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Solar Energy Conversion and Storage Using a Photocatalytic Fuel Cell Combined with a Supercapacitor

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Abstract: This work studies the production of electricity by a photocatalytic fuel cell and its storage in a supercapacitor. We propose a simple construction, where a third electrode bearing activated carbon is added to the device to form a supercapacitor electrode in combination with the supporting electrolyte of the cell. The photocatalytic fuel cell is based on a CdS-sensitized mesoporous TiO_2 photoanode and an air cathode bearing only nanoparticulate carbon as an oxygen reduction electrocatalyst.

Keywords: photocatalytic fuel cell (PFC); TiO_2; CdS; supercapacitor; activated carbon

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1. Introduction

Photocatalytic fuel cells (photo fuel cells, PFC) are simple photoelectrochemical devices which can photocatalytically oxidize a (usually) organic fuel and produce electricity. In their most common configuration, they are made of a photoanode electrode carrying an n-type semiconductor photocatalyst, a dark cathode electrode, and an electrolyte [1–6]. Absorption of photons by the photocatalyst generates electrons and holes. Holes are consumed by oxidation of the fuel, while electrons are led through an external circuit to the cathode electrode where they are consumed by reduction reactions, typically, atmospheric oxygen reduction. Variations of the PFC may involve a photocathode carrying a p-type semiconductor photocatalyst [4,5,7,8]; however, the standard choice is a dark air-breathing cathode, similar to hydrogen-air fuel cells. PFCs provide an alternative route for conversion of solar energy and at the same time, offer the possibility of using wastes or pollutants as fuel. Therefore, they are gifted with an additional environmental benefit. The present work studies the possibility of combining a PFC with a supercapacitor, thus, offering the extra benefit of energy storage. A supercapacitor is a high-capacity capacitor [9]. In its simplest version, it is made by combining a carbon electrode with an electrolyte and achieves separation of charges in a Helmholtz double layer at the interface between the surface of the electrode and the electrolyte. The proposed design is to simply add a third electrode to the PFC which may act as supercapacitor in combination with the existing supporting electrolyte in the PFC. As it is schematically illustrated in Figure 1, this is a simple device without any particular complications, and it is easy to make. The third electrode, i.e., the supercapacitor electrode, is simply immersed in the electrolyte of the cell and it is connected with a separate cable to the photoanode electrode. The experimental results show that the design is satisfactorily successful. Since it is a combination of a photocatalytic fuel cell with a supercapacitor (SC), it will be henceforth abbreviated as a PFC-SC device.

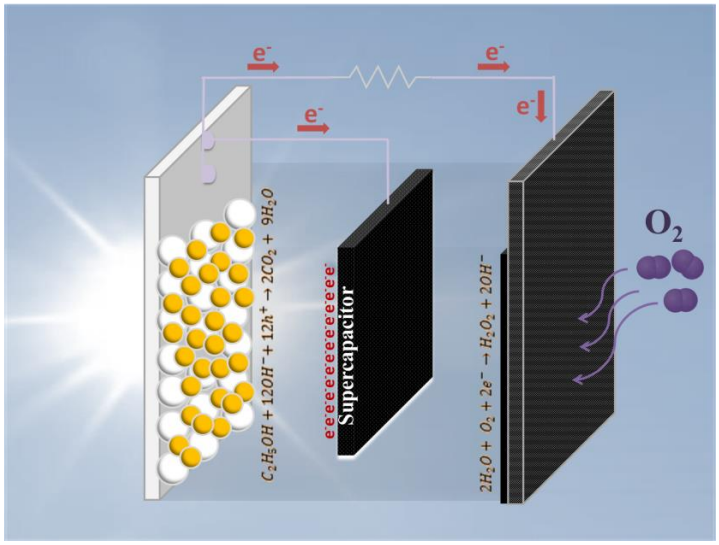

Figure 1. Schematic illustration of the combined photocatalytic fuel cell with a supercapacitor (PFC-SC) device.

The following choices were made in order to construct the electrodes of the proposed device: The photoanode electrode was a fluorine-doped tin oxide (FTO) transparent electrode on which a nanoparticulate photocatalyst was deposited. More specifically, a CdS-sensitized TiO_2 was used as the photoanode (CdS/TiO_2/FTO). It should be noted that a plethora of n-type semiconductor photocatalysts have been studied and published, but only a few among them have survived as a worthy choice, based on their efficiency and their stability, i.e., TiO_2, ZnO, WO_3, $BiVO_4$, and Fe_2O_3 [10–13]. Among these five, practically, only titania offers a PFC which can function without added electric bias. For this reason, we chose titania to make the photoanode for a functional PFC and CdS as titania sensitizer in the visible part of the solar spectrum, since titania absorbs only ultraviolet radiation. For the cathode electrode, we choose a standard air-breathing gas-diffusion electrode, similar to those used for several other air-cathode applications. Finally, for the construction of the supercapacitor electrode, we used commercial activated carbon derived from wood charcoal, i.e., charcoal obtained from a natural renewable source, adding further value to the construction of an environmentally friendly device. The use of charcoal produced by carbonization of plants has become a common practice in the construction of supercapacitors [14–19]. Charcoal is usually activated by treatment with KOH. In the present work, where the focus was on the applicability of the PFC-supercapacitor single-device combination, we used a commercial sample with medium supercapacitance.

The PFC-SC design can support the recent efforts for off-grid solar energy conversion and storage. To date, the most popular systems are the solar flow batteries, which combine a solar cell with a redox flow battery integrated in one single device [20–22]. Solar flow batteries constitute an already advanced technology. The advantages of the PFC-SC design is its simple construction and that it offers the possibility of combining solar energy conversion with photocatalytic degradation of wastes and pollutants.

2. Materials and Methods

All reagents used in this work were obtained from Sigma-Aldrich, unless otherwise specified, and were used as received. The photoanode electrode was made by following a standard protocol which is commonly followed and previously published by [3]. For this reason, construction of the photoanode electrode and its characterization is presented in the Supplementary Materials. The cathode electrode and the supercapacitor electrode were made by the following procedure: A piece of carbon cloth (CC; Fuel Cell Earth, Wobum, MA, USA) was cut in the appropriate dimensions to provide an active area of 1 cm^2. The active area was covered on one side by a paste made with the following

components: In the case of the supercapacitor, commercial activated carbon (AC) from wood charcoal (Merck, Germany) was mixed with carbon black (CB) (Cabot Corporation, Vulcan XC72, Billerica, MA, USA), water and polytetrafluorethylene as a binder (Teflon 60% wt. dispersion in water) at a weight ratio of 0.8:0.2:8:0.1, using a laboratory mixer operating at more than 4000 rpm. In the case of the air (cathode) electrode we did not use AC; therefore, the components were CB/water/binder at a weight ratio of 1:8:0.1. The paste was applied on the carbon cloth, dried at 80 °C for a few minutes, and then annealed at 340 °C. The procedure was repeated in order to reach a total load of 3 mg cm^{-2}.

The reactor was made of Plexiglas. It had two square windows on opposite sides. Thewindow on the left (cf. Figure 1) was sealed with the transparent photoanode electrode, while the window on the right was sealed with the cathode electrode, i.e., the air-breathing electrode bearing exclusively nanoparticulate carbon (carbon black). The distance between the anode and cathode electrodes was 1 cm. The cell was filled with 0.5 M aqueous NaOH, to which 5% w/w ethanol had been added. The supercapacitor electrode was simply immersed in the electrolyte in the space between the anode and cathode electrode. The anode electrode was connected with the supercapacitor electrode with a short cable, while the connection between photoanode and cathode was effectuated through a potentiostat in order to monitor the flowing current. All electrodes had an active area of 1 cm^2. Illumination of the photoanode was obtained with a Xenon lamp providing a light intensity of approximately 100 mW cm^{-2} at the position of the photocatalyst. The roles of the supercapacitor and the air electrode are well distinguished, despite their similarities, for the following reasons: The air electrode carries only carbon black, while the supercapacitor electrode is mainly loaded with activated carbon; the air electrode is exposed to ambient air which diffuses through its gas-diffusion layer, while the supercapacitor electrode is immersed in the electrolyte; and the latter is short-circuited with the photoanode electrode while the cathode is connected through an external load.

Electric measurements were made with an Autolab potentiostat PGSTAT128N, while field emission scanning electron microscopy (FESEM) images and energy dispersive X-ray (EDX) data were obtained with a Zeiss SUPRA 35 VP microscope.

3. Results and Discussion

3.1. Characterization of the Electrodes

The characteristics of the photoanode electrode were similar to those observed and published in previous cases; therefore, some details are given in the Supplementary Materials. Briefly, the CdS/TiO_2/FTO electrode was made of a nanoparticulate titania film of about 10 μm thickness, deposited on a transparent FTO electrode. The nanoparticulate CdS sensitizer was synthesized within the titania mesostructure and did not form a separate layer. The combined CdS/TiO_2 photocatalyst absorbed light with a band gap of about 2.4 eV, justifying a photocurrent density of about 7 mA cm^{-2}, according to published charts. The hydrophobic gas-diffusion layer of carbon black deposited on the carbon cloth electrode was also similar to those observed and published in previous cases; therefore, some of its characteristics are also presented in the Supplementary Materials. Briefly, it is made of carbon nanoparticles which present a substantial active surface, they are electrically conductive, and they form a hydrophobic layer that seals any electrolyte leak through the porous carbon cloth electrode at the same time providing channels for air diffusion through it. The nanostructure of the carbon black is also shown in Figure 2A. The picture was very different in the case of the activated carbon powder used in the present work. The activated carbon powder consists of size-polydispersed particles, which are solid blocks and lack nanostructure, at least at the scale of these FESEM images, as revealed by Figure 2B,C. During SEM measurements, one can roughly judge whether a material is conductive or whether it accumulates charges, since charge accumulation affects image clarity. Activated carbon was obviously accumulating charges, while carbon black was very conductive, and the charges flew though the substrate. However, the supercapacitor electrode was made of a mixture of 80% AC and 20% CB by weight. Both these materials show up in the film

forming the supercapacitor electrode, as seen in the FESEM image of Figure 3. The two materials were well mixed, with carbon black revealing its nanostructure and activated carbon appearing as solid blocks. The CB was needed to mechanically stabilizing the film and provide electric conductivity channels between the AC particles and the substrate electrode, which are both necessary to make a stable and effective supercapacitor electrode. Thus, the CB facilitates the transfer of electrons to the AC particles, and AC with its lower conductivity retains electrons, thus, providing energy storage.

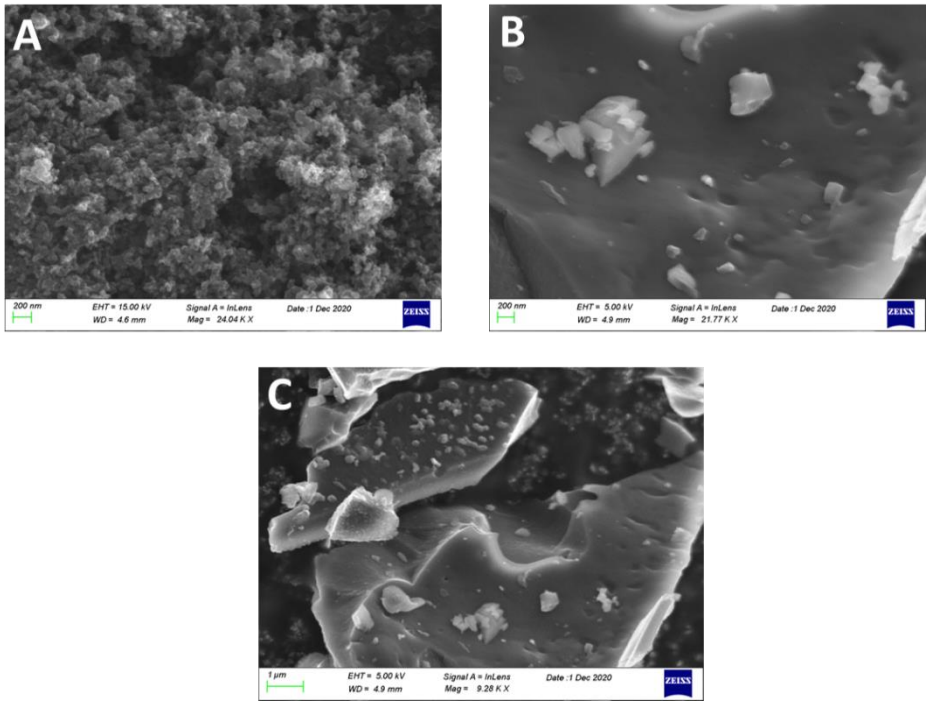

Figure 2. Field emission scanning electron microscopy (FESEM) images of carbon black (**A**) and activated carbon (**B**,**C**). The scale bar was 200 nm in (**A**) and (**B**) and 1 µm in (**C**).

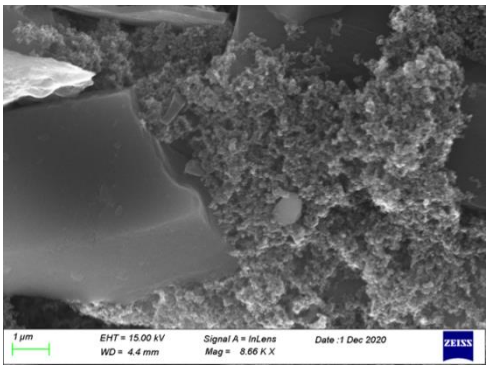

Figure 3. FESEM image of the film on the supercapacitor electrode showing the mixture of carbon black with activated carbon. The scale bar was 1 µm.

The materials used to make the above SC electrode have also been studied by EDX spectroscopy. The results are given in the Supplementary Materials. The spectra, in Figure S4 and Table S1, show some rather expected features. Activated carbon is composed of carbon and a substantial quantity of oxygen and potassium, and both are expected to come out during the activation procedure, which, as previously mentioned, usually occurs due to treatment with KOH. In the case of carbon black, as expected, no traces of K were detected, and the quantity of oxygen was lower, although detected with a large error.

The specific capacitance of the above SC electrode was measured by using a symmetric capacitor made of two identical SC electrodes immersed in the same electrolyte as the one used to run the PFC cell, i.e., an aqueous solution of 0.5 M NaOH containing 5% w/w ethanol. The corresponding charge–discharge curves are shown in Figure 4. The capacitance was calculated by the following formula: $C = 2 \times I \times t/V$, where I is the discharge current, V the maximum voltage, and t the discharge time. The factor 2 is due to the fact that a symmetric cell corresponds to two capacitors connected in series. Then, the specific capacitance is obtained by dividing C by the mass of the active material, i.e., 3 mg in our case. Thus, the specific capacitance of the presently used SC electrode was calculated to be 98 Fg^{-1}. The same measurement made with a symmetric capacitor composed of electrodes containing only carbon black was very small, i.e., of the order of mFg^{-1}, and thus it was considered to be negligible. The specific capacitance of the presently used supercapacitor electrode is substantially smaller than previously reported with other activated carbons [14,15]. In the most recently published work by [15], values of supercapacitance larger than 200 Fg^{-1} have been achieved. However, the purpose of the present work was not to compete with other supercapacitors, but to show that the construction of the simple device, shown in Figure 1, is possible and that the device is operational.

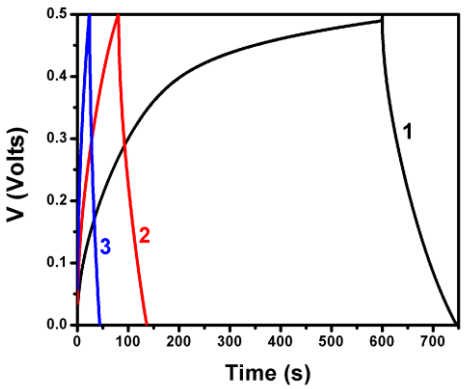

Figure 4. Charge–discharge curves for a symmetric cell made with identical electrodes carrying 3 mg of a mixture of activated carbon and carbon black, in the presence of an aqueous electrolyte containing 0.5 M NaOH and 5% w/w ethanol. The current was 0.5 mA (**1**); 1 mA (**2**); 2 mA (**3**).

3.2. Current-Voltage Characteristics of the Photocatalytic Fuel Cell

The abovementioned photocatalytic fuel cell alone, i.e., without the presence of a SC electrode, produced the current-voltage characteristics described in Figure 5. The curves were traced in a two-electrode configuration, i.e., without a reference electrode. The electrolyte was the one referenced above, that is, 0.5 M aqueous NaOH containing 5% w/w ethanol. Ethanol was added as a sacrificial agent that retains photogenerated holes, thus, increasing photocurrent and protecting CdS from self-oxidation. The device functions as a pure photo fuel cell without any external bias. Its open circuit voltage was approximately 0.85 Volts and the short circuit current density was approximately 3.8 mA cm^{-2}. The maximum photocurrent was reached quickly after the anodic photocurrent threshold, and then it plateaued. The increase in current with anodic bias greater than +1 V is simply due to electrolysis. Figure 5 confirms that the PFC device is a functional cell, and it can be employed to charge a supercapacitor. The next step was to show that energy storage in the supercapacitor did take place and that this affected the charge–discharge properties of the device.

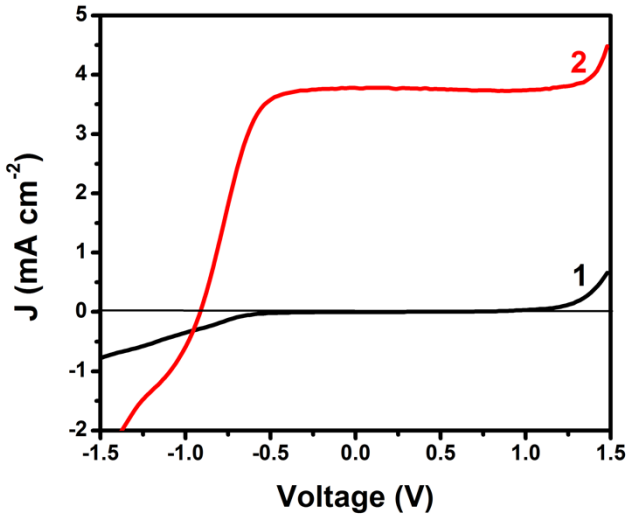

Figure 5. Current density–voltage curves for the presently studied PFC-SC device in the dark (**1**) and under illumination (**2**). The curves were run in a 2-electrode mode (no reference electrode). The electrolyte was an aqueous 0.5 M NaOH also containing 5% w/w ethanol.

3.3. Charge–Discharge Characteristics of the PFC-SC Device

Next, the abovementioned PFC-SC device was connected to a potentiostat and the short circuit current density was measured under potentiostatic conditions of 0 V (short-circuit conditions) and by turning a light on and off a few times. It should be noted that the SC electrode was short-circuited with the photoanode electrode. The results are shown in Figure 6. In the absence of the SC electrode, illumination of the photoanode resulted in an abrupt increase in current and, when the light was turned off, an abrupt drop in current to zero. Therefore, the obtained current was, in this case, pure photocurrent reaching a maximum under illumination and dropping quickly to zero in the dark. In the presence of the SC, the current never reached the maximum photocurrent value and in the dark never became zero. In this case, the increase and decrease in current were both slow, indicating charging and discharging of the supercapacitor electrode. In the present case, the capacitor was charged by accumulation of electrons within the activated carbon particles and the flow of electrons was facilitated by the conductive carbon black nanoparticles. The current did not reach maximum because part of the photocurrent was channeled into the SC electrode and was lost from the external circuitry that measures current. When the light was turned off, charging of the SC stopped and the SC was discharged through the external circuitry again, since the SC was short circuited with the photoanode electrode; thus, there was still a current flowing, even though the light was off. This pattern, as shown in Figure 6, continued unmodified for several hours.

Therefore, the above data show that it is indeed possible to make a functional PFC-SC device simply by adding a SC electrode and by operating with the same electrolyte as the electrolyte that supports the PFC operation. The question, then, arises as to the length of time that the discharge period might last when the light is turned off. This depends on the total capacity of the chosen SC electrode, i.e., the discharge period is longer with larger capacity. In our case, the discharge period lasted several minutes, as shown in Figure 7. The supercapacitor was charged for 2 min by illuminating the photoanode, and then the light was turned off. The current decreased, as expected, but it did not go to zero. The device behaved like a battery and produced a current density of about 0.5 mA cm^{-2} that lasted approximately 11 min. In future work, we expect to substantially increase this time period by modifying the quality of the activated carbon, the size of the SC electrode, the load of active material, and the nature of the supporting electrolyte.

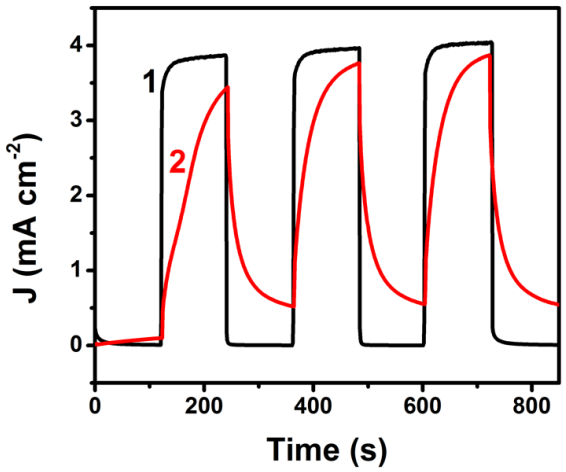

Figure 6. Variation of the short-circuit current density by turning light on and off under potentiostatic conditions of 0 V for a PFC device. (**1**) Without the supercapacitor (SC) electrode; (**2**) With the SC electrode.

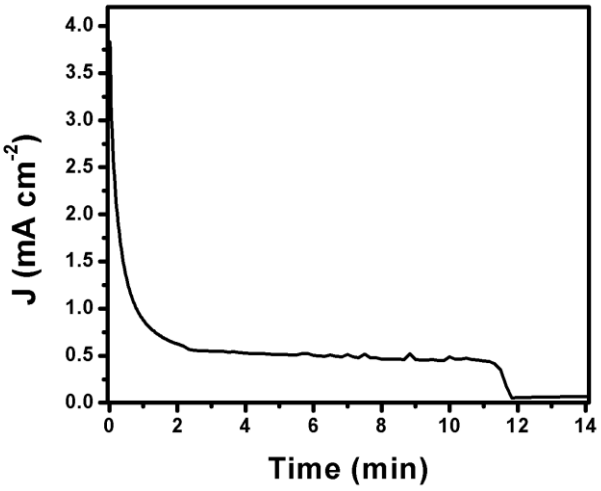

Figure 7. Evolution of the current produced by the charged supercapacitor in the dark.

4. Conclusions

This work shows that it is possible to add a third electrode carrying activated carbon in a photocatalytic fuel cell, and thus obtain charge storage in the form of a supercapacitor. An important characteristic of the proposed device is its simplicity, since a supercapacitor is obtained by combining the activated carbon-carrying electrode with the electrolyte of the cell. When the photoanode was illuminated by simulated solar light, a photocurrent ran through the cell without any external bias. Part of this photocurrent was consumed to store charges in the supercapacitor. When the light was turned off, the supercapacitor was discharged through the same circuitry as the one supporting the photocatalytic fuel cell itself. Thus, the current flow continued in the dark and for the present system lasted for about 12 min. Therefore, this simple device simultaneously provides the means to convert and store solar radiation.

Supplementary Materials: The following are available online at https://www.mdpi.com/2079-9292/10/3/273/s1, Figure S1. FESEM images of the TiO_2/FTO (A) and the CdS/TiO_2/FTO (B) film. Comparison of the two images reveals that CdS is formed within the mesostructure of titania and does not form a separate layer. The scale bar is 200 nm in both cases. Titania particle sizes ranged between 20 and 30 nm while CdS nanoparticles were smaller than 10 nm. Figure S2. FESEM image of carbon nanoparticles loading the carbon cloth electrode. Insert: The structure of the carbon cloth.

Figure S3. Diffuse reflectance absorption spectrum of the TiO_2/FTO (1) and the CdS/TiO_2/FTO film (2). Figure S4. EDX spectra of activated carbon (upper spectrum) and carbon black (lower spectrum). Table S1. Elemental composition of the materials used to make the supercapacitor electrode.

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Conflicts of Interest: The authors declare no conflict of interest.

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