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CONTRIBUTIONS TO THE IMPROVEMENT OF DYE-SENSITIZED SOLAR CELLS

PhD Thesis – Abstract

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In recent decades, research and development in photovoltaic technologies have sought innovative solutions to meet the growing demand for electrical energy in a sustainable and affordable manner, and the interest in third-generation solar cells, based on alternative materials to silicon and nanotechnology, has significantly increased. In this context, dye-sensitized solar cells (DSSCs) have attracted considerable interest and have emerged as a promising technology for the future of solar energy.

Dye-sensitized solar cells (DSSCs), developed by O'Regan and Grátzel, represent a revolutionary approach to the conversion of sunlight into electricity. Drawing inspiration from the photosynthesis process of plants, these cells use organic or inorganic dyes to absorb sunlight and generate a flow of electrons. Over the years, researchers have managed to optimize the structure and composition of these cells, achieving significant efficiencies and opening the way to practical applications.

One of the major advantages of dye-sensitized solar cells is their potential for cost reduction. Compared to traditional technologies such as crystalline silicon solar cells, DSSCs are manufactured using cheaper components and relatively simple manufacturing techniques, opening new opportunities for the widespread use of solar energy in both developed countries and in developing ones. In addition to the cost aspect, dye-sensitized solar cells have other notable advantages, being able to efficiently convert light energy into electricity, even in low-light conditions or on cloudy days. They can also be fabricated as flexible thin films, enabling their integration into a variety of applications and surfaces, from buildings and wearable electronics to charging mobile devices.

However, there are also technological challenges that the scientific community is working to overcome, with the performance and long-term stability of dye-sensitized solar cells representing critical aspects that require continuous improvement. Currently, methods are being sought to improve the efficiency of DSSCs through continuous research and development of materials and structures, as well as through optimization of manufacturing processes. A greater understanding of the factors affecting the long-term stability of these cells is also needed, with the aim of developing strategies to improve their durability and resistance to environmental factors and gradual degradation.

Thus, DSSC cells represent a promising photovoltaic technology, having the potential to revolutionize electricity production using renewable resources. With technological advances in the field and ongoing research efforts, these cells can play a key role in our energy future, helping to reduce carbon emissions and provide a clean and sustainable energy source.

Starting from these considerations, ***the aim of this thesis is to contribute to the improvement of dye-sensitized solar cells***, by making materials and optimizing the

photoelectrode in order to increase the efficiency of both p-type and n-type cells, but also by studying their behavior and stability in different lighting conditions and temperature range.

The objective of the research was to ***obtain and develop p-type materials (Cu_2O) and n-type (TiO_2) materials*** through energy-efficient and cost-effective processes, following the synthesis time, temperature and minimum amounts of precursors required to obtain materials with the properties desired, ***and their integration into dye-sensitized solar cells (DSSC)***. Also, in order to improve the solar cells made, it was aimed to change the energy bands through hydrogenation processes, to obtain p-type semiconductors with a low valence band, to obtain n-type materials with the role of scattering light radiation for a better collection its efficient use by the solar cell, optimizing the configuration of the photoelectrodes and introducing water into the electrolyte. The detailed physico-chemical analysis of both the materials synthesized for this purpose and the constructed DSSC-type cells, as well as the identification of the inner energetic mechanisms, were also the objectives of this thesis.

The thesis is structured in two parts, a first part in which is addressed, in two chapters, the role of DSSC-type solar cells in the current energy context, their operation and characterization, and a second part organized so as to present both a physico-chemical characterization of the nano and micro structured materials obtained as well as their integration in DSSC type devices by following the main efficiency parameters. Dye-sensitized solar cells were made by exploring different configurations and combinations of materials and tailored according to the efficiency requirements pursued.

The literature study that refers to the identification of the current state of research in the proposed topic is presented in **PART I of the doctoral thesis**. So:

In **Chapter 1**, a literature study on renewable energy is presented, with an emphasis on photovoltaic energy and its role in the current energy context, and the current state of research on DSSC-type solar cells is also presented. At the same time, the components of dye-sensitized solar cells, their operating principle, their limitations and application possibilities are presented. And **chapter 2** consists of a literature study in which the methods of synthesis and physico-chemical characterization of materials and electrical, respectively electrochemical of the DSSC type devices obtained in the research carried out for the elaboration of the doctoral thesis are described.

In **PART II** of the doctoral thesis, the original contributions are presented.

Chapter 3 presents our own achievements in terms of obtaining copper (I) and (II) oxides both in powder form and in the form deposited on a metallic copper substrate, and their integration into dye-sensitized solar cells of the type p with the aim of improving their efficiency. In this sense, the adsorption capacity of the dye on the surface of the photocathode was increased by obtaining porous structures of the materials and the modification of the energy bands of the materials was followed by treating them in an atmosphere containing hydrogen, as well as by obtaining CuO-Cu_2O compounds with low valence bands.

Thus, ***a simple hydrothermal synthesis*** method was proposed at 180°C for 24 h and with a low cost using polyvinylpyrrolidone (PVP) as a surfactant and copper acetate together with a copper plate as a source of copper in order to ***obtain, following a single synthesis, both the material for the photocathode and the counter electrode based on Cu_2O deposited on a flexible Cu metal substrate***.

Following this, a ***p-type dye-sensitized solar cell based exclusively on copper (I) oxide***, both in the photocathode and counter electrode composition, was made and tested, using P1 dye specific for p-type DSSC cells and electrolyte based on the I^-/I_3^- redox couple.

According to the results obtained from the scanning electron microscopy, it was found that the powder obtained from the hydrothermal synthesis has a porous structure, with two types of morphologies present, porous octahedra and a "stabilopode" type morphology, and the copper plate covered with a layer of Cu_2O shows a layered morphology, being formed by

overlapping layers of oxide.

It was carried out a study to investigate the influence of polyvinylpyrrolidone (PVP) on the process of forming porous structures, using the same experimental conditions. The results demonstrated that in the absence of PVP, no porous structures were formed, thus indicating its importance in pore generation.

At the same time, we carried out a study to better understand the processes that lead to the formation of the porous structures found in the composition of the studied compounds, varying the amount of copper acetate and the synthesis time. Thus, by using a 4 times smaller amount of copper acetate in the synthesis and by reducing the reaction time to 3 h, Cu2O in the form of microspheres was observed both in the obtained powder and on the plate surface. By increasing the reaction time to 6 h, the crystallization of Cu_2O in octahedral form was observed, the microspheres being still present on their surface, and by further increasing the reaction time to 24 h, the ***gradual dissolution of the microspheres*** took place, thus leading to the formation of the porous structure in the framework of the octahedra through a process ***of recrystallization of the microspheres***, a process also called ***Ostwald ripening***.

The photovoltaic parameters of the cell made exclusively with a photoelectrode based on copper oxide were compared with those of a cell made using a platinum counter electrode. According to the obtained results, the p-type dye-sensitized solar cell based entirely on Cu_2O electrodes, ***provided double values of J_{SC} and V_{OC}*** compared to the DSSC type cell using platinum counter electrode. The ***conversion efficiency of solar energy into electrical energy was improved by 60%*** using Cu_2O counter electrode on metal substrate compared to platinum based one.

Was carried out a study on the resistances present inside the DSSC type cells, by means of electrochemical impedance spectroscopy, and it was observed that the value of the ohmic resistance at the $Cu_2O/(Cu/Cu_2O)$ interface is much lower than that at the Cu2O/Pt interface, fact that is due to the high electrical conductivity of the Cu_2O particles grown directly on the surface of the metallic Cu substrate. It was demonstrated that the high values obtained for J_{SC}, respectively V_{OC} are due to the minimization of recombinations at the semiconductor oxide/dye/electrolyte interface, and the decrease in the filling factor when using Cu/Cu_2O as a counter electrode is due to the high value of the internal resistance compared to platinum.

Next, we pursued the improvement of the photocathode material with the aim of increasing the efficiency of the p-type DSSC cell and proposed a method of thermal treatment at 200°C, in an atmosphere with 2% hydrogen and 98% argon, of the Cu_2O powder used in making the photocathode. We have thus ***demonstrated the beneficial effect of hydrogen*** on the mechanism of p-type DSSC cells by conducting an experimental study of the interface between the dye and the hydrogenated semiconductor, a critical parameter in DSSC operation, and the presence of hydrogen in the copper oxide structure was proven by performing UV analyzes - Vis-NIR, FT-IR, PL and Mott-Schottky.

The hydrogenation treatment carried out at a temperature of 200°C does not lead to the formation of impurities or secondary crystallization phases, such as copper (II) oxide - CuO or metallic copper (Cu), demonstrating the successful achievement of the pure Cu_2O phase, and the morphology of the compound is not influenced by it.

It was demonstrated that the additional hydrogen in the system promotes a strong chemisorption of the COOH anchoring groups of the dye on the Cu_2O substrate leading to a higher adsorption of the dye, which implicitly led to a ***98% increase in the short-circuit current***, ***thus improving energy efficiency of p-type DSSC***. This increase in short-circuit current was also supported by the results obtained by analyzing the compounds by electrochemical impedance spectroscopy, the lower recombination resistance in the case of the hydrogenated sample compared to the non-hydrogenated one, suggesting that the recombination from the semiconductor oxide/dye/ electrolyte are reduced by the insertion of

hydrogen into the system.

We then ***followed the improvement of the electrode deposited on the metal substrate*** using besides polyvinylpyrrolidone and ethylcellulose (EC) with the role of forming porous structures, this being used as a photoelectrode and tested using a platinum counter electrode.

By analyzing the network stresses, determined using the Williamson-Hall equation, it was highlighted that the system is subject to a stretching stress, and the presence of surfactants influences the crystallization of Cu_2O particles, especially when using ethyl cellulose.

A comparative study of the cells both in the presence and in the absence of the dye on the surface of the copper plates covered with copper (I) oxide was carried out, the beneficial effect of the surfactants on the photovoltaic parameters, especially that of the ethylcellulose, being demonstrated both in the case of the cells sensitized with dye, as well as non-sensitized ones. The best obtained cell, using EC, generated a short-circuit current of 11.7 $mA \cdot cm^{-2}$, a voltage of 0.502 V, and a power conversion efficiency of 1.32%, thus confirming the beneficial properties of porous structure on efficiency. The increase in J_{SC} with dye loading of the cells demonstrates the presence of two photoactive materials in the system, both the dye deposited on the surface and the copper oxide grown on the plate surface. Ethyl cellulose is responsible for the increased value of J_{SC} due to its effect on improving the quality of the deposited film and increasing the porosity, which leads to a better fixing of the dye and implicitly to a higher capacity to absorb light radiation, the exact amount of dye loaded on the surface of each photocathode being determined by the adsorption-desorption technique. The high voltage value (V_{OC}) given by the Cu/Cu_2O_EC photoelectrode cell is attributed to the difference between the redox potential of the electrolyte and the quasi-Fermi level formed at the contact between the Cu_2O semiconductor and the metallic copper substrate.

We further ***proposed a series of mixed materials based on copper oxides (I and II) with low valence band***, obtained by using a simple and rapid synthesis method of low-temperature chemical precipitation, using sodium hydroxide and Pluronic P123 as a surfactant in order to change the valence band level of materials based on copper oxides (I and II) towards more positive values. The materials were proposed ***with the aim of using them in tandem DSSC solar cells, in order to improve the efficiency by increasing the photovoltage charged by the cells***.

Following the X-ray diffraction analysis, it was found that both the increase in the amount of NaOH in the system and the addition of the P123 surfactant lead to an increase in the CuO phase in the studied mixture. The increase in the basicity of the synthesis solution determined the oxidation of Cu^{1+} to Cu^{2+}, thus the CuO crystalline phase becoming the majority in the presence of P123.

It was highlighted that the variation of the NaOH concentration leads to an increase in the value of the valence band, a fact due to the positions of the edges of the valence bands of copper oxide-based materials that normally depend on the surface charge, which is also dependent on the defects of surface. Furthermore, P123 has a beneficial effect on the charge carrier density, thus providing a solution to optimize both parameters, both the absolute position of the valence band and the acceptor density. The beneficial effect of the amount of NaOH on the valence band was demonstrated, obtaining the highest value compared to other p-type materials analyzed in the literature, of 1.605 V.

The ***best photovoltage was estimated for a hypothetical tandem-DSSC*** cell, having TiO2-based photoanode and Cu_2O/CuO mixed compound obtained with 1.5 M NaOH as photocathode, suggesting improved V_{OC} performance of ***2.10 V***. The material thus obtained presents optimal characteristics for its implementation as a material for the photocathode in the composition of tandem-type DSSCs, but its implementation and testing is conditional on designing and synthesizing new organic dyes as sensitizers for p-type DSSCs, characterized by HOMO energy levels greater than 1.605 V vs. NHE.

The results obtained by UV-Vis-NIR spectroscopy showed that all materials obtained in these studies have bandgaps in accordance with those presented in literature studies, namely values between 1.7 eV and 2.2 eV for Cu_2O, and in the case of small compounds CuO/Cu_2O a decrease of the band gap was observed with the increase of the proportion of CuO in the system.

The results obtained by Mott-Schottky analysis revealed the conduction type of all the semiconductor materials characterized in this chapter, the negative slope of the line demonstrating the p-type conduction of the materials. Also, following the determination of the value of the energy levels, the energy diagrams of the DSSC cells built in the studies presented in this chapter were proposed and schematically represented.

Chapter 4 contains own results regarding obtaining materials with n-type conduction, namely TiO_2, with nano- and micrometric structure, each having a different role in the photoanode configuration, either as an absorption layer or as a light radiation scattering layer. The constructed DSSC-type cells were tested using three dyes that absorb either in the visible or in the ultraviolet range and electrolytes with different ratios of the I^-/I_3^- redox couple, with and without water content.

We carried out a study on the ***effect of water in the electrolyte composition on the photovoltaic performance of n-type DSSC*** cells by using electrolytes based on the I^-/I_3^- redox couple that contain water in different concentrations, from 0% to 40%. For this purpose, we used titanium dioxide (TiO_2) as n-type conducting semiconductor material, a simple sol-gel synthesis method was proposed at low temperature using Pluronic P123 as a surfactant, and titanium tetrachloride ($TiCl_4$) and titanium (IV) isopropoxide (TTIP), as a mixture of titanium precursors. ***The addition of electrolyte water up to 20%*** has been shown to be effective in improving DSSC efficiency by ***increasing the short-circuit current*** and fill factor but increasing the amount of water in the electrolyte up to 40% leads to a drastic decrease in efficiency energy, by up to 25-30%, decrease due to favoring the detachment of the dye from the surface of the semiconductor.

We have carried out a study on the ***effect of iodide/triiodide ratio on the energy performance*** of solar cells sensitized with two synthetic visible-absorbing dyes (N719 and N3), by using two electrolytes with different ratios of the I^-/I_3^- couple (named E1 and E2). A more pronounced increase in efficiency was observed for the sensitized photoelectrode using N3 dye compared to N719, and the highest efficiency was obtained using E2 electrolyte and N3 dye, namely a value of 3.39%. Thus, the addition of water to the electrolytes led to an increase in efficiency of up to 130% in the case of N719 sensitization and 73% in N3 sensitization.

The results obtained by Mott-Schottky analysis highlighted the type of conduction of the semiconductor powder obtained, the positive slope of the line demonstrating the n-type conduction of the material. Also, following the determination of the value of the energy levels, the energy diagrams of the DSSC type cells built in the presented study were proposed and represented schematically.

Next, we ***proposed two methods of hydrothermal synthesis*** at temperatures of 160 °C and 140 °C, for 2 h and 24 h, respectively, ***to obtain two micromorphologies of titanium dioxide with the role of light scattering,*** presenting two forms of crystallization, rutile and mixture of ~46% rutile and ~54% anatase. With these, we designed and constructed four photoanodes with different architectures and sensitized using the UV-absorbing dye DN-F01 in order to optimize them for their testing with platinum counter electrodes and aqueous and non-aqueous electrolytes. Through scanning electron microscopy, we have demonstrated the maintenance of the morphology of the particles obtained on the surface of the photoanodes, even after the process of making the paste, its deposition and thermal treatment at 500 °C.

We studied using UV-Vis-NIR spectroscopy, photoanode reflectance spectra and cell transmittance spectra obtained using the proposed photoanodes, confirming that the films composed of the spherical TiO_2 aggregates have a higher light scattering capacity due to the

larger diameters of the particles that are comparable to wavelengths in the visible spectrum and a transparency of cells over the entire range of photosynthetically active radiation. It was also found that the successive deposition of the light-scattering layers implicitly led to a decrease in the transparency of the cells, the values still exceeding a percentage of 20%.

We studied the role of each layer in the composition of the photoanode on the energy performance of the cells, ***a visible increase in efficiency being observed with the addition of each semiconductor layer in the composition of the photoelectrodes***, thus proving the beneficial effect of light scattering particles on the performance of the cells, leading to an increase of the efficiency by more than 50%, and further $TiCl_4$ treatment of the photoanode resulted in an even better improvement of up to 136% compared to the plain TiO_2 nanoparticle layer.

It has been proven that, also in the case of using the dye with absorption in the ultraviolet range, the addition of water in the electrolyte in a percentage of 10% has a beneficial effect on improving the efficiency of the DSSC, acting on the increase of the open circuit voltage, by up to 51 of mV, and of the filling factor, being ***higher efficiencies than those reported so far in the specialized literature in the case of using the DN-F01 dye as a sensitizer***, thus validating the success in optimizing photoanodes through the complex architecture of photoanode number 4 , with an energy efficiency value of ***3.47%.***

Chapter 5 consists in studying the effect of light radiation intensity and temperature on DSSC-type solar cells sensitized using UV-absorbing dye, with the aim of implementing them in wavelength-selective greenhouses.

We conducted a study on the ***effect of light radiation intensity*** as well as the ***effect of temperature on the energy performance of n-type DSSC*** solar cells made using the TiO_2-based complex architecture photoanode optimized in previous studies, sensitized using the DN-F01 dye with absorption in ultraviolet and tested using electrolyte based on the I^-/I_3^-redox couple, with and without water content, ***with the aim of implementing them in a wavelength-selective greenhouse***.

We demonstrated that ***the illumination of the cells in outdoor conditions, from strong sun to shade conditions***, namely between 20 $mW \cdot cm^{-2}$ and 100 $mW \cdot cm^{-2}$, resulted in short-circuit current values of circuit between 0.8 $mA \cdot cm^{-2}$ and 6.7 $mA \cdot cm^{-2}$, with ***a noticeable increase upon illumination at 60 $mW \cdot cm^{-2}$***, both using the all-organic solvent electrolyte and using the with 10% water. After studying the open circuit voltage, we noticed that this parameter increases slightly with increasing light intensity and becomes almost constant after illumination at 60 $mW \cdot cm^{-2}$, and the maximum powers discharged by the cells were obtained under illumination at 100 $mW \cdot cm^{-2}$.

The best efficiency was obtained when using the electrolyte with 10% water, resulting in values of approximately ***3.5%, this maximum efficiency being obtained at 60 $mW \cdot cm^{-2}$***, and the stability of the tested cells with increasing light intensity is highlighted by almost constant increase in fill factor, even when adding 10% water to the electrolyte.

After studying the internal resistances of the cells tested at 60 $mW \cdot cm^{-2}$ and 100 $mW \cdot cm^{-2}$, it was observed that the decrease of the charge transfer resistances from the photoelectrode/dye/electrolyte interface leads to the increase of the short-circuit current, and in the case of using water in the system, this increase is also due to the improvement of the photocatalytic activity.

We carried out a study on the thermal stability of cells ***investigating the effect of temperature variation on the photovoltaic performances*** of cells with and without water content in the electrolyte, and to quantify its impact we analyzed 3 characteristic temperature coefficients of short-circuit current (α), voltage of open circuit (β) and the maximum power delivered by the cell (γ). The beneficial effect of water on photovoltaic performances was found, this being highlighted by the increase of the open circuit voltage value by approximately 30

mV. This increase is the result of the higher solubility of the I- ion in water compared to the I3- ion, which causes a positive shift in potential. Therefore, the presence of water in the DSSC system contributes to an improvement in the voltage generated by the cell in the open state. Also, the thermal coefficient α is visibly reduced by the presence of water in the system, ***the improvement of the thermal stability of the adsorbed dye is directly reflected in the high α (+0.38%/°C), and an excellent maximum power factor (– 0.22%/°C), superior or comparable to other commercial generations of solar cells was obtained***.

We studied by means of Fourier transform infrared spectroscopy (FTIR) the dye-sensitized photoanode before and after testing the DSSCs in the temperature range from 25°C to 60°C under a solar illumination of 100 $mW \cdot cm^{-2}$ to provide information on ***the effect of anatase-rutile polymorphism on thermal stability*** and found that the presence of rutile in the photoanode structure causes the undesired bond breaks between TiO_2 and UV-absorbing dye molecules to be significantly reduced upon testing.

The research carried out in the framework of the thesis sought to obtain semiconductor materials, namely copper and titanium oxides and their application in DSSC type cells, with the aim of improving their properties. The conclusions of the PhD thesis demonstrated that this field is current and of interest, as dye-sensitized solar cells (DSSCs) can be successfully applied in various applications both outdoors and indoors.

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