF	η (%)	R_s (Ω)	R_{ct} (Ω)	Z_N (Ω)
CFG-500	16.00	0.75	0.49	5.82	16	30	420
CFG-550	16.96	0.77	0.51	6.71	17	20	330
CFG-600	19.04	0.79	0.54	8.07	16	18	180
CFG-650	17.28	0.75	0.53	6.80	16	22	320
CEG	7.56	0.65	0.14	0.70	27	2500	104

Wei Wei and her coworker also synthesis 3D crape myrtle flower graphene (CMFG) by similar reactions done above for 3DHG and CFG[59]. For synthesis of CMFG Sodium was reacted to CO_2.

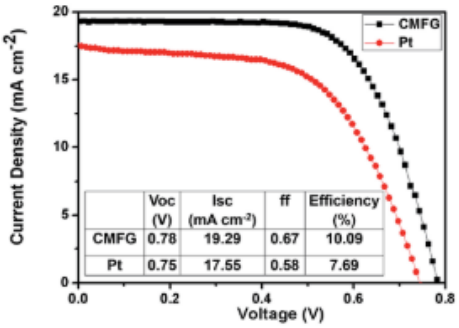
$$4Na_{(l)} + 3CO_{2(g)} \rightarrow C(\text{graphene}) + 2Na_2CO_{3(s)}$$

This reaction also has negative Gibbs free energy and enthalpy making it thermodynamically favorable reaction and energy economic as well. Simultaneous formation of Na_2CO_3 separates the graphene layer from each other hindering the formation graphite from graphene. 10 mmol of Na were reacted with CO_2 in ceramic tube reactor at 50 psi pressure and 600ºC for 24 hours. Again, the products were confirmed with X-ray diffraction measurement which showed the presence of Na_2CO_3. Products were washed with hydrochloric acid followed by DI to remove Na_2CO_3. XRD pattern shows interlayer structure of graphene where interlayer space is 0.356 nm. Further, examination on FESEM shows three dimensional CMFG structure. From TEM images thickness of graphene layer is observed which is 1.5 nm. In each layer there are 4 sheets of graphene. Performance of DSSC with CMFG as CE is much higher than DSSC with 3DHG and CFG. PCE of DSSC with CMFG as CE is 10.09% which is highest among DSSC with any CE. *Jsc* for this DSSC is 19.29 mAcm^{-2} and V_{OC} is 0.78 V with FF 0.67.

(a)



(b)



	Voc (V)	Isc (mA cm^{-2})	ff	Efficiency (%)
CMFG	0.78	19.29	0.67	10.09
Pt	0.75	17.55	0.58	7.69

Figure 13- (a) FESEM images of crape myrtle flower-like graphene.(b) Photocurrent density–voltage curves and performance parameters of DSSCs with Pt and carbon material CEs[59].

To enhance the properties of graphene as CE, Wang Yang and his coworkers used the 3D nanomesh graphene frameworks (NGFs)[60]. NGFs has very pore density hence very high surface area (1920 m^2g^{-1}) and large number of edge defects. Increasing number of edge defects helps in increasing active surface area of graphene but also increasing too much defect may effect on conductivity and may collapse the structure of graphene. NGFs is synthesis via chemical vapor deposition (CVD). For the source of carbon CH_4 is used. Reaction was carried out in vertical quartz fluidized-bed reactor at 900ºC in an argon gas flow with the rate of 1000 sccm min^{-1} at atmospheric pressure. CH_4 is added with the flow of 800 sccm min^{-1} in the reactor. After that about 30 g of MgO template is added. CH_4 was turned off after carbon is deposited in 10 to 20 minutes. Carbon is obtained in the form of black powder form which is then washed with hydrochloric acid and DI. To remove MgO template black powder is also washed with reflux simultaneously with acid for 1 hour. Finally, the black powder is dried at 80ºC in oven for one night. Obtained black powder (NGF) was mixed with binders like anhydrous terpineol, ethyl celluloses and ethanol and converted into slurry. Slurry was then coated on FTO rod by screen printing technique and heated at 450ºC for 40 minutes in Ar atmosphere. Morphology of NGFs CE was observed by FESEM and TEM. Performance of DSSC with NGFs as CE is almost like the DSSC with Pt CE.

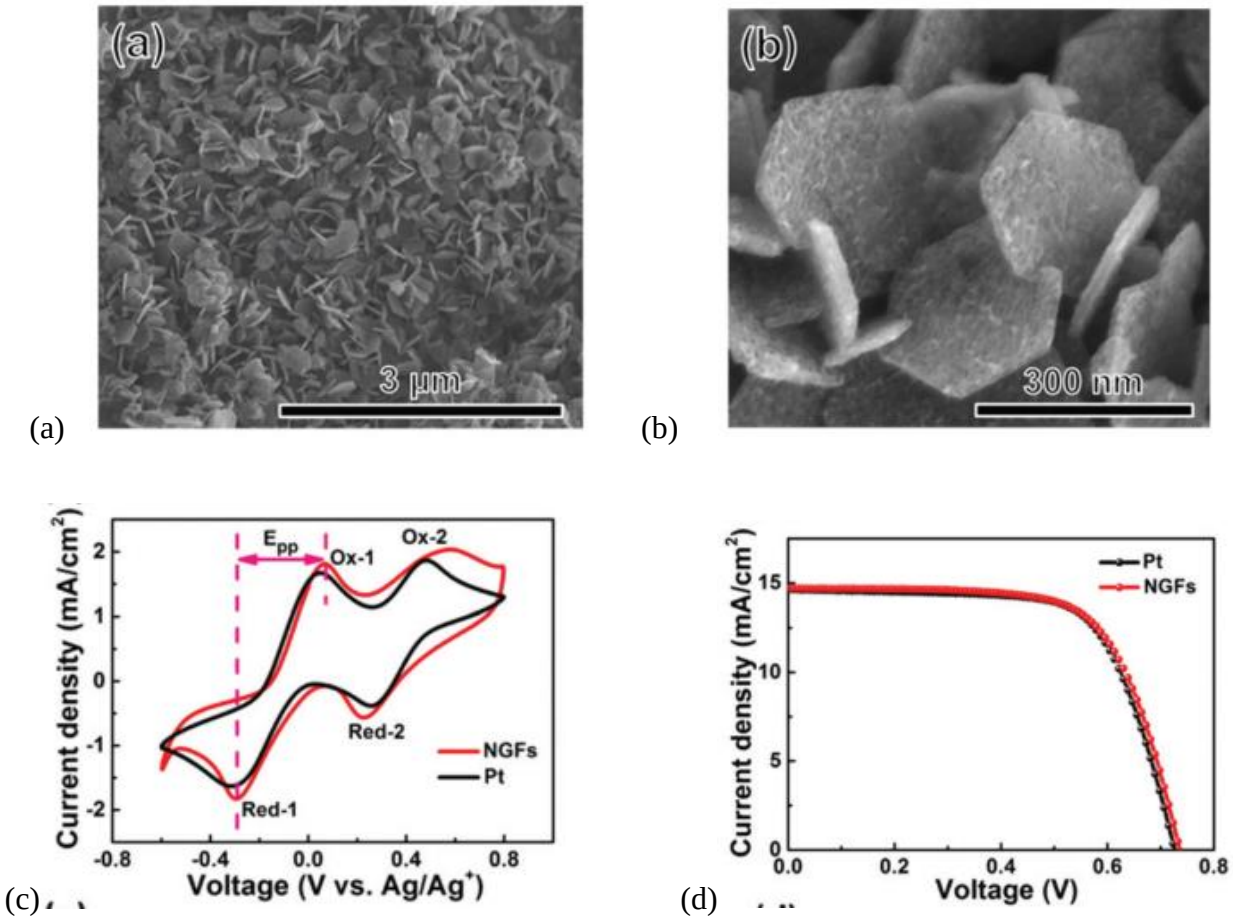


Figure 14. (a-b) SEM images of NGFs at different magnifications. (c) Cyclic voltammograms of various CEs for the I^-/I_3^- redox species recorded at a scan rate of 50 mV s^{-1}. (d) Current–voltage characteristics of the DSSCs with different CEs under one sun illumination (AM 1.5G)[60].

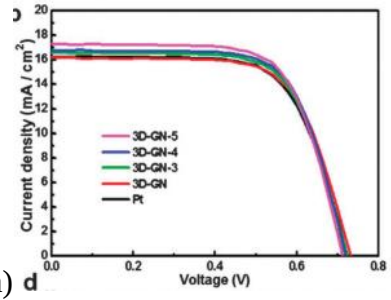
Table 6- Photovoltaic parameters of DSSCs with different CEs and the simulated data from EIS[60].

CE	I_{sc} (mA cm^{-2})	V_{oc} (V)	FF	PCE (%)	R_s (Ω cm^2)	R_{ct} (Ω cm^2)
NGFs	14.70	0.736	0.677	7.32	5.74	3.61
Pt	14.64	0.728	0.683	7.28	5.75	3.70

2.3 Doped Graphene as CE

Hyo-Jin Ahn and his coworkers found that p-Doped three-dimensional graphene networks is better than Pt as CE for DSSC[5]. 3D graphene nano-networks has very good electrical conductivity and large surface area prepared via precursor-assisted CVD. For CVD growth of graphene nontoxic iron is used as catalyst which is forth most common element found on earth so that's why it will increase applicability of graphene in fabrication cost term and in environmental safety. Conductivity of these 3D graphene is 7 S cm^{-1} and surface area is 1025 m^2 g^{-1}. They found out that doping of 3D graphene with functional group of N and O improves the electrical conductivity and catalytic activity. 3D graphene is immersed into HNO_3 to get doped with N functional group. It was immersed into solutions 50, 75 and 100% for 30 min and corresponding samples are named as 3D-GN-3, 3D-GN-4, and 3D-GN-5, respectively. Conductivity of 3D graphene increases linearly with immersion time of 3D graphene in HNO_3 solutions. It increased up to 7 S cm^{-1}. Electrolytes used to carry out cyclic voltammetry are 0.1 M $LiClO_4$, 10 Mm LiI and 0.5 mM I_2. Highest catalytic activity and electrical conductivity is shown by 3D-GN-5 because it has minimum ΔE_p and highest current density.

(a)



(b)

	N (%)	O (%)	S/cm	ΔE_p (mV)
Pt	-	-	-	386
3D-GN	0	2.5	5.0	331
3D-GN-3	0.3	8.3	6.0	314
3D-GN-4	0.4	9.1	6.3	289
3D-GN-5	0.5	9.6	7.0	269

Figure 15. (a) I–V curves. (b) Summary of physical properties of the samples[5].

Table 7- Photovoltaic characteristics of DSSC cells with Pt CE and 3D-GN CEs with various contents of N and O[5].

	R_s (Ω)	R_{ct} (Ω)	Z_w (Ω)	V_{oc} (V)	J_{sc} (mA cm^{-2})	FF (%)	η (%)
Pt	21.00	7.5	4.0	0.725	16.3	67.6	7.98
3D-GN	20.07	6.1	3.7	0.734	16.2	68.3	8.11
3D-GN-3	20.25	5.6	3.6	0.724	16.5	68.6	8.20
3D-GN-4	20.07	1.8	2.9	0.721	16.8	69.0	8.36
3D-GN-5	19.89	1.0	2.9	0.713	17.2	69.0	8.46

2.4 Composites of Graphene as CE

J. Baker and J. D. McGettrick used transparent ink of Graphene Nanoplatelet (GNP) and platinum and applied it on CE rod[36]. Impedance testing ink was coated by spin coating on fluorine doped tin oxide (FTO) electrode with varying speed to get varying ink load. Electrolyte used for this experiment was 0.8 M 1-methyl-3-propylimidazolium iodide, 0.1 M iodine, 0.3 M benzimidazole, 0.05 M guandinium thiocyanate in 3MPN. In this research GNP-Pt ink reached close to current limit of Pt electrode as seen from Tafel curve.

Table 8- Photovoltaic characteristics of DSSC cells with Pt CE, GNP-Pt T=71% and GNP-Pt T=87%.

Materials for CE	**Side**	**Voc(V)**	***J*(mA.cm^{-2})**	**FF**	***η* (%)**
GNP-Pt T=71%	Forward	0.71	10.12	0.73	5.2
	Backward	0.68	5.71	0.75	2.9
GNP-Pt T=87%	Forward	0.69	10.01	0.67	4.6
	Backward	0.68	6.79	0.71	3.3
Pt T=87%	Forward	0.70	10.15	0.72	5.2
	Backward	0.69	6.61	0.74	3.4

Cheng-En Cheng, Chi-Yuan Lin from Taiwan also tried to explored Pt graphene electrode in DSSCs and they also found increased current density and improved overall cell efficiency[45]. They found that using graphene can reduced use of Pt up to 75% which is significant to reduce the cost of DSSCs. The graphene layers were grown by chemical vapor deposition (CVD). They applied the graphene layer between Pt and FTO which reduce charge transfer resistance between Pt and FTO electrode. It enhanced short-circuit current density by 8% and PCE by 12.5%. Graphene layer is deposited in two formats, Few Layer Graphene (FLG) and Multi-Layer Graphene (MLG). FLG consist of 1-2 layers and MLG is consist of 10-12 layers.

Table 9 - Photovoltaic characteristics of DSSC cells with Pt CE, Pt/FLG CE and Pt/MLG.

Materials for CE	***J*(mA/cm^2)**	**V_{oc} (mV)**	**FF (%)**	***η* (%)**
Pt	16.0	559	63	5.3
Pt/FLG	17.3	559	64	6.3
Pt/MLG	11.4	579	67	4.4

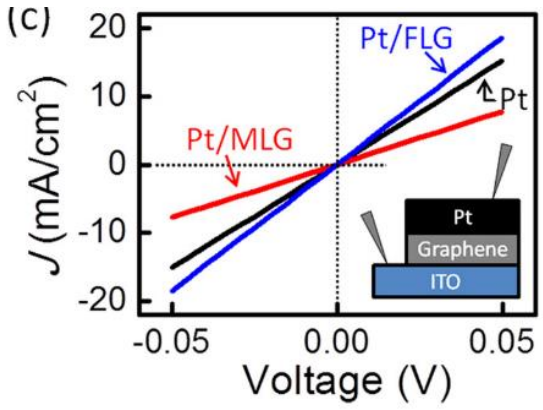


Figure 16. The J–V curves of Pt/graphene/ITO[45].

Although graphene has good electrical conductivity it lacks in electrocatalytic activity for redox reaction. To solve this problem nitrogen doped graphene with MoS_2 experimented as counter electrode by Miao-Syuan Fana, Chuan-Pei Leea and it showed much better result than any of these independent materials[33]. MoS_2 is intended to increase electrocatalytic site of composite material and nitrogen doped graphene is to increase the conductivity of material. A neutral cleaner, deionized water (DIW), acetone, and isopropanol were used sequentially to clean Fluorine–doped SnO_2 glasses (FTO, TEC-7, 7 $\Omega sq.^{-1}$, NSG America, Inc., New Jersey, USA). Direct-current sputtering on an FTO substrate method was used to prepare a Pt CE. The slurry of bare MoS_2 contained 4 wt% of its powder in a mixture of EtOH/Nafion1= 8/2 (V/V). The NGr/MoS_2 composites were prepared by mixing 4 wt%, 6 wt%, 8 wt %, 10 wt%, and 12 wt% of NGr with the slurry of bare MoS_2. For bare NGr slurry, 4 wt% NGr were added in a mixture of EtOH/Nafion1= 8/2 (V/V). On the other hand, the bare NGr slurry contained 8 wt% NGr in a solvent mixture of EtOH/ Nafion1= 8/2 (V/V) was prepared for comparison. Results shown by this composite were very close to the performance of Pt electrode. PCE showed with Pt as CE is 8.25 and PCE with NGr/MoS_2 found 7.82%.

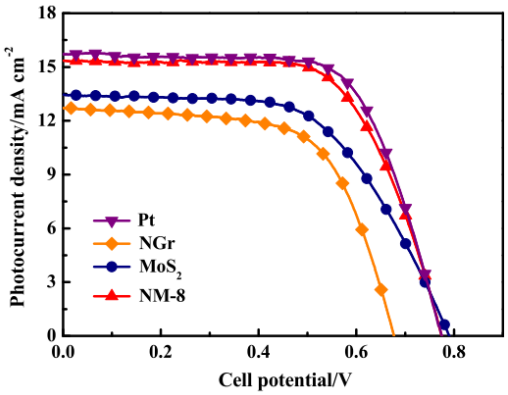


Figure 17. Photocurrent density-voltage (j-V) curves of the DSSCs with various CEs, under irradiation at 100 $mWcm^{-2}$ (AM 1.5G)[33].

Hyun-Young Kim and his coworkers used plasmonic enhanced graphene flake with gold nanoparticles in it[61]. Arrays of Au nanoparticles are deposited on Langmuir-Blodgett assembly after that graphene flakes were deposited on it. Graphene flakes were produced via thermal plasma jet. Ethylene gas (0.5 L/min) was used as carbon source and continuously injected into the plasma torch. 45mg graphene flakes were added to 30 mL of dimethylformamide (DMF) and then it dispersed the flakes by sonication for 1 hr. The graphene flake suspension (0.5 ml) and distilled water was mixed together and then ethyl acetate was poured onto the mixture. This helps to form self-assemble graphene film which later transferred to array of immobilized Au nanoparticles on FTO glass. In the results graphene flake and gold nanoparticle (hybrid) CE showed the PCE even more that Pt CE. Normal Pt CE showed the PCE of 9.08% and the hybrid CE showed the PCE of 9.78%.

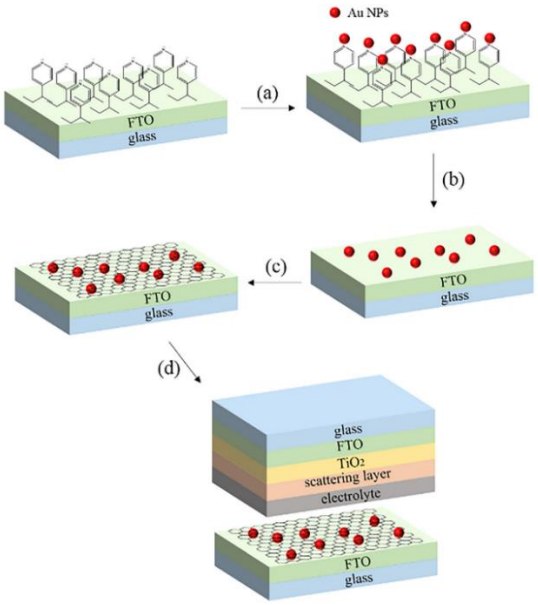


Figure 18- Scheme for construction of a DSSC with a nanostructure-based graphene flake counter electrode: (a) immobilization of Au NPs on FTO coated with P4VP, (b) sintering at 350ºC to remove P4VP, (c) deposition of graphene flakes, and (d) fabrication of a DSSC[61].

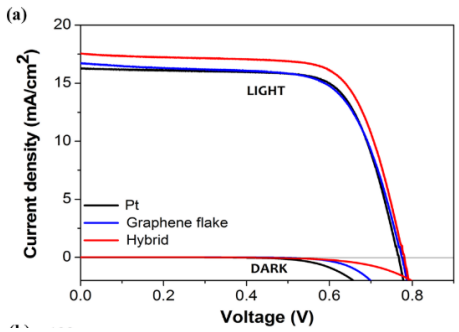


Figure 19- J–V curves and dark current characteristics[61].

TaNA and reduced graphene oxide (RGO) as counter electrode in the cell with the electrolyte $Co(bpy)_3^{3+/2+}$ (bpy = 2,2′-bipyridine) gives very good PCE of 7.65% which is very close to DSSC of Pt counter electrode[8]. Yan Li and his coworkers made the counter electrode of RGO and TaNA with very unconventional electrolyte which is $Co(bpy)_3^{3+/2+}$. polypyridine complexes of Co(II)/Co(III) coupled with donor (D)-π-bridge-acceptor (A) structured organic dyes really helped to increase efficiency of DSSCs. The intermediate state of graphene and graphene oxide, RGO contains many oxygen-containing functional groups (−OH, =O, −COOH) and surface defects which are believed to help in electrocatalytic active site which interact with metals, metal oxides or metal nitrides. 0.50 g of $TaCl_5$ powder, 0.14 g of $CaCO_3$ and 0.75 g of urea were mixed into the 2 mL of methanol successively. This solution is dried with stirring later till it becomes gel. For 6 hours after that it was calcinated in pure N_2 at 120 Pa and 780ºC. Resulting powder was treated with 1 M HCL for a day and washed with deionized water to remove Ca species. TaON NPs were obtained by ultrasonically dispersing in ethanol. Graphene oxide was obtained from graphene paper via modified hummers method. By hydrazine hydrate reduction of 0.25 mg mL^{-1} of exfoliated graphene suspension in water the reduced graphene oxide suspension was obtained. To get graphene oxide/ TaON nanocomposites some amount TaON/ethanol suspension was added to 10 mL of GO solution in ultrasonic condition. Finally adding 25 µL hydrazine hydrate and heating

in oil bath 98ºC about 5 hours we get RGO-TaON nanoparticles. The result indicated that combined effect of RGO and TaON helps in the reduction of the $Co(bpy)_3^{3+}$ at the interface of electrolyte and CE. PCE obtained with RGO and TaON as CE is 7.65% which is very much close to PCE of Pt counter electrode 7.91%. While individually using RGO and TaON as CE materials showed very poor results with PCE of 4.62% and 2.54% respectively.

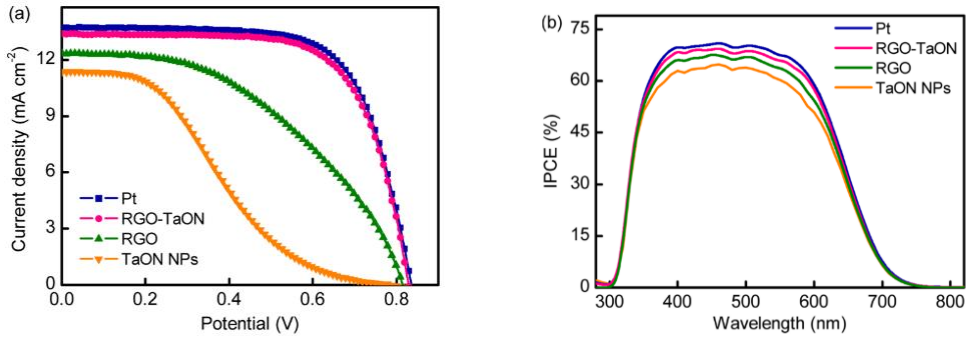


Figure 20. (a) *J*-V characteristics of DSSC with different CE materials. (b)IPCE spectra for cells[8].

Table 10 - Photovoltaic Performance and EIS Parameters of the DSSCs with Different CEs[8].

CE	V_{oc} (mV)	J_{sc} (mA cm^{-2})	*FF*	η (%)	R_s (Ω cm^2)	R_{ct} (Ω cm^2)	Z_N (Ω cm^2)
Pt	835	13.73	0.69	7.91	1.7	1.8	2.7
RGO–TaON	829	13.38	0.69	7.65	2.1	1.9	3.0
RGO	814	12.33	0.46	4.62	1.9	6.9	7.4
TaON NPs	773	11.35	0.29	2.54	1.8	46.9	8.6

Not just TaON but MoS_2 also gives very good result with RGO as CE. Chia-Jui Liu and his coworkers also did similar experiment in which they used MoS_2 instead of TaON with RGO as CE[9]. They used I_3^- as electrolyte. This CE was synthesized by mixing graphene oxide nanosheet with an ammonium tetrathiomolybdate and then at 650ºC in H_2 flow they converted the solid intermediate into MoS_2/RGO nanocomposite. Due to combining MoS_2 with RGO active surface area increased which helped in increasing current density. Also, after consecutive 100 tests

MoS_2/RGO CE showed no sign of degradation means it has high chemical stability. Furthermore MoS_2/RGO CE showed very low charge-transfer resistance for reduction of I_3^-. Single layer GO nanosheets were manufactured by modified Hummer's method.

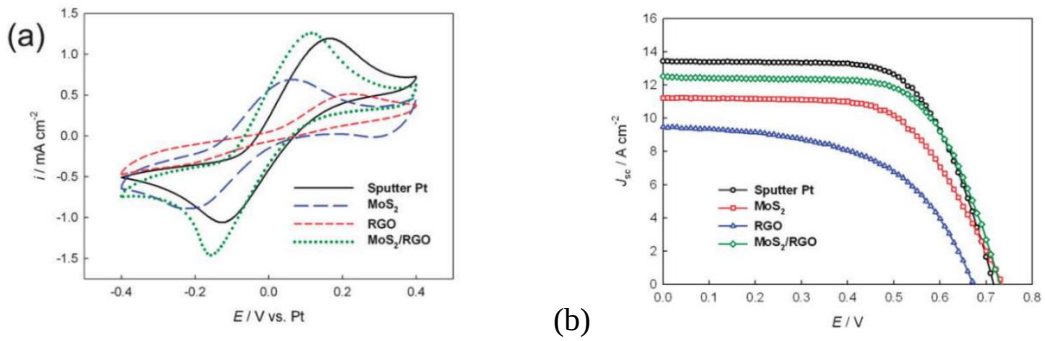


Figure 21 - (a) CVs of I^-/I_3^- for the four different CEs at a scan rate of 10 mV s^{-1}. (b) Photovoltaic performances of DSSCs based on MoS2, RGO, MoS2/RGO and sputtered Pt CEs[9].

Table 11. Photovoltaic Performance and EIS Parameters of the DSSCs with Different CE[9].

	R_s (Ω cm^2)	R_{ct} (Ω cm^2)	C_μ (μF cm^{-2})	J_0 (mA cm^{-2})	J_{sc} (mA cm^{-2})	V_{oc} (V)	FF	η (%)
Sputtered Pt	6.06	1.93	1.15	3.88	13.42	0.72	0.66	6.38
MoS_2	8.29	2.11	20.85	2.94	11.21	0.73	0.62	5.09
RGO	6.07	6.09	22.47	0.25	9.47	0.67	0.54	3.43
MoS_2/RGO	6.46	0.57	45.42	5.89	12.51	0.73	0.66	6.04

Graphene-tungsten nanocomposite also explored as CE by B. Munkhbayar and his coworkers[13]. Average thickness of GN used is 8 nm which consist of 20-30 monolayers and they are of 550 nm in lateral size. The average size of tungsten is around 100 nm. Usually, GNs have hydrophobic surfaces so they tend to aggregate and precipitate in base fluids if dispersant is not there. Using nitric acid (HNO3) and sulphuric acid (H2SO4) with concentration of 63% and 98% GNs are purified as they help in better dispersion. Aggregation reduce the catalytic activity of CE material, so to improve performance of DSSCs dispersion of CE material is very important. To remove impurities and to improve exterior activity ultrasonication 1510E-DTH was performed for 5 hours. This purified GN and tungsten was mixed at room temperature. They were

ultrasonicated in anhydrous ethanol for 40 minutes. Concentration of particles in ethanol was 2.0% by weight. This material was deposited on FTO by spin coating method. Material was deposited for 30s at 2000 rpm. Then it was heated for 15 minutes at 350ºC to maintain good adhesion between the material and FTO rod. Result of this composite electrode were really impressive. Purified and grounded GN showed the PCE of 4.55% whereas Pt electrode showed the PCE of 5.92%.

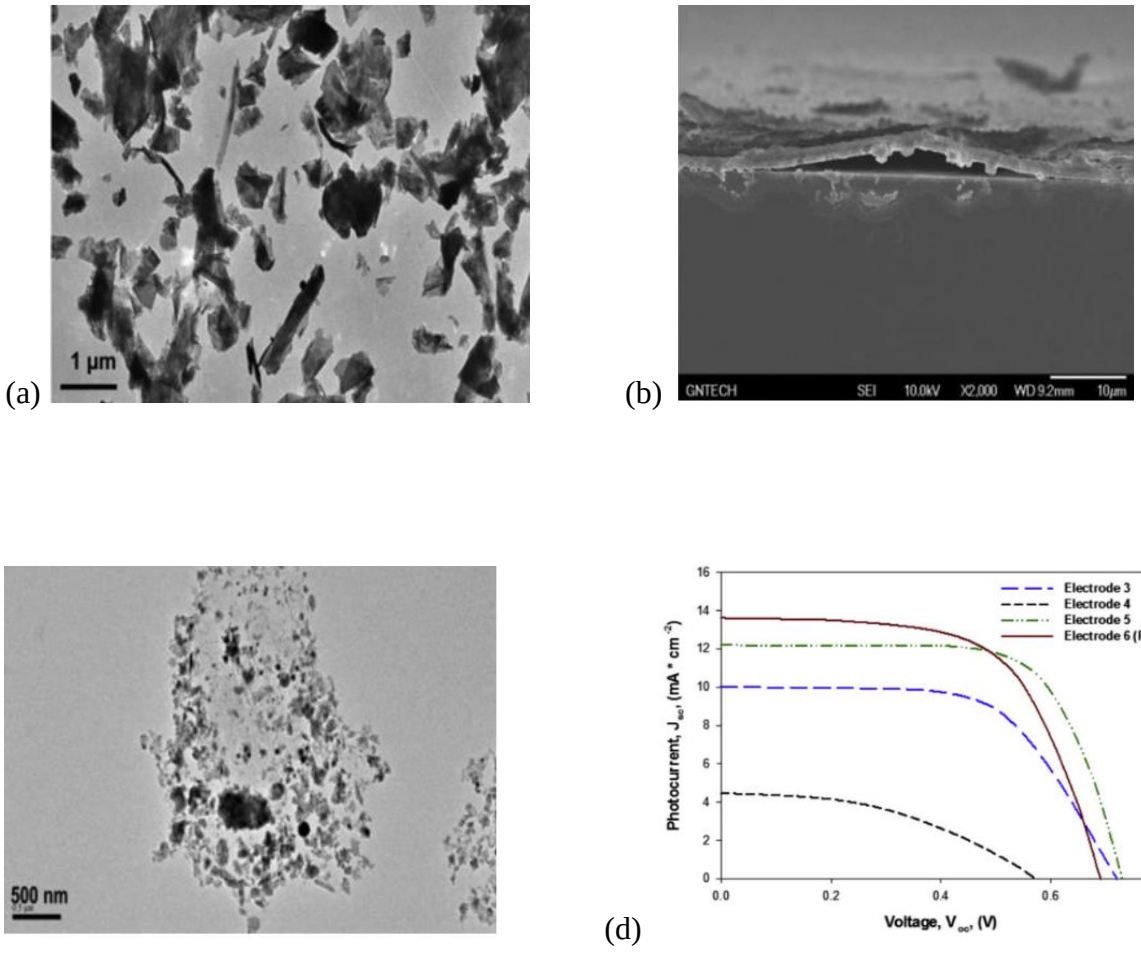


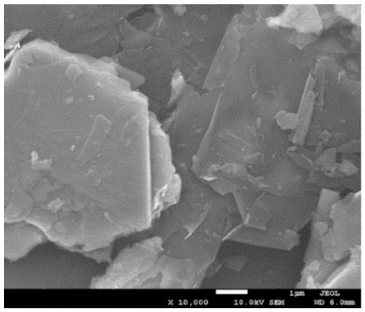
Figure 22- (a) TEM image of purified and ground GN films. (b) Cross sectional of SEM image purified and ground GNs (c) HRTEM image of GN-tungsten composite. (d) The photocurrentevoltage (JeV) characteristics of DSSCs with GN, tungsten and GN-tungsten composite counter electrode structures[13].

Table 12. The J-V curve characteristics of DSSCs with various GNs counter electrodes[13].

Where, Electrode 1: - Raw Graphene; Electrode 2: -Purified Graphene; Electrode 3: - Purified and Ground Graphene; Electrode 4: - Pt;

Counter Electrode	J_{sc} (mA cm^{-2})	V_{oc} (V)	FF	η (%)
Electrode 1	8.00	0.58	0.58	2.71
Electrode 2	8.28	0.64	0.59	3.18
Electrode 3	10.02	0.72	0.63	4.55
Electrode 6 (Pt)	13.62	0.69	0.63	5.92

Gentian Yue has done the research on the composite of MoS_2 and graphene flakes[29]. He found that when high conductive graphene flakes were introduced to MoS_2 film it helped to enhance electrocatalytic activity of composite counter electrode. MoS_2 has layered structured which is analogous to the structure of graphene which why the synergetic effects work well in between them. MoS_2 and graphene nanopowder with 8 nm flakes were mixed using ball machine for 30 minutes. Then that mixture is converted into slurry with 80 wt.% of MoS_2 and Graphene mixture, 10 wt.% acetylene black and polyvinylidene fluoride dissolved into N-methyl-2-pyrrolidinone. Subsequently the slurry is coated on FTO rod by doctor blade method where thickness is controlled by adhesive plaster layer. SEM images and XRD analyses has been done to confirm the composition of material. These tests confirmed the synthesis of MoS_2/Graphene composite.

(a) 

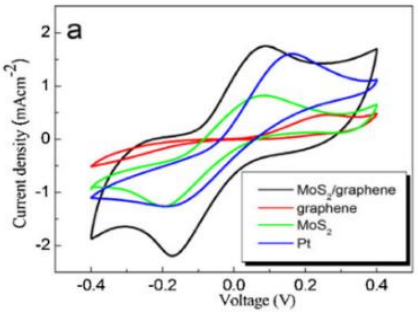
(b) 

Figure 23. (a) The SEM images of MoS2/graphene powders. (b) CVs for the Pt, MoS2, graphene and MoS2/graphene CEs at the scan rate of 10 mV s^{-1}[29].

Table 13. The resultant EIS and photovoltaic parameters of DSSCs based on the Pt and MoS2/graphene CEs[14].

Electrodes	R_s (Ω cm^2)	R_{ct} (Ω cm^2)	V_{oc} (V)	J_{sc} (mA cm^{-2})	FF	η (%)
MoS_2/graphene	24.42	4.94	0.71	12.41	0.68	5.98
Pt	24.72	4.72	0.75	12.43	0.67	6.23

NiS incorporated in Reduced Graphene Oxide (RGO) shows impressive results as CE in DSSC. Xueqin Zuo and his coworkers in their experiment synthesized NiS/RGO CE for DSSC[40]. They synthesized NiS/RGO CE through facile solvothermal route. 1 mmol nickel and 3 mmol thiourea dispersed in 15 mL absolute alcohol followed by stirring for 30 min to get clear solution. Above solution is mixed with 15 mL alcohol consisted of 10, 20, 30, 40 or 50 mg of GO powders and sonicated for 1 hour with stirring. After that solution goes through solvothermal reaction for 38 hours at 200ºC in 50 mL Teflon-lined autoclave. Solution was stirred for 1 hour to mix 0.5 g of L-Ascorbic acid into it and again reaction at 200ºC for 10 hours. After all these processes NiS particles were homogeneously attached to RGO surface. For the preparation of CE NiS/RGO was mixed up with polyethylene glycol powder (0.012g) and after that it was dispersed in 4 mL ethanol and stirred to form colloid. With an area of 0.5 × 0.5 cm^2 colloid was doctor-bladed on FTO glass. Finally, CE was annealed for 1 hour at 375ºC in argon atmosphere. Performance of DSSC with NiS/RGO as CE is pretty good compare to solo NiS and RGO CEs. It is even more than DSSC with Pt CE. PCE of DSSC with Pt as CE is 7.21% and with NiS/RGO CE is 7.67%.

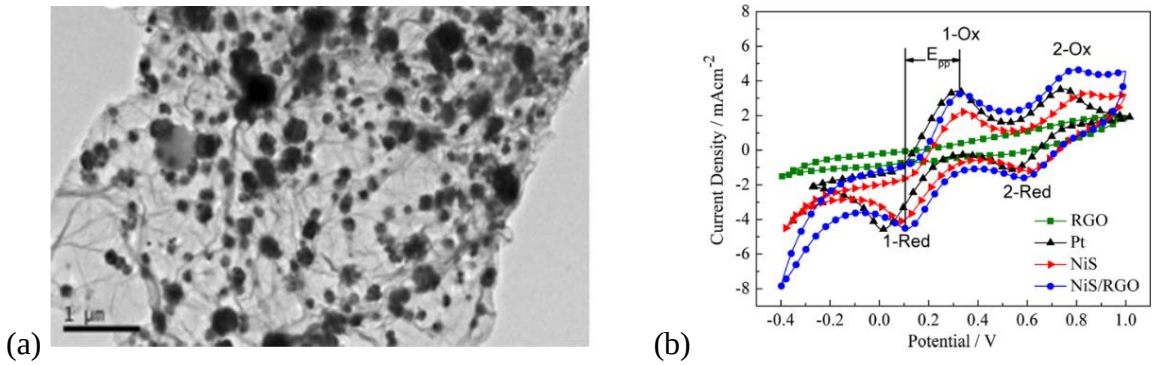


Figure 24. (a) images of NiS/RGO composites (b) CVs curves of RGO, Pt, NiS and NiS/RGO electrodes[40].

Table 14. EIS and photoelectric parameters of the DSSCs with various CEs[40].

CEs	V_{oc} (V)	J_{sc} (mA cm^{-2})	FF	PCE (%)	R_s (Ω cm^{-2})	R_{ct} (Ω cm^{-2})	Z_W (Ω cm^{-2})	E_{pp} (V)
Pt	0.748	14.10	0.68	7.21	7.65	0.60	0.90	0.30
RGO	0.623	11.58	0.40	2.92	6.39	32.73	–	0.62
NiS	0.729	13.59	0.63	6.25	5.62	1.23	1.18	0.25
NiS/RGO	0.750	14.82	0.68	7.67	6.13	0.63	0.68	0.22

Quanhong Chang and his coworkers synthesized 3D GF by CVD using Ni foam as a template[7]. They heated at 880ºC in the quartz tube for 30 minutes at heating rate 10ºC/min under the pressure of 10 Torr with mixed gas flow of H_2/N_2 to clean the surface of Nickel foam. With an addition C_2H_2 and N_2 for 5 min CVD growth of GF is started. After that graphene-Ni foam is cooled rapidly with a rate of 100ºC/min under H_2/N_2 flow. Ni foam is taken away from the graphene with $FeCl_3$/HCl solution at room temperature. This is how they finally got pristine 3D graphene and 0.2 mm thickness of this 3D graphene was placed on FTO conducting substrate. Graphene layer was attached to FTO substrate with Vandar Waal forces.

They also synthesized large-sized heat-reduced graphene nanosheets (H-GNs) and small-sized laser-reduced graphene quantum dots (L-GQDs). Graphite powder (2g) was mixed up with concentrated H_2SO_4 at 2ºC. $KMnO_4$ (9g) was mixed up with solution gradually. Mixture is maintained below 20ºC with continuous cooling. Mixture was stirred for next 7 days at room temperature. To increase temperature to 98ºC, 250 ml distilled water is added. Solutions was

maintained at 98ºC for 15 minutes. H_2O_2 (6 mL, 30%) is then added to solutions at 60ºC. Solution was washed repeatedly with DI water and HCl. To get Graphene Oxide (GO) solution is dried in vacuum oven for 24 hours at 60ºC. To get an H-GN graphene oxide is heated for 30 minutes in quartz boat in H_2 containing atmosphere at 700ºC. After that to get a L-GQDs further process is done. Purified GO is dispersed into DI water using ultrasonication for 1 hour. At pH 9 ammonia was added to GO suspension in solution. GO suspension becomes Yellow-Brown in color. 15 ml of this solution was then loaded into quartz tube. After that solution goes through pulsed laser reduction with laser energy 250 mJ and 3 Hz frequency. Finally, for 1 hour GO sheet went through pulsed laser irradiation and reduced into L-GQDs. This H-GNs and L-GQDs were dispersed into 2-propanal and sonicated for 5 minutes and 15 minutes respectively. After that solution was kept for night to separate big particles from sedimentation. Concentration of solutions were adjusted by varying dispersion number of H-GNs and L-GQDs. Subsequently solution is spin-coated on GF/FTO rod with rpm rate of 1000 for 20 seconds. Finally, this hybrid CEs was dried in vacuum at 120ºC overnight. TiO_2 photoanode of thickness 14 µm was used in this experience. To increase porosity electrodes were sintered at 500ºC for 30 minutes in air. Performance of L-GQDs/GF CE DSSC is the best among all. It showed efficiency even more than Pt CE DSSC. H-GNs also shows results close to Pt CE DSSC but little less.

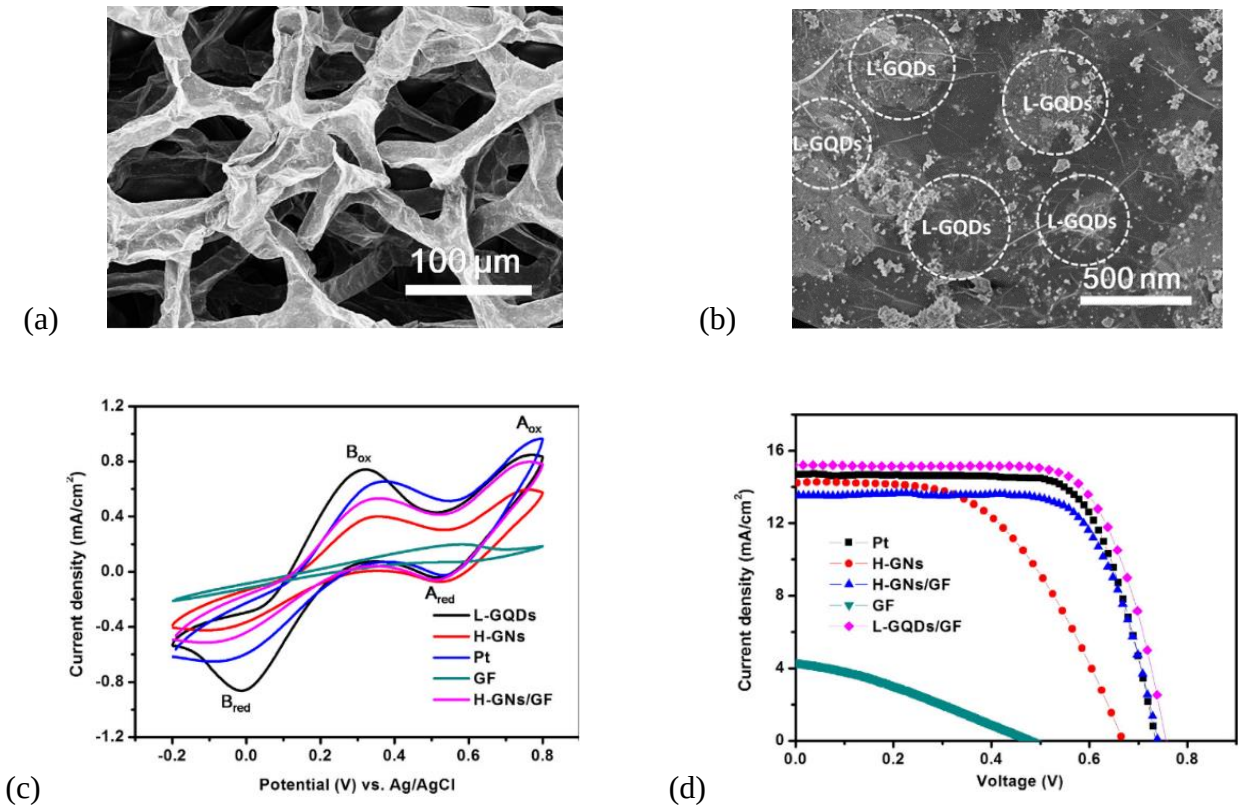


Figure 25. (a) SEM image of the GF structures showing a continuous network of 3D interconnected graphene sheets (b) SEM images of the hybrid L-GQDs/GF. (c) Cyclic voltammetry of CEs with various CEs (Pt, GF, HGNs, H-GNs/GF and L-GQDs/GF) obtained at scan rate of 50 mV s^{-1}. (d) Photocurrent–voltage characteristics of DSSCs with various CEs (Pt, GF, H-GNs, H-GNs/GF and L-GQDs/GF)[7].

Table 15- Photovoltaic parameters of DSSCs fabricated with various CEs[7].

CE	R_S	R_{CT} (Ω cm^2)	V_{OC} (V)	J_{SC} (mA/cm^2)	FF (%)	η (%)
H-GNs	13.00	8.70 ± 0.20	0.66	14.26	51	4.90
GF	13.50	38.00 ± 0.30	0.48	4.31	33	0.68
Pt	12.82	1.18 ± 0.04	0.75	14.57	71	7.68
H-GNs/GF	12.85	1.30 ± 0.03	0.74	13.52	70	7.10
L-GQDs/GF	12.83	1.20 ± 0.02	0.76	15.21	72	7.70

CHAPTER 3

OBJECTIVE AND HYPOTHESIS

Increase in a usage of renewable energies is so crucial nowadays because of the climate change but the cost of renewable still keeping people away from using it. Our research's objective is to use low cost carbon materials for making DSSC and bring down the cost of solar panels in future. From the literature review we can see how carbon materials have so much potential to be good counter electrode for DSSC.

Hypothesis statements that are considered in this research work:

Hypothesis I: Activated carbon has a good conductivity, so it would be excellent counter electrode for the DSSC.

Hypothesis II: Doping KOH into carbon and providing a heat will improve the structure of the activated carbon and increase catalytic activity of carbon.

CHAPTER 4

EXPERIMENTAL SECTION

4.1 Preparation of the Photoelectrodes

Photoelectrodes were prepared applying TiO_2 layer on a standard Fluorine doped-Tin Oxide (FTO) glass. First, an FTO glasses were cleaned using the acetone. Complete area was not used to apply the TiO_2 layer but area of 0.5*0.5 was marked by using Scotch tapes. TiO_2 solution was prepared using 200 mg of TiO_2 powder and 2 mL of ethanol for approximately 10 samples of photoelectrodes. After a TiO_2 powder is mixed well in ethanol, solution was applied on FTO glass using doctor blade method. 2-3 layers of solutions were applied to get good thickness. Photoelectrodes were left to dry for approximately 30 minutes. Once they are completely dry tape was removed from the FTO glasses and then they were heat treated. For heat treatment FTO glasses with TiO_2 layer were kept in furnace at $500^{O}C$ for 40 minutes. Finally after furnace cools down photoelectrodes were taken out and kept in dye solution which was prepared using cisbis(isothiocyanato)bis(2,2′-bipyridyl-4,4′-dicarboxylato)ruthenium(II)-bis-tetrabutylammonium (N719, 0.3 mM in ethanol) for 72 hours.

(a) (b)

Figure 26- Thermo Scientific furnace and oven used in this research.

4.2 Preparation of the electrolyte

In this research, Redox (I^-/I_3^-) solution is used as electrolyte. This solution contains 0.6 M 1-butyl-3methylimidazolium iodide, 0.025 M LiI, 0.28 M tert-butyl pyridine, 0.04 M I_2 and 0.05 M guanidinium thiocyanate with acetonitrile as Solvent.

Figure 27- Ohaus weight balance used in this research.

4.3 Preparation of the Counter-electrode

There were 3 types of counter electrodes prepared for the experiment, Pt CE, AC CE and KOH doped AC CE. For Pt CE, $H_2PtCl_6.H_2O$ precursor was taken. 10 mM solution was prepared from precursor by adding alcohol. Prepared solution was put on the FTO glasses and heat treated after dried. For the heat treatment electrodes were put in furnace at 450^oC for 20 minutes. For AC CE AC was taken and grinded for approximately 20 minutes with ethanol to make particles of AC very fine and get more surface area with lesser thickness. Clean FTO glasses were taped with an area marked 05*05 and grinded AC was applied on it. After the counter electrodes dried they were untaped and put in oven for at least 2 hours to remove all of the moisture. And finally, for KOH doped AC CE, AC was added with KOH in two weight ratios, 5:1 and 5:2. Samples were added

with 20 mL of deionized water and mixed in ultrasonic vibrations for 1 hour. Liquid solution then heated in microwave for various times and 5 samples were made. Times for which samples were heated was 0,1,3,5 and 8 minutes. After the heating, samples were washed with deionized water several times to bring pH down to 7. Once the samples reached to pH 7 they were kept in oven at $70^{O}C$ for drying overnight. After drying all samples counter electrodes were prepared for all of them similarly as AC CE were prepared.

4.4 Preparation of DSSC

Prepared photoelectrodes and counter electrodes were then joined together using the electrolyte. Photoelectrodes taped with scotch tape again around the TiO_2 layer after cleaning the dye solution from it. While taping some space is left untapped for clamping the alligator clips and make connection with instruments. Electrolyte is dropped on the photoelectrode with syringe making sure that electrolyte covers all TiO_2 layer and tape. Electrolyte is strong enough to meltdown a tape and make it sticky glue so that counter electrode can be put on it and hold both electrodes strong together as a cell. Prepared cell was evaluated with Gamry instrument under illumination of AM 1.5.

4.5 Characterization

Brunauer–Emmett–Teller (BET) test was performed to find area of the AC samples prepared. Micromeritics ASAP 2000 adsorption instrument which uses nitrogen adsorption technique to measure surface was used at very low temperature of 77K. For the structure analysis field emission scanning electron microscope (FESEM) and energy dispersive spectroscopy (EDS) was performed using Hitachi-4700.

Figure 28- Micromeritics ASAP 2000 for BET test.

Figure 29- Hitachi 4700 used in FESEM test.

I-V test were performed using VersaSTAT 3F instrument under illumination of AM 1.5. Voltage range was taken from -0.1V to 0.8V with step size 0.01V. Effective area of cell was 0.25 cm^2. All of the current values were recorded over the voltage range -0.1V to 0.8V.

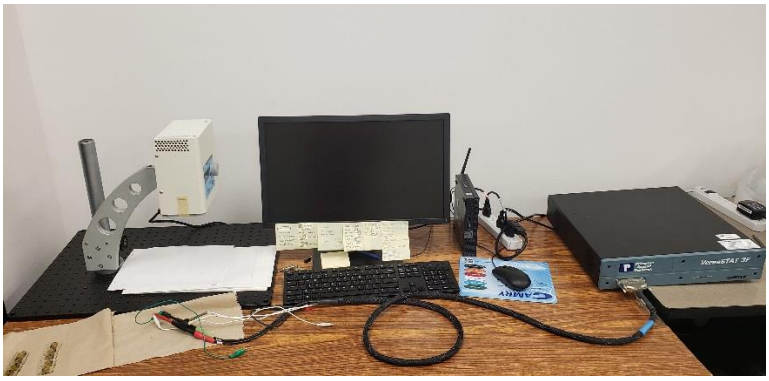


Figure 30- Setup of solar illuminator and VersaSTAT 3F electrochemical workstation for I-V test.

Cyclic voltammetry test was performed on AC and KOH doped AC CE's using the VersaSTAT 3F electrochemical workstation under dark conditions. 3 electrode system was arranged with AC CE's as working electrode, Ag/AgCl as a reference electrode and Pt as CE. All three electrodes were immersed well inside solution containing 10 mM LiI, 1 mM I_2, and 0.1 M $LiClO_4$. Electrochemical impedance spectroscopy (EIS) test was also performed on the DSSC with same VersaSTAT 3F instrument over the frequency range of 0.1 to 100 k Hz in dark conditions. Resistance of all AC CE and KOH doped AC CE's was obtained through EIS test.

CHAPTER 5

RESULTS AND DISCUSSION

The photovoltaic performances of DSSC with dye immersed TiO_2 photoelectrode, iodide/ triiodide and KOH doped with AC as a counter electrode were analyzed under simulated sunlight with an intensity of 100 $mWcm^{-2}$. Another DSSC with Pt CE and AC CE were also prepared and tested for the comparison. All the parameters for the photovoltaic performance test like short-circuit current density (Jsc), open-circuit voltage (Voc), fill factor (ff), and power conversion efficiency (PCE) are summarized in a table 1. Maximum efficiency was achieved by a sample which had 5:1=AC:KOH weight ratio and 0 minute microwave heating. 5.81% maximum efficiency was achieved. While with Pt CE maximum efficiency achieved was 5.61%. Efficiency in DSSC with pure AC as CE was achieved as 4.68%.

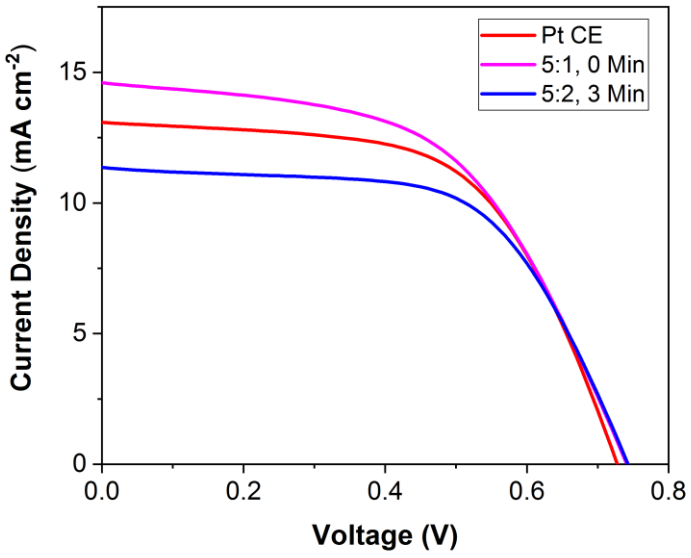
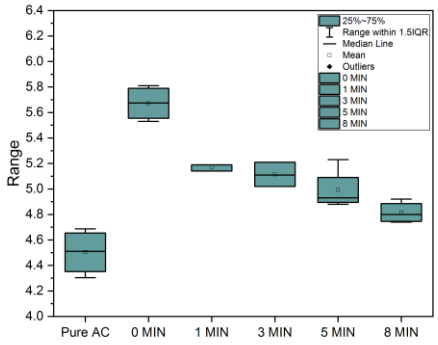


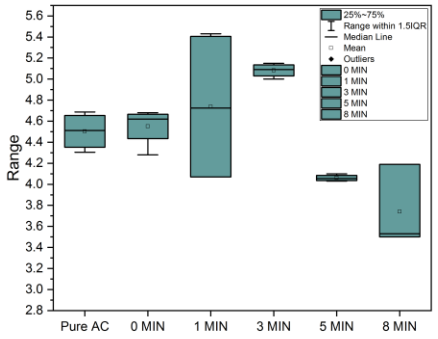
Figure 31- I-V curve for Pt CE DSSC, 5:1 AC:KOH, 0 minute CE and 5:2 AC:KOH CE DSSC.

Table 16- shows the short circuit current, open circuit voltage, filling factor and efficiency of the Pt CE, AC CE, AC:KOH=5:1, 0MIN and AC:KOH=5:2, 3MIN CE in DSSC.

Samples	***Isc***	***Voc***	***FF***	***%EFF***
Pt CE	13.1	0.72	59.0	5.61
AC	14.8	0.72	43.9	4.68
AC:KOH=5:1, 0MIN	14.60	0.74	53.52	5.81
AC:KOH=5:2, 3MIN	11.35	0.74	60.99	5.15



(a)



(b)

Figure 32- Average efficiency graph various time heated samples (a) 5:1 weight ratio (b) 5:2 weight ratio.

As mention in experimental section CV test was performed with using electrode systems, Pt CE as counter electrode, AC CE's as working electrode and Ag/AgCl as a reference electrode. In the graph of CV test usually 2 pairs of peaks are noticed. So, pair of peaks towards negative side shows redox reaction I_3^-/I^- and pair of peaks towards positive side shows the redox reaction I_2/I_3^-. In CV curves peak to peak separation is inversely proportional to electrochemical rate constant. First graph is for the weight ratio 5:1 of AC:KOH and second graph is for the weight ratio 5:2 of AC:KOH. From the first graphs we can see that for 8 min sample has lowest peak to peak separation distance and reasonably high current density peak which means it has highest catalytic

activity. Similarly, for 5:2 weight ratio again the 8 min sample has less peak to peak separation distance and highest current density peak. For both the weight ratio samples we got least peak to peak separation distance in 8 minutes samples which proves that microwave heating increases the electrocatalytic activity of the KOH doped AC sample.

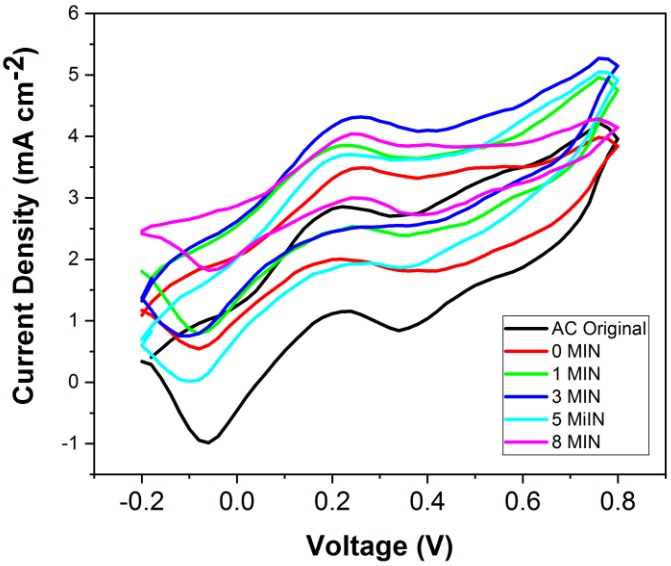


Figure 33- shows the CV curves for AC:KOH=5:1 CE's heated at various temperature in DSSC.

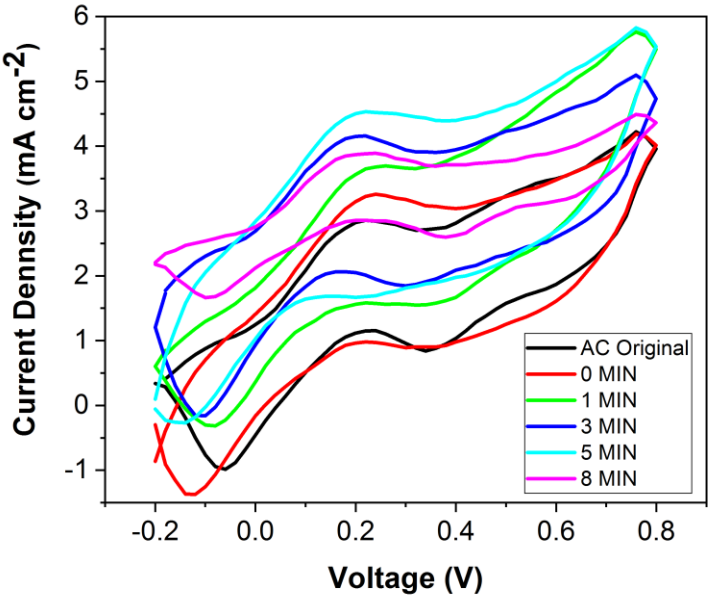


Figure 34- shows the CV curves for AC:KOH=5:2 CE's heated at various temperature in DSSC.

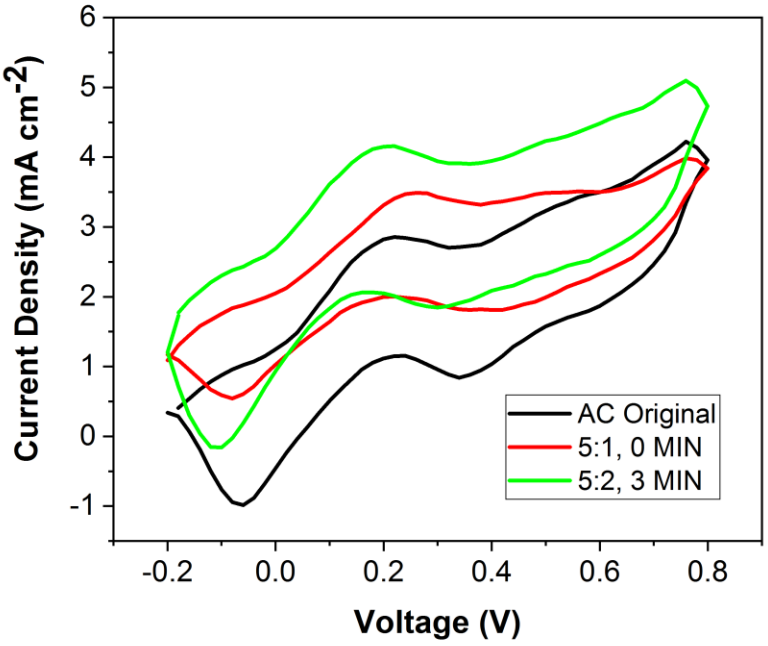


Figure 35- shows the CV curves for pure AC CE, AC:KOH=5:1 0 minute heated and AC:KOH=5:2 3 minute heated CE's in DSSC.

To find out the internal resistance of the DSSC cell EIS test was performed. From the results of the EIS test Nyquist graphs were drawn. Generally, the Nyquist graphs have 2 semicircles in it. The intercept from first semicircle gives us a series resistance of cell whereas the semicircle distance shows us charge transfer resistance. First semicircle shows the charge transfer resistance from CE to electrolyte and the second semicircles shows charge transfer resistance from electrolyte to photoelectrode. From the graph we can see that series resistance for all the DSSC cells is almost similar. Adding the KOH largely increased the resistance of AC.

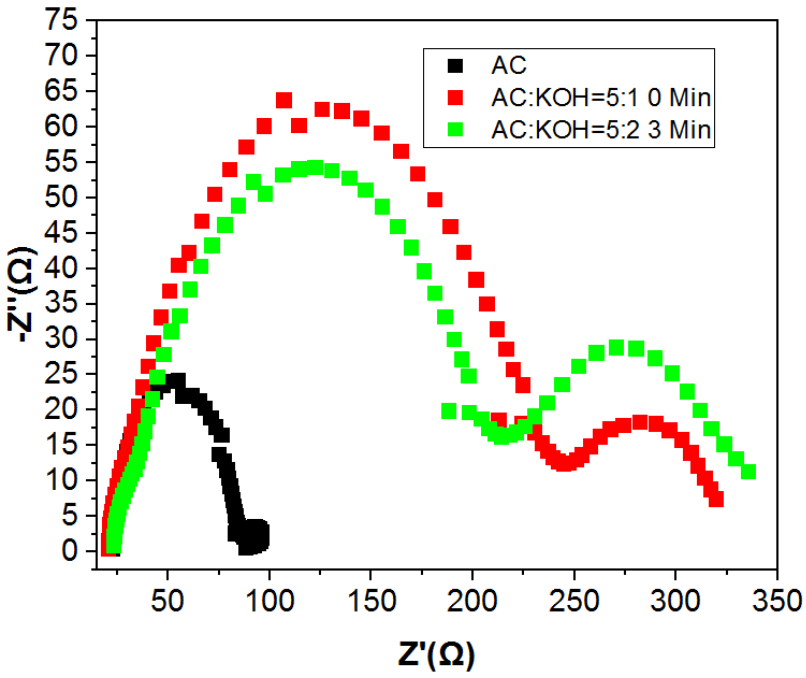
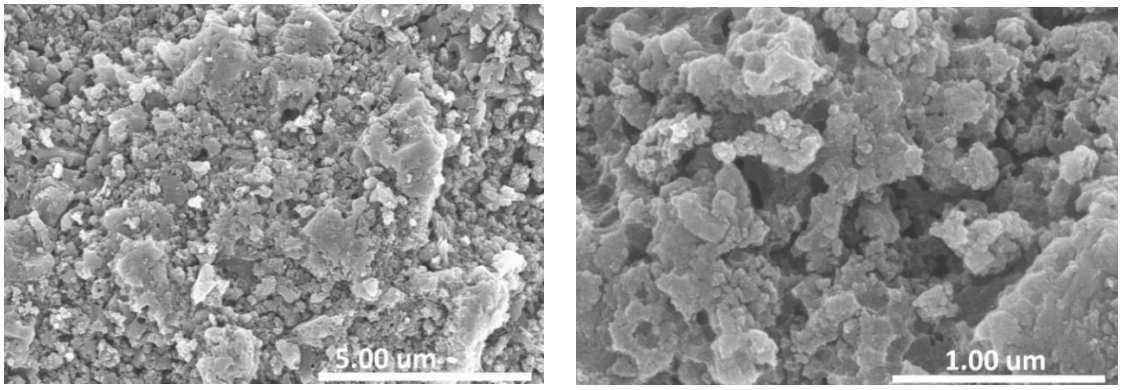


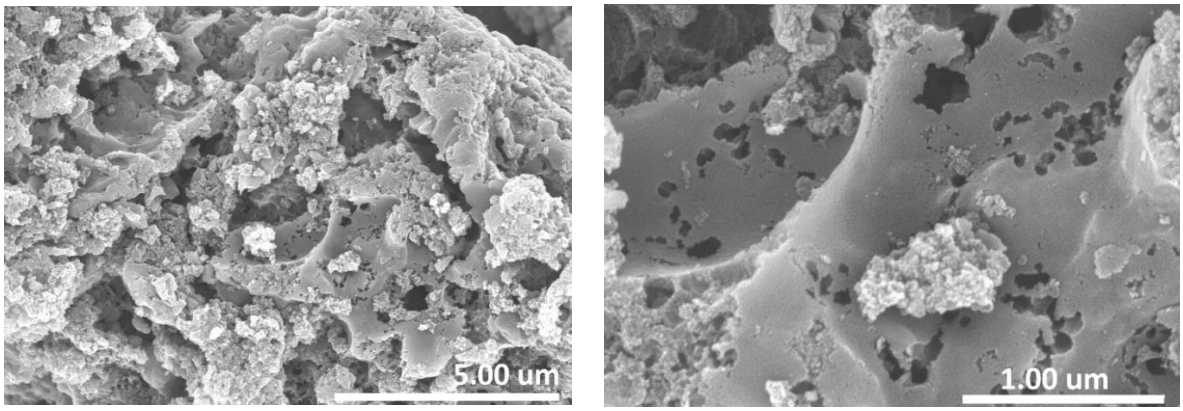
Figure 36- Nyquist plot for DSSC with Pure AC CE, AC:KOH=5:1 0 minute heated and AC:KOH=5:2 3 minute heated CE's showing the series and charge resistance.

From the FESEM images we can see that particle size of the AC is increasing after doping KOH in it. Although a particle size is increasing but a particle structure has improved. After adding KOH particles have more edge defects and pore sizes which helps in charge transfer.



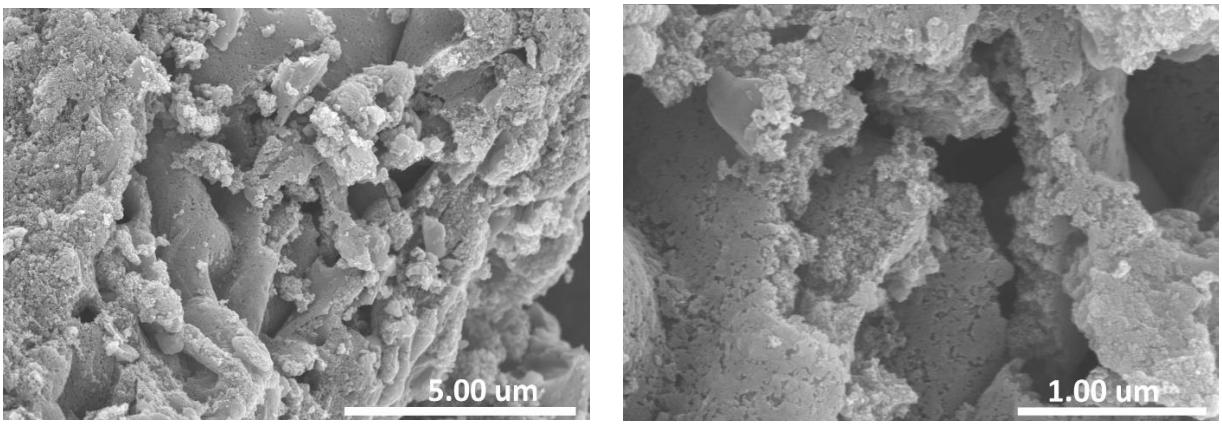
(a) (b)

Figure 37- (a) & (b) are showing FESEM images of AC without any KOH at 5 µm and 1 µm scale respectively.



(a) (b)

Figure 38- (a) and (b) are showing FESEM images of KOH doped AC for weight 5:1 with no microwave heating at 5 µm and 1 µm scale.



(a) (b)

Figure 39- (a) and (b) is showing FESEM images of KOH doped AC for 5:2 weight ratio with 3 minutes microwave heating at 5 µm and 1 µm scale.

For the first sample of pure AC we got BET surface area of 39.89 m^2/g, similarly we got BET surface of 174.71 m^2/g, 152.32 m^2/g for 5:1 KOH doped AC 0 minutes heated and 5:2 KOH doped AC 3 minutes heated respectively. BET area shows that adding KOH significantly increase the area of AC with refined pore size and pore distribution.

CHAPTER 6

CONCLUSION

In conclusion, KOH doped AC sample prepared through microwave heating process provide us inexpensive alternative as CE for DSSC instead of the Pt. When pure AC showed poor results as CE because of the less catalytic activity, adding KOH helped it to refine its structure and pore size. It showed excellent performance achieving the efficiency of 5.81%, which is better than Pt CE with the efficiency of 5.61%.

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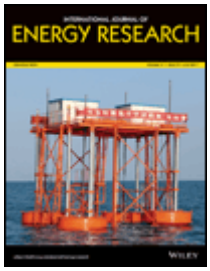

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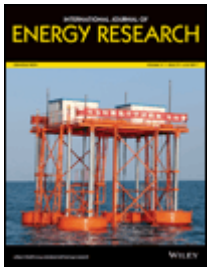

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Expected presentation date	Jul 2019

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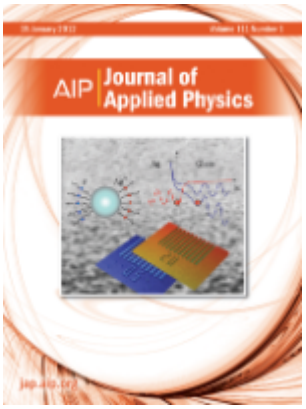

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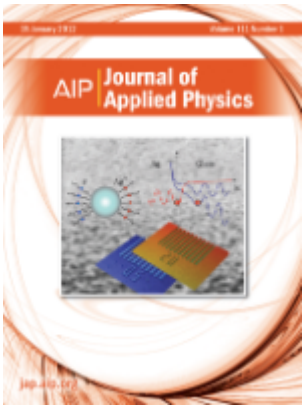

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