Innovative hybrid organic/inorganic materials for hydrogen production by solar electrolysis using low cost photovoltaic devices

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PROJECT ASSESSMENT FORM/
FICHE D'EVALUATION DES PROJETS

DATE: 2011-04-27

PROJECT TITLE/TITRE DU PROJET

Innovative hybrid organic/inorganic materials for hydrogen production by solar electrolysis using low cost photovoltaic devices

PARTNERS/PARTENAIRES

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AUTRES PARTENAIRES: UNVERSITES OU CENTRES DE RECHERCHE OU INDUSTRIELS

None

BRIEF DESCRIPTION OF THE PROJECT AND ITS SCIENTIFIC OBJECTIVES/ BREVE DESCRIPTION DU PROJECT ET DE SES OBJECTIFS SCIENTIFIQUES

Table 1: Project timeline in the proposal.

Task	1st year				2nd year				3rd year			
	Qt1	Qt2	Qt3	Qt4	Qt1	Qt2	Qt3	Qt4	Qt1	Qt2	Qt3	Qt4
HG visits Ottawa	→								→			
JD visits Valencia						→						
Perovskites & zeolite (CSIC)	→	→	→	→	→	→						
Monomer & polymer (NRC)	→	→	→	→	→	→						
Zeolite/polymer composite			→	→	→	→	→	→				
Materials characterization		→	→	→	→	→	→	→	→	→	→	→
Device design & test					→	→	→	→	→	→	→	→
Modification & optimization					→	→	→	→	→	→	→	→

The objective of this collaboration is to develop novel hybrid materials by incorporating conjugated polymers with nano inorganic semi-conductors. These materials are used as photoanodes in photovoltaic (PV) electrochemical cells for splitting water to generate hydrogen using sun light, where the inorganic nano semi-conductors are served as catalysts for effective water splitting as well as n-type materials for charge separation and transporting, and the conjugate polymers are used for promoting the sun light absorption and as p-type materials. The key issue to achieve the goal of high efficient and long term stable devices is to develop new materials for the photoanode with a strong absorption in solar spectrum and a high stability in aqueous and photo-electrochemical environments.

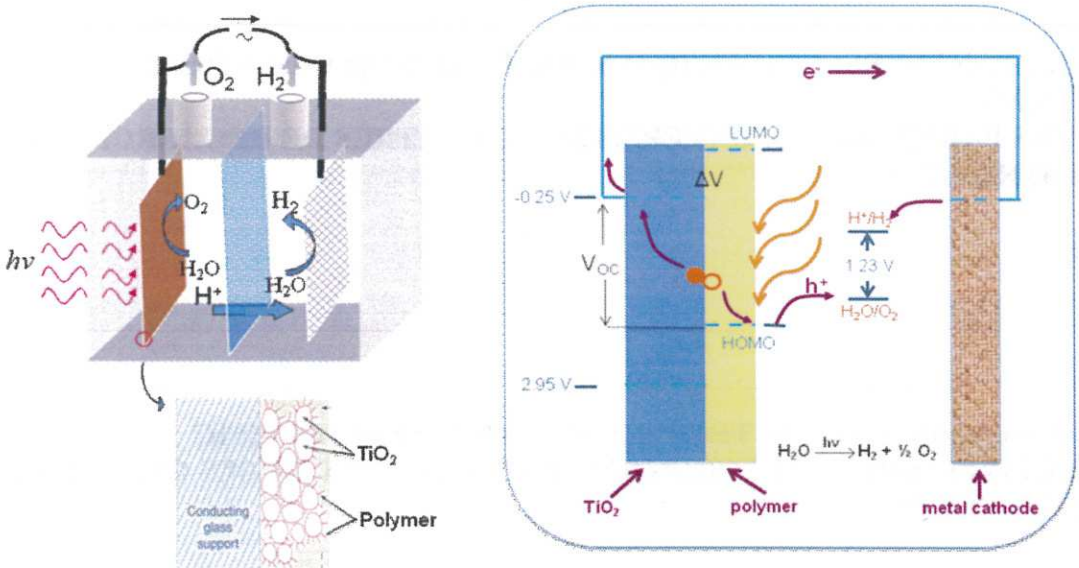


Figure 1. The design of the photovoltaic water electrolysis device.

Therefore we proposed to prepare photoanodes using zeolites, layered perovskites, periodic mesoporous metal oxides and anodized macroporous materials as hosts, with conjugated polymers or oligomers inside the pores. The inorganic hosts can play active roles as semiconductors with controlled phase arrangement and also provide spatial ordering and accessibility to the polymers. The conjugated polymer will offer wide absorption spectrum. Being encapsulated inside the porous host enhances the stability of the polymer, produces a spatial organization for efficient charge separation and vectorial charge transfer, promotes efficient mass transfer and enhances the light absorption due to the large interfacial area of the porous host. As a consequence, the formed photoanodes can be expected to have high conversion efficiency and better long term stability. The ultimate goal is to prepare photoelectrochemical cells using this series of innovative hydrid polymer/inorganic materials with power conversion efficiency above 15 % and durability better than 1000 h.

After a survey on the inorganic semiconductors regarding their energy levels of their valance band and conduction band, which should be lower than the oxidation potential and higher than the reduction potential of water respectively, as illustrated in Figure 2, their catalytic activity for water splitting and their stability under working conditions. TiO_2 has been selected as the target material as the n-type material to form composite with polymer for the photoanode preparation. Different forms of the TiO_2 including titania nano-tube film, well-dispersed nano-particles, and sintered nano-particle films are tested in CSIC-ITQ.

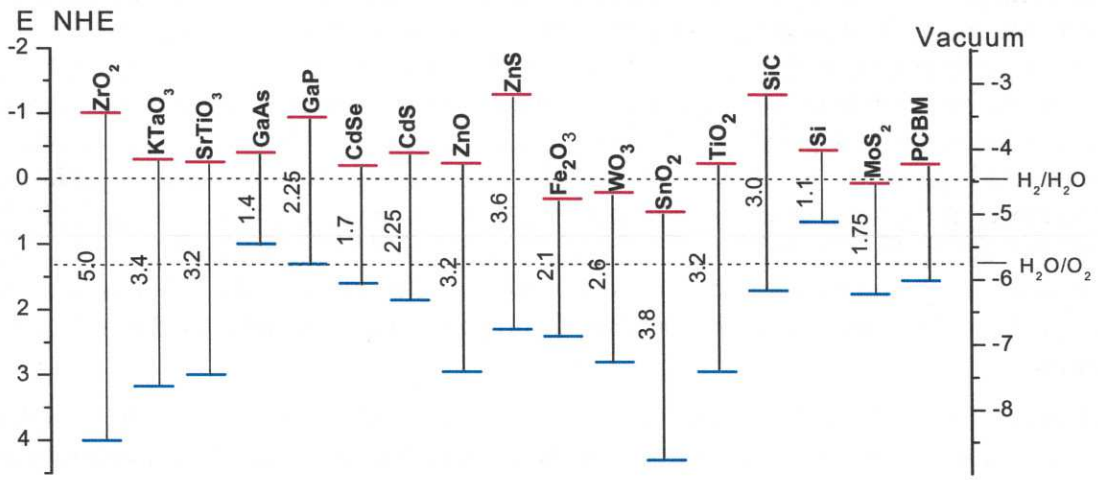


Figure 2, Inorganic semiconductors and their energy levels compared with the energy required to split water.

Conjugate polymers has been designed and prepared and the structures have been further modified at NRC-ICPET. The molecular structures of the polymers have been designed to effectively tune the optical and electronic properties, such as band gap, LUMO and HOMO energy levels, charge mobility and conductivity. Thus the polymer synthesis are focused on the following three aspects:

1) Introducing electron Donor/Acceptor (D/A) structures into the conjugated main chain to achieve a reduced energy gap in the range of 1.5 to 2.0 eV, so that the absorption can cover a large part of the solar spectrum, meanwhile to a proper LUMO level with the value about 0.3 eV higher than the conduction band of TiO_2. which are important for charge separation at the polymer/inorganic interface.
2) Introducing polar side group on the polymer to enhancing the interaction with TiO_2 nano-materials.
3) Making the polymer water soluble in order to act as a surfactant to suspend TiO_2 nano-particles in water to promote the water splitting by enhance the contact of the nano-particle with water.

Polymer/TiO_2 nanocomposites have been prepared and tested for splitting water to produce hydrogen in CSIC/ITQ. Gas evolution has been demonstrated from the PV electrolysis cells, which were prepared in our laboratory with the composite materials used as the photoanode. However, the extremely hush working condition with large amount of free radicals produced in the presence of water during the photo-electrolysis make the conjugated polymer very unstable, and thus it only can last for a short period of time. As suggested in our proposal, we expected to solve this problem by encapsulating the conjugated polymer by TiO_2 nanostructures. Therefore, TiO_2 nanotube films have been prepared, SEM study showed the film possesses promising structure to composite with organic p-type materials for PV applications (see Figure 6). Consequently great effort has been made in the preparation of the composite materials of the TiO_2 nanotube film with conjugated polymers. But the result indicates to fill the nanotubes with the conjugated polymer is almost impossible after trying many possible techniques. Therefore, at the same time to investigate the new encapsulating technique in CSIC/ITQ for the direct PV electrolysis of water to produce hydrogen, indirect solar water electrolysis by combining PV solar cells and a water electrolysis cell has also been adopted for the purpose to the produce hydrogen using sun light as the sole energy source.

Although two individual devices have to be used in the latter design, it completely removed the polymer/water contacting in the system, thus eliminated the weakest part in our original design, and finally simplify the device structure because no additional polymer protection structure is required. Two series novel conjugated polymers have been designed, prepared and used for the PV solar cell fabrication in the last year of this project (see publication: #3, 5, 6 and 7). Device efficiency close to the world best has been demonstrated. And one of the materials has also been proved simultaneously by another group in USA to be suitable for industry fabrication using printing techniques (Wei You, et.al, *J. Am. Chem. Soc.*, **2011**, *133*, 4625–4631, Wei You, et.al. Angew. Chem. 2011, 50, 2995–2998).

PROJECT STATUS AND DURATION/ETAT DU PROJET ET DUREE EFFECTIVE

Start Date/Date de démarrage	Planned completion date/Date prévue d'achèvement
July 1, 2007	Sept. 30, 2010

FUNDING/FINANCEMENT

	NRC cash & (in-kind)	CSIC cash & (in-kind)
Total funds allocated for project/Total des fonds alloués au projet	$409,030	
Funds received/Fonds reçus	66,020+137,010+140.000 +66.000 = $409,030	
Funds allocated/Fonds alloués		
Forecast/Prévision		
Breakdown of funds expended to date/Ventilation des dépenses engagées à ce jour		
Human resources/Resources humaines	159,700+160,000+ 66.000 = $385,700 (55,000+60,000+65,000) ($180,000)	
Equipment/Equipement	($28,000)	
Operations/Frais de fonctionnement	$13330 ($40,000)	
Travel/Frais de déplacement	$10,000 ($10,000)	
Other/Autres frais		
Total expenditure/Total de dépenses	409,030 + (258,000) =$667,030	

SCIENTIFIC ACHIEVEMENT/REUSSITES SCIENTIFIQUES

Results already obtained/Résultats d'ores et déjà obtenus:

- **Polymers for PV electrolysis of water:** Four series of novel conjugate polymers with ED or/and EA units in the main chain have been prepared. Their properties in terms of

energy level suiting for water splitting application were compared in Figure 3. Oxadiazole and perfluorophenyl unit demonstrate their highly potential for this application.

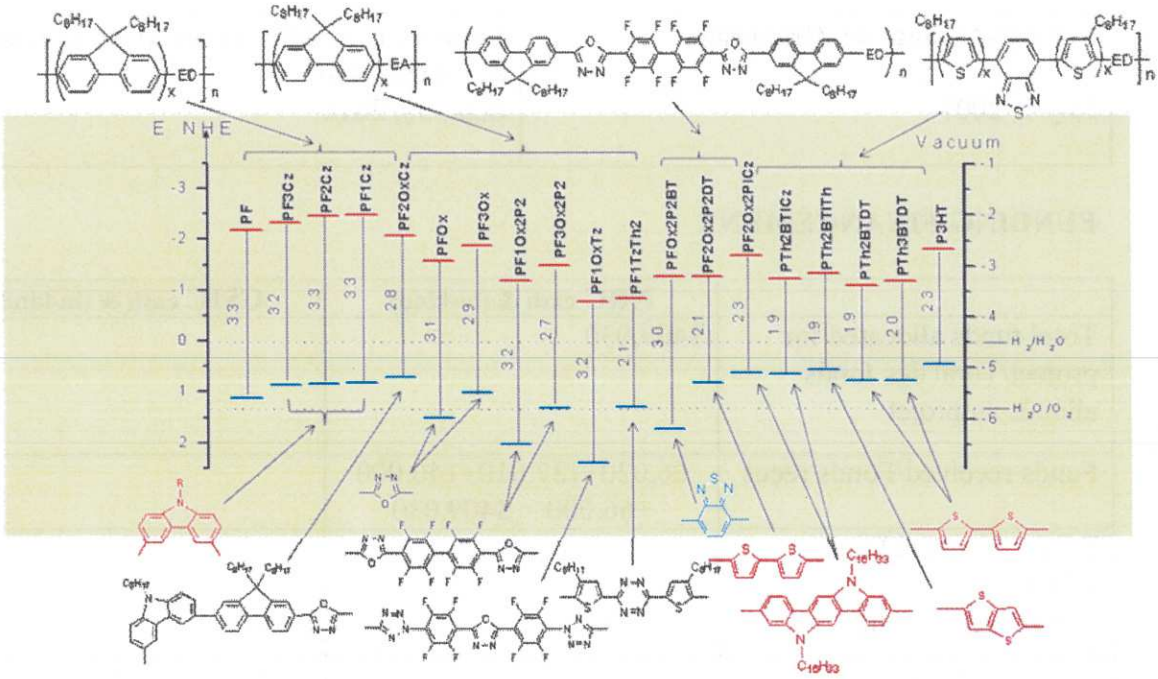
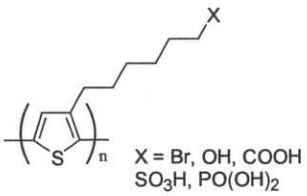


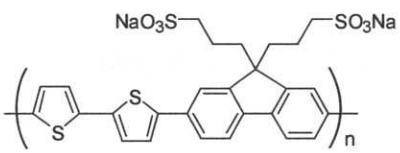
Figure 3. The comparison of LUMO and HOMO levels of the prepared 4 series of polymers and the energy requited for water splitting.

- **Polymers with polar side groups:** A series conjugated polymers with polar side groups including ether, hydroxyl, carboxylic acid, sulfonic acid, phosphoric acid have been prepared (Scheme 1). Their strong interaction with the semi-conductor nano-particles has been demonstrated. Figure 4 displayed an IR study showing the surface ligands of nano-particles can be successfully replaced by a thiophene polymer containing ether side groups due to the strong interaction of the ether side group with the nano-particle surface. This treatment is expected to enhance the charge transfer between polymer and inorganic nano-particles owing to the improved interface structure.

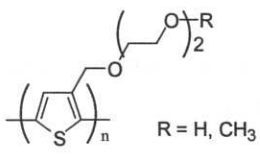
1. P(3XHT)



2. P(DTh-alt-SPF)



3. P(3DEOMT)



Scheme 1, Structures of the polymers with polar side groups.

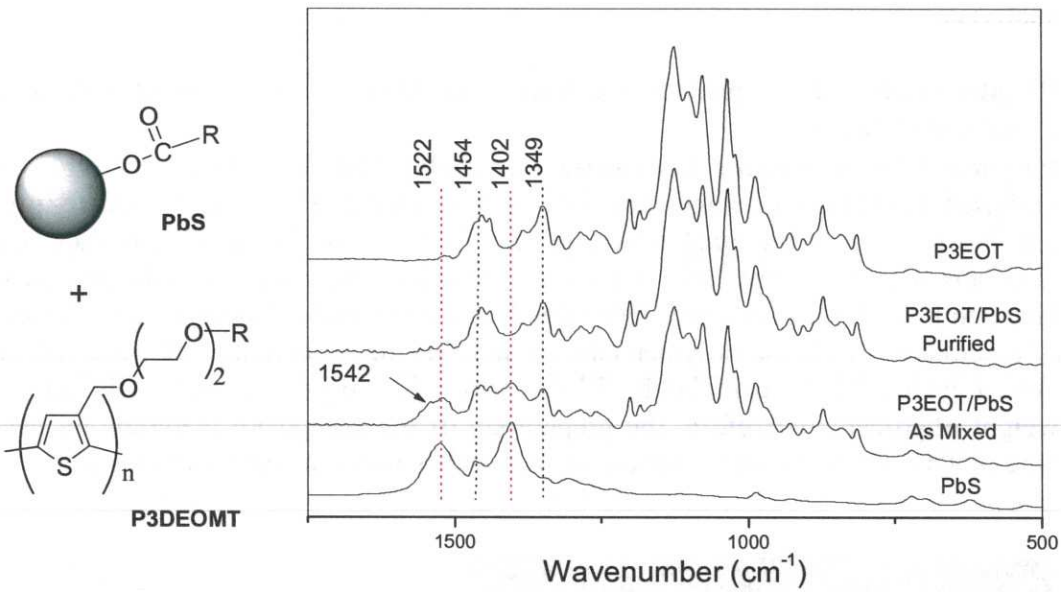


Figure 4. FT-IR spectra of PbS nano-particles, P3DEOMT, and their mixture before and after purification, the disappearance of the peaks at 1542 and 1522 cm^{-1} indicates the surface ligand (olic acid) was successful replaced by P3DEOMT.

Figure 5. Photovoltaic electrochemical water splitting system.

- **PV hydrogen generation testing system:** A testing system for photovoltaic

electrochemical water splitting has been set up in CSIC-ITQ as shown in Figure 5. Gas evolution has been demonstrated from polymer/ inorganic nano-particle composite materials

- **PV photolysis cell:** PV photolysis cell has been designed and prepared with the structure illustrated in Figure 6.
- **Polymer/TiO_2 nano-tube composite materials:** The photoanode assembly has been designed from the TiO_2 nano-tube film as illustrated in Figure 6. Therefore, great effort has been made in the preparation of the TiO_2 nano-tube films and their composite materials with polymers. TiO_2 nano-tube films have been successfully prepared both in ITQ and ICPET with the controllable depth of the nanotube. However, after tested a series of techniques to fill the polymer into the pore of the nano-tube, our work indicated it is almost impossible to completely fill the pore with polymers, even with low molecular weight oligomers. Therefore, the preparation of the composite materials by using other type of TiO_2 nano-strucutres instead of nano-tubes was considered and tested.

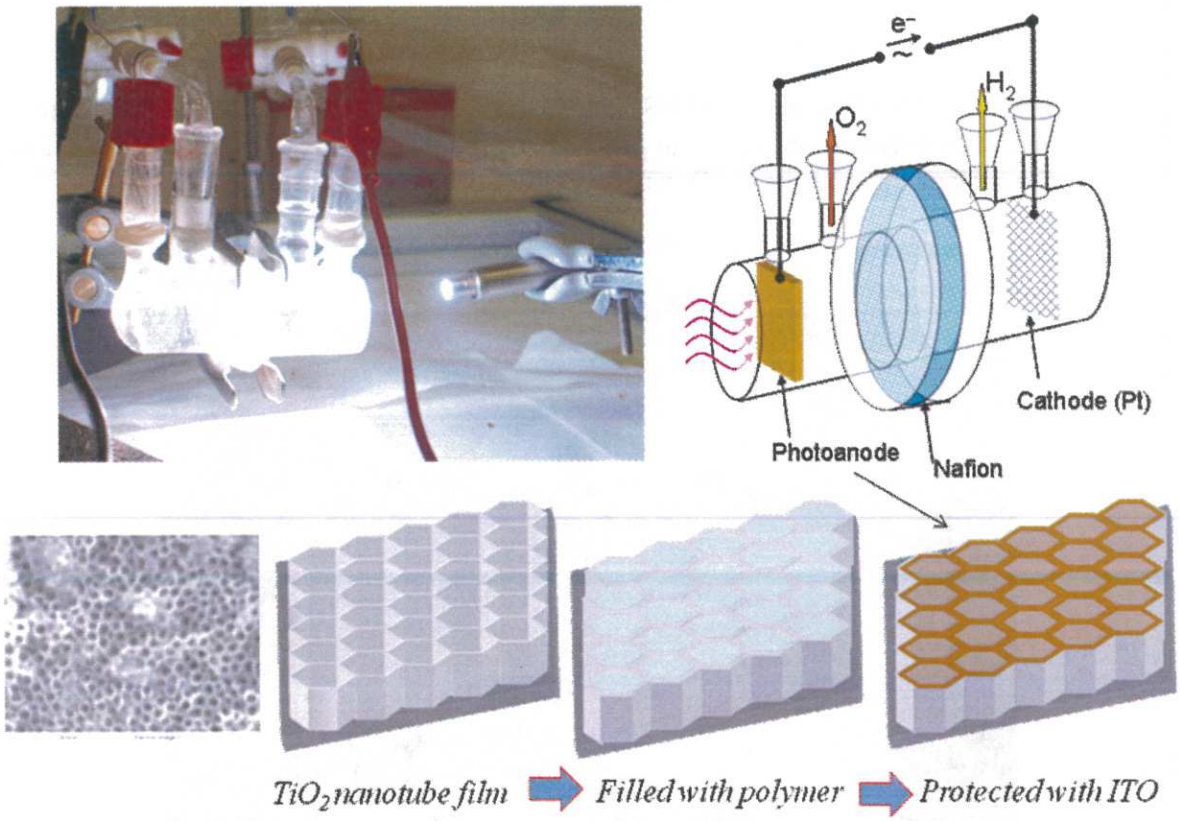


Figure 6. PV photolysis cell (top) and the photoanode prepared from TiO_2 nanotube film.

- **Polymer/TiO_2 nano-particle composite materials** Photoanode assembly has been prepared based on the sintered TiO_2 nano-particle film with the procedure shown in Figure 7. Their performance has been tested for the polymers selected from those listed Figure 3. Gas evaluation has been demonstrated using the system shown in Figure 5. The

results led to the following conclusions:

1) Copolymers containing carbazole and OH-functionalized fluorene are suitable for photoelectrochemical water splitting applications.
2) The presence of a conjugate polymer increases the efficiency of Ti-based photoanodes for overall water splitting.
3) Lamellar nano-structure of TiO_2 (Ti_LDH) prepared in CSIC-ITQ displayed a higher efficiency than the best commercial available nano-particle (P25) in the photoanode composite material with Carbazole/Fluorene copolymers (PFxCz).

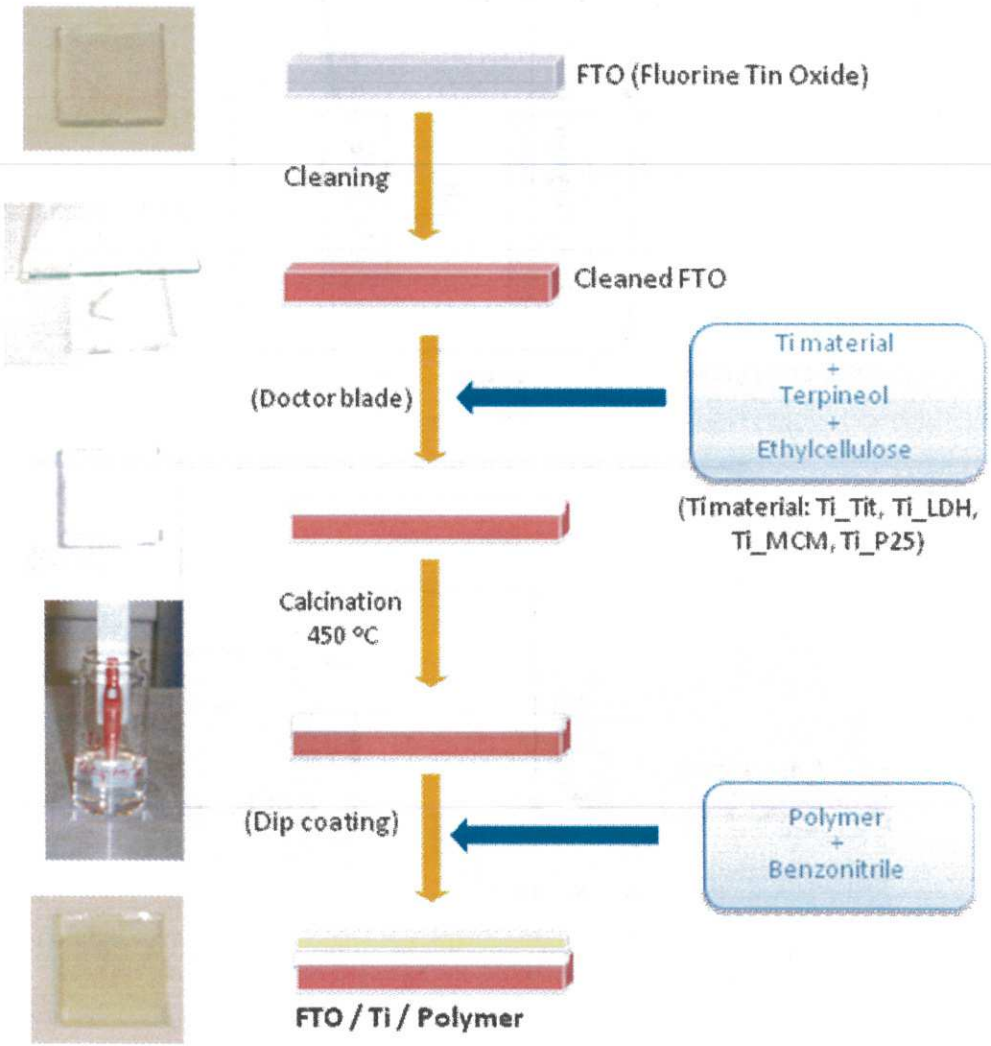


Figure 7, Processing for the preparation of the photoanode assembly based on polymer/TiO_2 composite materials.

- **The use of indirect PV water electrolysis:** We have encountered a great difficulty in the direct PV photolysis of water to use the TiO_2/polymer composite photoanode with the polymer directly contacts with water as described above. The difficulty is associated with the degradation of the polymer during testing. The produced charge in the active materials

under sun light irradiation make the polymer become extremely unstable when contacting with water. Therefore to prevent the polymer directly contact with water is a key issue to make the designed system practicable in hydrogen production. Although the encapsulation of the polymer by TiO_2 nanotube appears to be an efficient way for protecting the polymer from degradation for the photovoltaic application. It is hard to achieve due to the extreme difficulty to fill the polymer inside the nanostructures of the TiO_2.

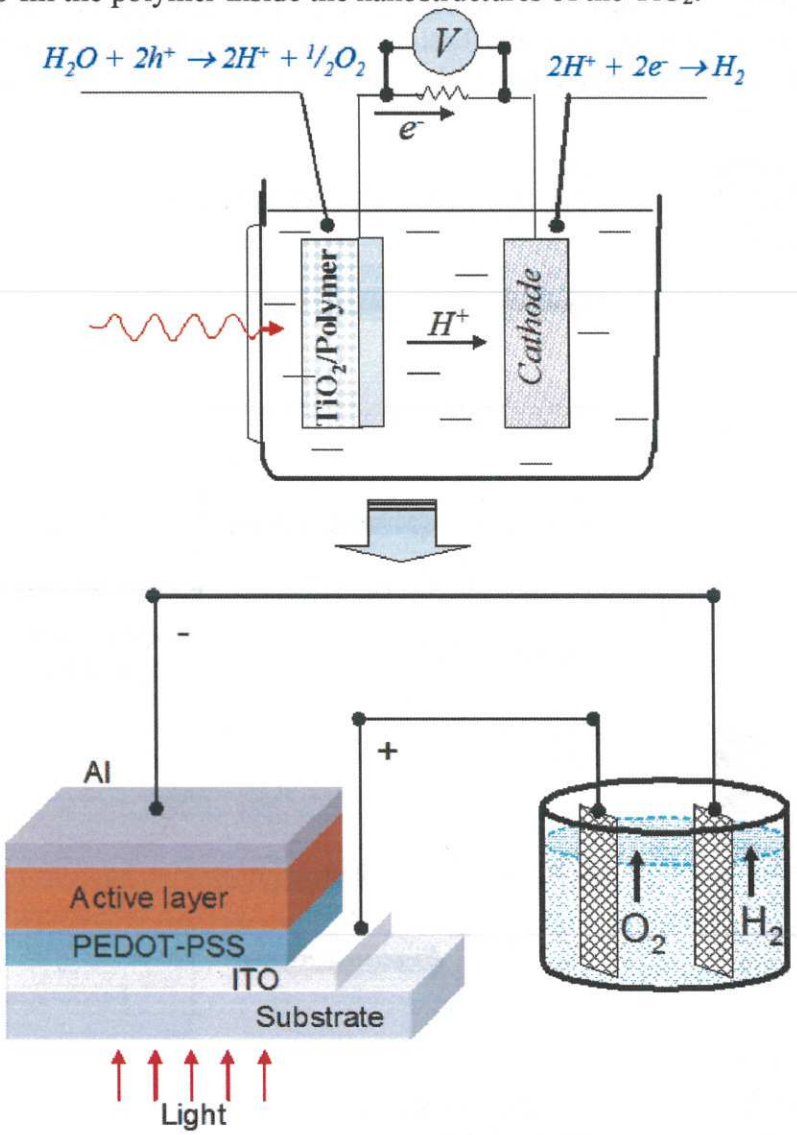


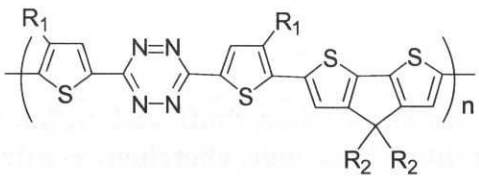
Figure 8. illustration to shift the major focus from the original design of the direct PV electrolysis of water to a new design of indriect electrolysis of water by combining a polymer solar cell and an electrochemical cell.

Therefore an alternative technique, indirectly PV electrolysis of water as illustrated in Figure 8 has been adopted for the purpose of efficiently generating hydrogen by using sun light. This design is actually use the same concept and the same device design principle as the original design. The only difference is that the two functions of the PV electrolysis cell in the original design is separated into two devices, ie. a PV polymer solar cell and a

electrochemical cell, which are communicated by an external circuit. This design will eliminate the direct contact of polymer with water, and thus removed the weakest point in the original design. This new design will also potentially reduce the cost for the whole system, because no expensive protecting structure is required. Another additional advantage of the new design is that the voltage outputted from the solar cell to the electrochemical cell is adjustable by simply combining two or more PV cells in series.

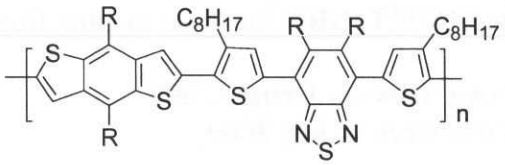
- **Polymers for PV polymer solar cells:** Two series of novel conjugated polymers (Tz based and DFBTD based) with the structures shown in Figure 9 have been prepared and used for high efficient polymer solar cells (see publication: #3, 5, 6 and 7). We have applied patents for both series of polymers Power conversion efficiency (PCE) of 5.4% was obtained for the solar cell of Tz polymers. The DFBTD polymers have been proved the most efficient polymer in the world for solar cell application with PCE up to 7.0% demonstrated by another group (Wei You, et.al, *J. Am. Chem. Soc.*, **2011**, *133*, 4625–4631, Wei You, et.al. Angew. Chem. 2011, 50, 2995–2998). The most important advantage of the DFBTD polymer is that it is suitable for the fabrication of thick film device (>200 nm). This property makes the material suitable for the industry large scale manufactory of solar panels using roll to roll printing technique. Only two polymers have been found ti have this property so far in the world (Wei You, et.al, *J. Am. Chem. Soc.*, **2011**, *133*, 4625–4631. Ye Tao, et.al. *J. Am. Chem. Soc.*, **2011**, *133*, 4250–4253).

1. Tetrazine (Tz) based polymers



P1: R_1=2-ethylhexyl, R_2=2-ethylhexyl
P2: R_1=2-ethylhexyl, R_2=hexyl
P3: R_1=hexyl, R_2=2-ethylhexyl
P4: R_1=hexyl, R_2=hexyl
P5: R_1=methyl, R_2=2-ethylhexyl

2. Difluorobenzothiadiazole (DFBTD) based polymers



P1: R = H
P2: R = F

Figure 9. Chemical structures of Tz and DFBTD based polymers.

Remaining objectives/Objectifs restant à atteindre:

1. As can been seen from Figure 2, among the tested 4 series of polymers for direct PV water electrolysis, only very limited number of the polymers showed the LUMO and HOMO level low enough to cover the water reduction and oxidation energy, which is required for efficient water splitting. To improve this property, from the polymer structure point view, stronger EA unit needs to be introduced into the polymer main chains. On the other hand, using indirect PV water electrolysis could be a much easier and ultimate way to solve this problem. In this case, the output voltage can be easily promoted by simply combining two or more solar cell together in series.

2. Although the composite polymer/inorganic nano composite material are much advanced than any other type of composite materials regarding sun light absorption, charge generation and transporting for photovoltaic applications, the low stability of the polymer materials when they directly contact with water under the photovoltaic operating condition is still an unsolved issue. It significantly shortens the life time of the photovoltaic water splitting system as those originally proposed in this project.
3. Encapsulation of polymers with nanostructure of the inorganic materials in theory can efficiently protecting polymer from degradation. However, to form this type of nano-composite is extremely difficult in practice. For ensuring an efficient conversion of the sun light to electricity, nanostructures with the size less than 50 nm are required. To fill polymers or even small molecular weight oligomers into such small cavities is almost impossible.
4. Therefore, other novel techniques have to been investigated to prepare this type of composite materials. One of the possible ways is micro-emulsion process.
5. On the other hand, inorganic/inorganic nano-composite with a p-n junction structure might be a better candidate for the direct PV water electrolysis for such system as proposed in the present project due to the higher stability of the materials.
6. Polymer/inorganic nano-composite materials are still the promising candidate for PV application due to their high sunlight absorption effciency and energy conversion. The best way to use this type of materials for splitting water to generated hydrogen is probably the indirect PV electrolysis of water such as the system presented in this report. High efficient polymers for PV solar cells have been generated from this project.

OUTCOMES/RESULTATS

Exchanges (originating organization, Senior Researcher, Ph.D student/intern, planned dates and duration)/Echanges (sens de l'echange, chercheur confirmé, thésard/stagiaire, dates et durée prévue)

1. Prof. Herme García visited Ottawa for one month in August 2007.
2. Ms. Laura Teruel, a PhD student visited ICPET, NRC for three months from July to September 2008.
3. Dr. Jianfu Ding visited ITQ, CSIC for two weeks in January 2009.
4. Prof. Herme García visited Ottawa for one week in July 2009.

Joint publications (specify date)/Publications conjointes (préciser les dates)

1. de Miguel, Maykel; Ferrer, Belen; Teruel, Laura; Garcia, Hermenegildo; Jin, Yinan; Li, Yuning; Ding, Jianfu. Photophysics of Fluorene Copolymers: Control of Fluorescence and Charge Separation by the Presence of Carbazole, Oxadiazole, or Biphenyl Units. Journal of Physical Chemistry C 2009, 113(19), 8471-8477.
2. Jianfu Ding, Maykel de Miguel, Jianping Lu, Ye Tao and Hermenegildo Garca, "Rapid Switching and High Contrast Electrochromic Property by Electrochemical Reduction of an Alternating Copolymer of Fluorene and Oxadiazole" *J. Phys. Chem. C*, **2010**, *114* 5168–5173
3. Li, Zhao; Ding, Jianfu; Song, Naiheng; Lu, Jianping; Tao, Ye, "Development of a New s-Tetrazine-Based Copolymer for Efficient Solar Cells" Journal of the

American Chemical Society (2010), 132, 13160-13161.

4. de Miguel, Maykel; Alvaro, Mercedes; Ding, Jianfu; Garcia, Hermenegildo, "Photophysics of Fluorene Copolymers Containing 1,3,4-Oxadiazole or 1,3,4-Oxadiazole and Carbazole Units" Journal of Physical Chemistry C (2010), 114(33), 14255-14260.
5. Ding, Jianfu; Song, Naiheng; Li, Zhao, "Synthesis, characterization and photovoltaic applications of a low band gap polymer based on s-tetrazine and dithienosilole" Chemical Communications (2010), 46(45), 8668-8670.
6. Zhao Li, Jianping Lu, Shing-Chi Tse, Jiayun Zhou, Xiaomei Du, Ye Tao and Jianfu Ding "Synthesis and applications of difluorobenzothiadiazole based conjugated polymers for organic photovoltaics". J. Mater. Chem. (2011) 2011, 21, 3226-3233
7. Jianfu Ding, Zhao Li, Zhe Cui,‡Gilles P. Robertson, Naiheng Song, Xiaomei Du *and Ludmila Scoles*, "The Preparation of a *Sterically Hindered s-Tetrazine and Its Conjugated Polymers*" J. Polym. Sci. Polym. Chem. (2011) submitted
8. Zhao Li, Yanguang Zhang, Sai-Wing Tsang, Xiaomei Du, Jiayun Zhou, Ye Tao, Jianfu Ding, "Alkyl Side Chain Impact on the Charge Transport and Photovoltaic Properties of Benzodithiophene and Diketopyrrolopyrrole Based Copolymers" Journal of Physical Chemistry C. (2011) submitted.

Visits, including joint participation in conferences, workshops, presentations, etc. (specify date)/Visites, incluant la participation commune à des colloques, ateliers et présentations (préciser dates)

- Prof. Herme Garcia gave a presentation to ICPET-NRC on the photovolatic solar cell research during his visiting in 2007.
- Dr. Jianfu Ding gave a presentation to ITQ, CSIC on polymers for photovoltaic applications in January 2009.
- "Conjugated Polymers Containing Strong Electron Acceptors for Photovoltaic Applications" 34th Canadian High Polymer Forum (2011, Ste. Adele, Qc, Canada)
- "Design, Synthesis and Characterization of Low Band Gap Polymers for Organic Solar Cells" 34th Canadian High Polymer Forum (2011, Ste. Adele, Qc, Canada)

Teaching/Enseignment

- 4 post doctoral research associates (two for two years and two for 10 months) have been trained in ICPET, NRC.

Technology transfers, patents and licences, contracts with industry/Transferts de technologie, brevets et licences, contrats avec l'industrie

- PCT patent application: PCT/CA2010/001732 "Fluorinated Monomers, Oligomers and Polymers for Use in Organic Electronic Devices"
- U.S. provisional patent application 61/361,637, "Tetrazine Monomers and Copolymers for Use in Organic Electronic Devices".

FUTURE RESEARCH PLANS/PLANS DE RECHERCHE FUTURS

This is the final report for the present project. Therefore no future plan has been made within this project. But we like to summarize some major points obtained in this project, which we believe will be valuable for future research in this field.

1. Although the composite polymer/inorganic nano composite material are much advanced than other type of composite materials in sun light absorption, charge generation and transporting for photovoltaic applications, the low stability of the polymer materials when they directly contact with water under the photovoltaic operating condition is still an unsolved issue. It significantly shortens the life time of the photovoltaic water splitting system as those originally proposed in this project.
2. Encapsulation of polymers with nanostructure of the inorganic materials in theory can efficiently protecting polymer from degradation. However, to form this type of nano-composite is extremely difficult in practice. For ensuring an efficient conversion of the sun light to electricity, nanostructures with the size less than 50 nm are required. To fill polymers or even small molecular weight oligomers into such small cavities is almost impossible.
3. Therefore, other novel techniques have to been investigated to prepare this type of composite materials such as micro-emulsion process.
4. On the other hand, inorganic/inorganic nano-composite with a p-n junction structure might be a better candidate for the direct PV water electrolysis for such system as proposed in the present project due to the higher stability of the materials.
5. Polymer/inorganic nano-composite materials are still the promising candidate for PV application due to their highly efficiently sunlight absorption and energy conversion. The best way to use this type of materials for splitting water to generated hydrogen is the indirect PV electrolysis of water such as the system presented in Figure 8. High efficient polymers for PV solar cells have been generated from this project.

BENEFITS OF COLLABORATION/AVANTAGES DE LA COLLABORATION

The development of an efficient and sustainable process for hydrogen production has become a bottle neck in the development of hydrogen economy. It is evident that the use of solar energy for hydrogen generation will potentially generate large economic benefits and enormous social impact. NRC and CSIC have been identified as the leading organizations all over the world for fuel cell and solar energy conversion researches. This project combines the existing strengths from the both sides to establish an innovative and original strategy to solve the fuel supply problem for fuel cells from a completely renewable energy source. The output of this project has strengthen the leading position of both organizations and significantly impacts the development of the solar energy conversion. Proprietary rights on efficient materials for water splitting will give NRC and CSIC a prominent position in new technologies based on the use of hydrogen.

Furthermore, the research teams on both sides have wide collaborations inside and outside CSIC and NRC on the applications of the developed materials in opto-electronic and fuel cell application. The team of CSIC works with REPSOL and some other companies potentially interested in solar energy conversion. The team of NRC has collaborated with about 15 groups inside NRC and from Canadian universities for the photovoltaic solar cells researches. They also networked with many other teams and industry whose works cover areas including solar cells, hydrogen storage, catalyst, and proton exchange membrane for fuel cells and so on. The output from this project starts to create impact in the solar cell and printable electronic areas and industry connection is been building up. This cooperative project is also based on common interests and complementarities and the main result of this collaboration is to produce a synergy increasing the quality and impact of the joint research as compared to that of the teams working individually. Thus, this collaboration serves to strengthen the long term Spanish-Canadian cooperation.

One of the major benefits of this project is to train and the exchange young researchers between the teams. Several PhD students and postdoctoral fellows worked under this project. In this way a long-term benefit of this project can be guaranteed.

PROBLEMS AND DIFFICULTIES ENCOUNTERED IN THE IMPLEMENTATION OF THE PROJECT/PROBLEMES ET DIFFICULTES RENCONTREES POUR LA MISE EN ŒUVRE DU PROJET

See above "Remaining objectives" and "Further research plan".

OTHER COMMENTS/COMMENTAIRES ADDITIONNELS

See further research plan.

ASSESSMENT REVIEWED AND APPROVED BY THE DIRECTOR GENERAL OF (name of NRC institute)/EVALUATION REVUE ET APPROUVEE PAR LE DIRECTEUR GENERAL DE (nom de l'institut)

	26.04.2011
Director General/Directeur général	**Date**