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# TiO<sub>2</sub> as a low cost, multi functional material

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#### Abstract

 $TiO_2$  is a technologically important, naturally occurring, low cost material used in energy, environmental, health and many other applications. This paper reports the possibility of using commercial  $TiO_2$  in several different applications. The efficiency of a silver nanoparticle incorporated  $TiO_2$  dye sensitized solar cell increased by 27% evidently due to the plasmonic effect. In a dye sensitized solar cell when nanopowder  $TiO_2$  photoanode was replaced by a composite  $TiO_2$  photoanode containing  $TiO_2$  nanopowder and  $TiO_2$  nanofibres, the cell efficiency enhanced by more than 30%. In an electrochromic device of configuration, FTO glass/ $TiO_2$ /Poly (methylmethacrylate) electrolyte/SnO<sub>2</sub>/FTO glass, an impressive reversible colour change was observed. Mg rechargeable battery fabricated with a  $TiO_2$  cathode exhibited a discharge capacity of 220 mAh g<sup>-1</sup> showing that Mg<sup>++</sup> can be reversibly intercalated in to the  $TiO_2$  structure. An electrospun nanofibre membrane prepared from a biodegradable polymer and functionalized with  $TiO_2$  nano powder was capable of removing arsenic (As) from drinking water efficiently.

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#### 1. Introduction

Titanium dioxide or  $TiO_2$  is an environmentally benign, technologically important and naturally occurring, low cost material used in photo catalysis and solar cells, electronic and optical devices, air and water purification and in numerous medical and technological applications. Titanium dioxide occurs in nature as well-known minerals rutile, anatase and brookite. It is mainly sourced from ilmenite ore. This is the most widespread form of titanium dioxide-bearing ore around the world. Rutile is the next most abundant and contains around 98% titanium dioxide in the ore. Anatase and rutile have a tetragonal structure, while the structure of brookite is orthorhombic. Degussa P25, the most commonly used commercial photocatalyst is a mixture of anatase and rutile (3:1) and shows much higher photocatalytic activity than pure anatase or rutile.  $TiO_2$  is a semiconductor and its molecular orbitals can be approximated with bands, out of which the valence band (highest occupied, HOMO) and the conductive band (lowest unoccupied, LUMO) are separated by the energy gap of 3.2 eV for anatase and 3.0 eV for rutile. Transmission electron microscopy shows that in Degussa, P-25  $TiO_2$  powder the anatase and rutile particles have separately formed their agglomerates and the average sizes of the anatase and rutile elementary particles are 85 and 25 nm respectively.

Titanium dioxide exhibits good photocatalytic properties and hence it is used in antiseptic and antibacterial applications to degrade organic contaminants and germs. It is also used as a UV-resistant material, a photosensitive material in the manufacture of printing ink and inself-cleaning ceramics and glass coating etc.  $TiO_2$  is also widely used in making cosmetic products such as sunscreen creams, whitening creams, morning and night creams and skin milks. It is used in the paper industry for improving the opacity of paper. Since the beginning of the  $20^{th}$  century, $TiO_2$  has been used extensively in the paint industry as well as in the paper and plastic industry which accounts for about 80% of the world's titanium dioxide consumption [1].

In 1972 Fujishima and Honda discovered the photocatalytic splitting of water using  $TiO_2$  materials [2]. In the recent past many new applications from photovoltaics to photo-electrochromics, sensors and energy storage as well as self cleaning surfaces for air refreshing or medical use have been reported for  $TiO_2[3,4,5,6]$ . Most of these applications can be roughly divided into "energy" and "environmental" categories, many of which depend not only on the properties of the  $TiO_2$  material itself but also on the modifications of the  $TiO_2$  material host (e.g. with inorganic and organic dyes) and on the interactions of  $TiO_2$  materials with the environment.

 $TiO_2$  based materials are promising as negative electrodes for the energy storage in lithium ion and magnesium ion batteries [7]. They benefit from properties like low cost, protection against Li and Mg plating, fast charge/discharge, thermal stability, excellent cycling stability due to small volume changes and good capacity. Therefore, it is a capable candidate for thin film all solid state and polymer batteries.

In this paper we report our own research findings on the use of  $TiO_2$  in several different areas.  $TiO_2$  nano powder is the most widely used and best-performing anode in mesoscopic dye sensitized solar cells due to its band structure and a reasonable conductance of nanostructured  $TiO_2[8]$ . The efficiency of dye sensitized solar cells was significantly enhanced by plasmonic effect by incorporating silver nanoparticles into the  $TiO_2$  structure [9]. In dye sensitized solar cells of configuration  $FTO/TiO_2$  anode/electrolyte/Pt/FTO, the use of a composite photoanode made from a  $TiO_2$  NP/NF/NP sandwich structure consisting of  $TiO_2$  commercial paste (NP) and  $TiO_2$  electrospun nanofibres (NF), the solar cell efficiency was significantly increased. In an electrochromic display device (ECD) of configuration, FTO glass/TiO<sub>2</sub>/Poly (methyl methacrylate) (PMMA) electrolyte/SnO<sub>2</sub>/FTO glass, an impressive reversible colour change between blue and colourless states was observed with impressive coloration efficiency [10]. Mg<sup>++</sup> ion rechargeable batteries fabricated with cell configuration Mg/PEO:EC:PC:Mg(CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub>/TiO<sub>2</sub>-C using a PEO based gel polymer electrolyte and TiO<sub>2</sub> cathode exhibited an impressive discharge capacity. An electrospun nanofibre membrane prepared from a biodegradable polymer and functionalized with TiO<sub>2</sub> nano powder was capable of removing arsenic from drinking water efficiently [11].

#### 2. Plasmonic dye sensitized solar cells with Ag nanoparticle incorporated TiO<sub>2</sub> anode

In this study, we investigated the use of silver nanoparticles (NPs) incorporated in  $TiO_2$  photoanode for the enhancement of the photocurrent of the DSSC by the surface plasmon resonance effect. Ag NPs with 10-20 nm size were synthesized as a nanoparticle colloidal suspension following the procedure described by Ratyakshi et al [12].

Photo-anodes were prepared by Doctor Blade technique, starting from P25 TiO<sub>2</sub> Digussa powder by mixing different volumes of Ag suspension solution. Photoanodes were sensitized by soaking them in 3 ml of ethanolic solutions of the Ruthenium dye N535at room temperature for 24 h. The effect of Ag NPs on the DSSCs was investigated using UV-VIS spectroscopy and *J*-*V* characteristic of DSSCs. It was found that the energy conversion efficiency of the DSSC was dependent on the amount of Ag NPs incorporated. The electrolyte solution for the DSSCs was prepared by adding tetrapropyl ammonium iodide ( $Pr_4NI$ ) and  $I_2$  to a pre-cleaned volumetric flask containing molten ethylene carbonate (EC) and acetonitrile. The solution mixture was stirred overnight. DSSCs were fabricated by sandwiching the above electrolyte solution containing  $I'I_3^-$  redox couple in the configuration FTO/TiO<sub>2</sub>/dye/electrolyte/Pt/FTO. The photo current - voltage (*I-V*) characteristics of the cells were measured under the illumination of 100 mW cm<sup>-2</sup> (AM 1.5) simulated sunlight.

The short circuit photocurrent density,  $J_{sc}$  open circuit voltage,  $V_{oc}$ , the fill factor, *FF* and the efficiency,  $\eta$ , derived from photo current voltage measurements of the three types of DSSCs are summarized in the Table 1. When preparing these DSSCs, it was made sure to have the same TiO<sub>2</sub> film thickness of~5 µm and the same amount of dye loading. As seen from Table 1, the presence of Ag NPs in TiO<sub>2</sub> anode has enhanced the solar cell efficiency due to improved short circuit photo current density. The short circuit photocurrent density and the efficiency of the DSSC have increased from 11.36 mA cm<sup>-2</sup> and 5.12 % to 13.86 mA cm<sup>-2</sup> and 6.51 % respectively due to the presence of Ag nanoparticles in the TiO<sub>2</sub> photoanode. The enhancement of DSSC efficiency due to the Ag NPs was 27%.

Table 1. Photovoltaic parameters of the plasmonic DSSCs with (A) TiO2 and (B) TiO2-Ag NP photoanode.

DSSC Photoanode	$J_{\rm sc}~({\rm mA~cm}^{-2})$	$V_{\rm oc}~({ m mV})$	FF %	η %
(A) TiO <sub>2</sub>	11.36	771.3	58.42	5.12
(B) TiO <sub>2</sub> -Ag NP	13.86	729.1	64.37	6.51

When the photovoltaic parameters of the two DSSCs (A) and(B) shown in Table 1 are compared, one can clearly see that the short circuit photocurrent density  $(J_{sc})$ , the fill factor (FF) and the energy conversion efficiency  $(\eta)$  values of the plasmonic DSSC (B) have increased but the open circuit voltage  $(V_{oc})$  value has decreased. Based on research findings reported in the literature on plasmonic DSSCs, it is clear that the Ag NPs incorporated plasmonic DSSCs exhibit two processes: (a) high optical absorption characterized by a red shift in the incident photon to current conversion efficiency (IPCE)<sub>max</sub> peak and an significantly enhanced optical absorption induced by the surface Plasmon resonance effect, enhancing the short circuit current density,  $J_{sc}$  and the efficiency and (b) low open circuit voltage,  $V_{oc}$  resulting from energy band gap narrowing due to the presence of metal NPs in TiO<sub>2</sub> and reduced electron life times increasing the electron recombination with redox species at the TiO<sub>2</sub>/electrolyte interface [13].

#### 3. Dye sensitized solar cells with TiO2nanoparticle/nanofibre/nanoparticle compsite photoanode

A TiO<sub>2</sub> film of thickness about 3  $\mu$ m was prepared by spreading commercial (Solaronix D) titania paste on a precleaned FTO glass plate using the doctor blade technique and sintering at 250 °C for 30 min to obtain a thin, porous TiO<sub>2</sub> layer. TiO<sub>2</sub> nanofibres were electrospun directly onto the nanoparticle layer from a mixture containing poly(vinylacetate), titanium(IV) isopropoxide (TiP) and acetic acid as a catalyst for sol-gel reaction in N-N dimethylformamide (DMF). Electrospinning was done by a NaBond electrospinner (NaBond Technologies, Hong Kong). The thickness of the nanofiber membrane was varied by varying the duration of electrospinning as 10 min, 15 min, 20 min, 25 min and 30 min. The sintering of the TiO<sub>2</sub> nanofibre membrane was carried out stepwise at each temperature as: 100 °C (15 min), 150 °C (15 min), 325 °C (5 min) and 450 °C (1h) in air. Subsequently commercial (Solaronix D) titania paste was coated on the electrospun TiO<sub>2</sub> nanofibre membrane by the doctor blade technique and the TiO<sub>2</sub> NP/NF/NP three layer composite structure was sintered again in air at 450 °C for 45 minutes. Fig. 1 shows a schematic diagram of the TiO<sub>2</sub> nanofibre layer, which was turned to "elongated beads" after sintering was about 1.5 µm and the thickness of the final TiO<sub>2</sub> nanoparticle layer was also about 1.5 µm.



Fig.1. Schematic diagram of the TiO<sub>2</sub> nanoparticle/nanofibre/nanoparticle composite electrode.

DSSCs were fabricated with a liquid electrolyte consisting of 0.738 g of tetrapropyl ammonium iodide, 0.06 g of iodine, 3.6 ml of ethylene carbonate (heated to melt) and 1.0 ml of acetonitrile. Photovoltaic characteristics of the DSSCs were measured by using a solar simulator under the illumination of 1000 W m<sup>-2</sup> using a Xenon 500 lamp with an AM 1.5 filter. In Table 2 the short-circuit photocurrent density ( $J_{SC}$ ), the open-circuit voltage ( $V_{OC}$ ), the fill factor (*FF*) and the energy conversion efficiency ( $\eta$ ) of the best solar cell fabricated with TiO<sub>2</sub> NP/NF/NP composite photoelectrode of thickness about 6.0 µm are compared with corresponding parameters of the solar cell made with TiO<sub>2</sub>NP layer (thickness about 6.0 µm) alone. The TiO<sub>2</sub> NF layer in the composite electrode corresponds to 20 min of electro spinning time.

Table 2. Photovoltaic parameters of the DSSCs with TiO<sub>2</sub>single layer nanoparticle electrode and TiO<sub>2</sub>NP/NF/NP three layer composite electrode with optimized TiO<sub>2</sub> nanofibre layer thicknesses by using 20 min. electro spinning time.

Photoanode	$J_{\rm SC} ({\rm mA  cm^{-2}})$	V <sub>OC</sub> (mV)	FF (%)	η (%)
TiO <sub>2</sub> NP	11.24	710.6	67.3	5.38
TiO <sub>2</sub> NP/NF/NP	15.75	714.6	63.0	7.09

The increased light harvesting by scattering within the  $TiO_2$  nanofibre (elongated beads) medium is the quite likely possibility for the observed increase in photocurrent and enhanced efficiency of the DSSC compared to the DSSC with an electrode made with  $TiO_2$  nanoparticles alone [14]. The light trapping is enhanced due to the sandwich-type photo anode structure where the light scattered in the nanofibre layer is again used by the two nanoparticle layers on either side, thereby contributing to enhanced generation of additional photoelectrons. By UV-VIS absorption experiments of the dyed composite photoanode and the dyed normal  $TiO_2$  nanoparticle photoanode, we have observed that the amount of dye loading of these two photoanodes are essentially the same and therefore, the improved  $J_{sc}$  of the composite photoanode is not due to dye loading effect.

From this study, it can be suggested that, under the same preparation conditions and with the same photoanode thickness, DSSCs made from  $TiO_2$  nanopartice/nanofiber/nanoparticle composite photoanode possess significantly higher solar cell efficiencies by as much as 32 %, compared to DSSCs made from  $TiO_2$  nanoparticles alone

#### 4. Electrochromic devices based on TiO<sub>2</sub>

Electrochromism is the reversible and visible change in optical transmittance/reflection that is associated with an electrochemically induced oxidation-reduction reaction. The colour change is commonly between transparent (bleached) state and a coloured state or between two coloured states. A typical electrochromic device (ECD) consists of a three layer, "sandwich-type" configuration in which an electrochromic electrode and a counter-electrode (CE) are separated by a liquid or solid or gel electrolyte. Colour changes occur by charging/ discharging this electrochemical cell through the application of an electrical potential [15].

Most EC devices consist of expensive metal oxides such as  $WO_3$ , NiO, MnO<sub>3</sub> or IrO<sub>2</sub> as the electrochromic material (active electrode) and CeO<sub>2</sub> as the counter electrode. In this section of the paper we report the possibility of using a transparent nanoparticulate TiO<sub>2</sub> film as the electrochromic material and SnO<sub>2</sub> as the counter electrode separated by polymethyl methacrylate (PMMA) based gel polymer electrolyte in a novel design of an ECD.

In order to prepare the ECD electrolyte used in this study, a known amount of PMMA (Aldrich) was dispersed in propylene carbonate (PC) and ethylene carbonate (EC) co-solvent and the mixture was heated up to 50 °C under magnetic stirring for about five minutes for complete dissolution of the polymer. Known amounts of LiClO<sub>4</sub>,

LiCF<sub>3</sub>SO<sub>3</sub> and ionic liquid 1-ethyl-3-methylimidazolium-bis (trifluoromethylsulfonyl) imide (EMITFSI) were added to this solution under continuous stirring until the gel electrolyte is formed.

To prepare the electrochromic layer, a compact high density nanocrystalline TiO<sub>2</sub> film of thickness ~ 100 nm was first spin coated on a pre-cleaned fluorine-doped conducting tin oxide (FTO) glass plate and sintered at 450  $^{\circ}$ C for 45 minutes. Mesoporous nanocrystalline TiO<sub>2</sub> film was coated on the top of the above compact layer using TiO<sub>2</sub> paste (Solaronix T) by the "doctor blade" method. This EC structure was sintered again at 450 °C for 45 minutes and cooled down to room temperature and used to fabricate the EC device. Transparent conductive SnO<sub>2</sub> films were deposited on FTO glass substrates by spin coating a colloidal solution of SnO<sub>2</sub> at 5000 rpm for 1 minute and sintering at 550 °C for 30 minutes followed by cooling down to room temperature. ECDs with the configuration *FTO glass/TiO<sub>2</sub>/polymer electrolyte/SnO<sub>2</sub>/FTO glass* were fabricated by sandwiching the polymer electrolytes between the TiO<sub>2</sub> and SnO<sub>2</sub> electrodes.

Temperature dependence of the bulk electrical conductivity of the polymer electrolytes were determined by complex A.C. impedance spectroscopy. The UV-visible optical spectra of the ECDs were recorded with a Shimadzu 2600 spectrophotometer in the wavelength range between 190 and 1100 nm. The temperature dependence of the conductivity followed the classical Arrhenius behaviour according to the equation  $\sigma = \sigma_0 \exp(-E_a/kT)$  where  $\sigma$  is the ionic conductivity,  $\sigma_0$  is a constant,  $E_a$  is the activation energy and k is the Boltzman constant. Figure 2 shows three different states of the EC device: (a) as prepared, (b) partially coloured and (c) fully coloured.



Fig.2. Three different states of the devices (a) as prepared (applied voltage, 0 V), (b) partially coloured (applied voltage 2.2 V), (c) fully coloured (applied voltage 4.0 V).

Electrochemical status of the device was changed by applying different voltages and the average transmittance of the electrochromic devices with the configuration, *FTO glass* /*TiO*<sub>2</sub>/*polymer electrolyte* (*PMMA*) /*SnO*<sub>2</sub>/*FTO glass* at different voltage status in the visible region are shown in Fig. 3. The device showed more than 65% transmittance in its bleached state and 1% of transmittance in its coloured state due to the reduction of TiO<sub>2</sub> and the simultaneous insertion of Li<sup>+</sup>.



Fig.3. Transmittance spectra of ECD with configuration FTO glass/TiO2/polymer electrolyte (PMMA) /SnO2/FTO glass at various voltages.

Preliminary testing of the stability of these EC devices was done by cycling them between the coloured and bleached states at potentials of -0.25 V and +4.0V (90 s) respectively. When applying a higher potential (4 V) between the two electrodes, the ECD did not come to its initial state. No loss in the transmittance responses and the stability of the device was observed even after 1000 cycles.

This work shows that  $TiO_2$  and  $SnO_2$  are promising materials to be used in ECDs because of their low cost and the easy preparation techniques involved.

#### 5. TiO<sub>2</sub> as a cathode material for Mg<sup>++</sup> ion rechargeable batteries

In the energy storage scenario, rechargeable batteries appear to offer the best solutions available at present. In the field of modern advanced power sources, lithium-ion (Li-ion) batteries are the systems of choice, offering high energy density, flexible and lightweight design, and longer lifespan than comparable battery technologies. However, a major concern is the limited availability of lithium metal in the earth's crust, thereby making the cost of lithium batteries to go up rapidly in the near future. Relative abundance of Mg, Ti and Li metals in earth's crust is Mg: 29,000 ppm, Ti: 6600 ppm and Li: 17 ppm. Therefore, the possibility of developing a rechargeable Mg<sup>++</sup> ion batteries have received a considerable attention in the recent past [16,17].

In this work we have studied the possibility of using low cost TiO<sub>2</sub> as the cathode martial in magnesium ion batteries comprising a quasi-solid polymeric electrolyte based on polyethylene oxide (PEO) as the host matrix. The polymer electrolyte based on PEO complexed with Mg (CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub>, in ethylene carbonate (EC) and propylene carbonate (PC) co-solvent has been characterized using AC impedance spectroscopy, Cyclic Voltammetry (CV) and DC polarization measurements. With visually best mechanical properties and optimized composition of PEO (12.20 wt%), (CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub>Mg (14.6 wt%), EC (36.6 wt%) and PC (36.6 wt%) the ionic conductivity of the electrolyte as extracted from complex impedance measurements was in the order of  $2.52 \times 10^{-3}$  S cm<sup>-1</sup> at room temperature. The ionic conductivity increased with temperature following the usual Arrhenius behavior and showed a value of  $3.44 \times 10^{-3}$  S cm<sup>-1</sup> at 60 °C. The estimated values of Mg<sup>+</sup> ion transference number and the total ionic transference number were 0.21 and 0.98 respectively.

The batteries with cell configuration Mg/PEO:EC:PC:Mg(CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub> /TiO<sub>2</sub>-C were fabricated by varying the amount of carbon in the cathode until it gave the best battery performance. The best battery exhibited a discharge capacity of 220 mAh g<sup>-1</sup> with an open circuit voltage of 1.85 V and a reasonably good cycling capacity. Charge/discharge cycling characteristics of the Mg<sup>++</sup> ion battery are shown in Fig. 4.



Fig.4. Charge - discharge curves of a Mg/GPE/TiO2-C cell at room temperature.

#### 6. TiO<sub>2</sub> as a functional material for the removal of arsenic (As) from drinking water

Arsenic contamination in groundwater has become a serious threat to environmental and human health. $TiO_2$  nanoparticles have the ability to remove many toxic metals, including As from water by surface adsorption [18]. Electrospun nanofibres made from a biodegradable polymer, incorporating  $TiO_2$  nanoparticles were prepared using an electrospinning solution containing the polymer, acetic acid acetone, DMSO and commercial  $TiO_2$  powder (P25). The voltage applied between the syringe needle and the drum collector was 17.50 kV and the distance between the needle and the drum was kept at 7.0 cm. The drum speed was 106 rps and the injection rate was 2 ml/h. The polymer nanofibre membrane was deposited on tea bag paper substrate to provide an added mechanical strength to the polymer nanofibre membrane.

A stock solution containing As at 41.5 ppb concentration was prepared using DI water. The  $TiO_2$  incorporated polymer nanofibre membrane was soaked in the arsenic stock solution and left for different time periods to allow As to be adsorbed by the  $TiO_2$  powder incorporated into the polymer nanofibre matrix. The remaining arsenic concentration in the solution was measured using Atomic Absorption Spectroscopy (AAS).



Fig. 5. Electrospun polymer nanofibre membrane incorporating TiO<sub>2</sub>P25 nanoparticles.

From this study it was observed that polymer nanofibre membrane with  $TiO_2P_{25}$  nanoparticles and soaked in As containing water for 4 hours shows the best As removal capacity reducing the As content to 1.21 ppb from 41.5 ppb.

#### 7. Conclusions

In plasmonic dye sensitized solar cells the efficiency could be increased by 27% by incorporating silver nanoparticles into the TiO<sub>2</sub> structure. The improved performance was evidently due to enhanced photon absorption by the silver nanoparticle in TiO<sub>2</sub> electrode. Using a novel, three layer TiO<sub>2</sub>nanoparticle/nanofibre/nanoparticle composite photoanode, the efficiency of a dye sensitized solar cell could be increase by more than 30% compared to the solar cell made with standard commercial TiO<sub>2</sub> powder alone. In an electrochromic display device (ECD) of configuration, FTO glass/TiO<sub>2</sub>/Poly (methylmethacrylate) (PMMA) electrolyte/SnO<sub>2</sub>/FTO glass, an impressive reversible colour change between blue and colourless states could be observed. In a Mg rechargeable battery fabricated with TiO<sub>2</sub> as the cathode, an impressive discharge capacity of 220 mAh/g with an open circuit voltage of 1.85 V could be obtained. An electrospun nanofibre membrane prepared from a biodegradable polymer and functionalized with TiO<sub>2</sub> nano powder could effectively remove arsenic (As) from drinking water.

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