

Copper Oxide Inked with Dye Obtained from the Lactarius Indigo Fungus for Energy Production

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Abstract

The results obtained from the characterization of a copper deposit on indium doped tin oxide (ITO), inked with natural dye extracted from the Lactarius indigo fungus, for use in Gratzel type solar cells are reported. An electrolyte composed of 0.1 M HNO₃ and 0.5 M CuSO₄ was used, this solution was prepared for copper deposits on the ITO. Cyclic voltammetry was performed at different scan rates to obtain the reduction zone for deposition between potentials of -100 to -500 mV. The dye was obtained from the indigo Lactarius fungus from maceration, once the inked deposits were obtained, characterizations were performed, the initial test was to obtain the Ultraviolet-Visible (UV-visible) of the pure dye, and later the same test was performed on the inked oxide. Electrochemical Impedance Spectroscopy (EIS) was performed on the samples, as well as Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM), to characterize the material properties for its application.

Keywords

Copper Oxide, Ink Dye, Energy Production, Indigo Milk Mushroom

1. Introduction

In the last 50 years, the world's population has grown exponentially. In addition to population growth, there has been a change in the culture of consumption, incurring greater use of non-renewable natural resources to meet their needs. Renewable energies have been developed as an alternative for electricity production that, unlike fossil fuels, help mitigate the problems caused by the indiscriminate production and use of electricity from conventional sources, as they avoid the emission of greenhouse gases and environmental pollution. The solar cell, a converter of visible electromagnetic energies into electrostatic energies, offers one of the renewable energy sources in today's human society that can provide a sustainable option for the future. One of the most relevant applications of solar cells is large-scale energy production, which allows reducing pollutant emissions (mainly CO_2) [1].

Cucaita in 2017, presented different solar cells made of different materials (copper, indium, gallium, selenium/sulfur), which are mainly characterized by their high adsorption coefficient, but among all, it highlighted cadmium telluride (CdTe) solar cells, as it showed that they are highly efficient and low cost for potential use in industry, however, it has limitations such as dependence on the availability of radiation and the use of silicon as raw material for solar cells [2].

On the other hand, Pilar Gonzáles presented a paper in which she studied advanced aspects of multi-junction solar cells designed to work at ultra-high concentrations. To this end, it was necessary to develop a three-dimensional distributed circuit model with which she simulated the behavior of triple-junction solar cells under different operating conditions, as well as an advanced characterization of this type of cells to better understand their mode of operation and thus be able to contribute to improving their efficiency [3].

Dye-sensitized solar cells (DSSCs) are cost-effective, environmentally friendly and efficient competitors to conventionally used silicon solar cells. The fundamental components of a DSSC are the following: metal oxides, sensitizer dye and electrolyte. However, there are many challenges surrounding the development and applications of DSSCs, such as: long-term stability, degradation, efficiency, etc. The key factor that has been identified to improve the performance of DSSCs is the right combination of energy levels when selecting the dye and semiconducting oxide. An ideal semiconducting oxide should possess properties such as: high electronic conductivity, chemical and mechanical stability, high catalytic activity, corrosion resistance and preferably low cost. Numerous metals are used as semiconducting oxides, including copper, aluminum, platinum, and tungsten.

A group of Brazilian students conducted an interesting practical experiment on the preparation of dye-sensitized solar cells (DSSCs), which were extracted from blueberries, as well as from raw and cooked beets. These dyes were used to sensitize the TiO₂ films that composed the photoanode in the DSSCs. They obtained a maximum solar-to-electrical energy conversion of 0.26% \pm 0.02% for the solar cell prepared with these fruit extracts [4].

For Grätzel, dye-sensitized solar cells are a technically and economically credible alternative concept to current p-n junction photovoltaic devices. Unlike conventional systems, in which the semiconductor is responsible for both light absorption and charge carrier transport, here both functions are separated. The use of broadband absorption sensitizers together with oxide films of nanocrystalline morphology allows a large fraction of sunlight to be captured, achieving overall solar energy (standard 1.5 AM) to current conversion efficiencies (IPCE) of over 10% [5].

The Sharma brothers emphasized on establishing a relationship between photosensitizer structure, interfacial charge transfer reactions and device performance, which is essential for the development of new metal-organic and metal-free dyes. With the main objective to perform a comprehensive review of new materials for photoanodes, counter electrodes, electrolytes, and sensitizers to provide low cost, flexible, environmentally sustainable and easy to synthesize DSSCs [6].

Copper is the material of choice for various thermal and electrical applications due to its desirable properties, such as high thermal and electrical conductivity, durability, corrosion resistance, ease of fabrication and economic availability. Copper oxides are p-type semiconductors; cupric oxide (CuO) has a bandgap of 1.21 to 2.1 eV, while cuprous oxide (Cu₂O) has a bandgap of 2.2 to 2.9 eV and a cubic crystal structure. These values indicate a very attractive absorption potential in the visible range. The use of selective solar absorbers is vital for both thermal and photovoltaic applications, and recently there has been increasing interest in copper oxides [7].

A group of Mexicans developed a coating with an acrylic (using soot), as a binder to adhere the coating to the metallic surfaces, with the purpose of characterizing the thermal behavior of the metallic surfaces, obtaining a good thermal characterization [8].

In a study carried out in Colombia, solar cells with Mo/CZTS/ZnS/ZnO structure using as absorber layer copper and tin films grown by simultaneous evaporation of their precursors from a coaxial evaporation source that was built with an advanced design which allowed to significantly improve the homogeneity in the chemical composition throughout the volume. Conversion efficiencies of 5.6%, a short circuit current of 18.3 mA and an open circuit voltage of 0.52 V were obtained [9].

In 2017, a work was carried out where different methods to prepare perovskite photovoltaic devices were studied. Successfully establishing a stable and reproducible method for the fabrication of solution-processed perovskite solar cells using lead acetate and MAI as precursor materials. It was also possible to tune the bandgap of the mixed bromide-iodide perovskites to reach a value of 2.0 eV, taking advantage of the stabilizing properties of the Cs+ and FA cations. In addition to investigating double halide and double cation perovskites fabricated using the antisolvent method. This absorber is optimized so that it can be complementary to MAPI in a perovskite/perovskite double junction solar cell [10].

Research on the use of organic dyes as solar cell sensitizers is essential for the development of new energy saving projects. It constitutes an advance in the scientific and environmental field since the properties, characteristics and functions of the dyes favor the implementation of solar cells due to their feasibility of application on light and/or flexible substrates, increase the efficiency levels

reached in a relatively short period of time and provide possibilities in conditions of low radiation intensity [1].

Copper oxides, particularly cupritic (CuO) and cuprous (Cu₂O) oxides, have been investigated for photovoltaic and proton reduction photoelectrochemical applications, because copper has a high natural abundance in the earth's crust and low toxicity. On the other hand, cupric oxide (tenorite as a mineral name) is another attractive p-SC, which may be valuable for fabricating pDSSCs, because it has a higher dielectric constant ($\varepsilon = 18.1$) than NiO. To our knowledge, there are only two reports dealing with the preparation of p-DSSCs with CuO nanocrystalline films. First, Suzuki *et al.* investigated four different dyes with the iodide/triodide redox pair on CuO-based p-DSSCs, resulting in modest efficiencies (around 1%). Very recently, Guldi *et al.* demonstrated interesting yields (around 2%) with zinc phthalocyanine dyes. These favorable characteristics have prompted further investigation of the potential of CuO for developing pDSSCs.

In 2016, CuO nanowires were synthesized, for which Cu (CH₃COO)₂-H₂O (1 g) and NaOH (2 g) were dissolved in 50 mL H_2O_3 , respectively. The two solutions were mixed and a large amount of blue precipitate was formed immediately. After filtering and washing the precipitate, $Cu(OH)_2$ was obtained. The precursor was sintered again at 300°C for 1 h in air atmosphere and finally black CuO nanowires were obtained. The nanowires were convolved with three types of dyes and two types of electrolytes, the electrolytes used are iodinated/triodinated and cobalt-based, the dyes used were P1, DPP-ND1 and YF1. These nanowires were characterized by XRD, UV-Vis, TEM and XPS, according to the results of the characterizations it was found as a result that CuO is unstable in the presence of iodide/triodide electrolytes, which excluded its use as a redox mediator. Complexes with bipyridine ligands are certainly suitable for redox. Secondly, it was observed that the transport time and medium in CuO is shorter than in NiO, probably due to the higher conductivity of CuO because of its wire-like morphology. Third, the deep absorbance of CuO cannot contribute significantly to the photocurrent production, no doubt due to the short diffusion length of the electron/hole pair generated in CuO.

Instead, the intrinsic absorbance of CuO shadows the absorbance of the sensitizer or can quench the excited state of the dye by energy transfer that limits the photocurrent density of the cell. This effect is more pronounced as the dye absorbs below 500 nm, such as P1 and DPP-NDI. However, if the sensitizer has a high extinction coefficient and absorption bands above 600 nm, the contribution of the dye can be significant. This was demonstrated by the comparison of the IPCE of P1, DPP-NDI and YF1. Therefore, it can be concluded that CuO could represent a valuable cathode material for solar energy provided it is sensitized with a low energy dye such as those above 700 nm. In that case, it can certainly be successfully used for DSSC fabrication in tandem with a photoanode that exploits most of the sunlight photons and leaves the lower energy part [11].

One study showed that electrochemical devices are emerging as the most viable option for an eventual replacement for fossil fuels, but they suffer from problems such as durability, operability, etc., but heterogeneous arrays of nanostructures have attracted a great deal of attention and have produced favorable electrochemical performance. Therefore, this study focused on the interconnection between the various components within each individual unit to correlate microscopic electrochemical processes with macroscopic performance. The conclusion is that the heterogeneous array of nanostructures proves to be an excellent idea for electrochemical energy conversion and storage [12].

Copper oxide is a promising anode material for lithium-ion batteries. However, the rapid capacity fading associated with large CuO volume variation has limited its practical application. In 2019, the fabrication of one-dimensional CuO nanochains via a facile wet chemical route was realized. The chain structure consists of particles, with an average crystallite dimension of ca. 20 - 25 nm, which helps to promote charge transfer at the electrode/electrolyte interface and accommodate oxide volume variation. Due to these structural merits, the CuO nanochains showed excellent performance and stable electrochemistry [13].

In 2020, a study was conducted comparing solar cells made of copper oxides combined with TiO_2/Cu_2O and cascaded TiO_2/CuO heterojunction systems. The efficiency of the solar cells ranged from 0.0005% to 1.62%. The thickness of the cupric oxide and TiO_2 layers ranged from 0.06 to 16 µm and from 0.18 to 1.5 µm, respectively, depending on the fabrication method [14].

Function of the Dyes

The cell dye is responsible for absorbing the photons of solar radiation. To carry out this function, the dye must absorb within the entire visible region and a certain part of the near infrared, since most of the solar radiation is found precisely in these regions. Among the dyes evaluated are some inorganic ruthenium complexes, natural dyes such as chlorophyll, carotenoids and anthocyanins, and some organic molecules such as phthalocyanins, porphyrins and conjugated polymers. One of the advantages of using organic molecules is the chemical versatility that can be obtained, since they can be designed according to the researcher's taste. At this point we can speak of "molecular engineering" of organic dyes designed a priori to meet specific needs. Among the characteristics that can be controlled are the region of the spectrum in which they absorb, solubility, the presence of anchoring groups, etc [15] [16].

Some important characteristics of light sensitizers in CSSC should be light absorption in the visible region of the electromagnetic spectrum, high molar extinction coefficient, chemical stability, and affinity for the inorganic semiconductor. Many sensitizers have been explored for use in CSSC, such as metal complexes of ruthenium, osmium, copper and other metals, as well as highly conjugated molecules such as porphyrins and phthalocyanines. In addition, several organic dyes such as indoles, coumarins, thiophenes, carbazoles and triarylamines have also been studied. From these studies, porphyrins stood out for their high solar conversion efficiency. Another type of molecules that have attracted attention are structures formed by donor and acceptor groups, traditionally referred to as donor- π -acceptor. These systems have a rather large molar extinction coefficient value in the visible region, which allows the use of TiO₂ films with a very small thickness. This type of molecules attracts attention because their chemical structure allows directing the electron towards the conduction band of the oxide, which allows minimizing energy losses along the way. In addition to the numerous advantages, they provide in optoelectronic properties [16].

2. Experimental Methodology

2.1. Obtaining the Dye

The colorant was obtained from indigo Lactarius mushrooms from the state of Hidalgo, they were dehydrated in the sun, placed in a ventilated container, and covered with a mosquito net, once dehydrated they were stored in flasks. Subsequently, 30 g of dehydrated mushrooms were weighed and poured into the capsule, and 20 ml of methanol were measured to the capsule, then the contents were poured into the mortar and maceration was started for 20 minutes. At the end of the maceration the content was filtered on Whatman #40 paper, placed inside the funnel and introduced into the nozzle of the amber type of flask. Once the amount of dye, approximately 200 ml, was obtained, it was refrigerated at 5°C, until its use for the analysis by ultraviolet-visible spectroscopy (UV-VIS).

2.2. Copper Electrodeposit

Copper deposits on ITO were performed by cyclic voltammetry and chronoamperometry, the first technique was performed on Autolab potentiostat-galvanostat using Nova 2.1 software, the electrolyte used was 0.1 M H₂SO₄ and 0.005 M CuSO₄. The ITOs were cut with a size of 2.5×2.5 cm, washed with deionized water for 10 minutes in the sonicator and followed in ethanol twice for 10 minutes, first a family of voltammograms was performed at different scanning speeds, then the reduction area was obtained to work with the conoamperometry to perform the copper deposits, it was chosen to use the potential of -0.44mV. The obtained deposits were analyzed by UV-Vis, Infrared and XRD.

2.3. Characterizations

The obtained dye was characterized by UV-Vis using a Perkin Elmer/Lambda Model 35 UV-Vis Spectrometer.

The characterization by Electrochemical Impedance Spectroscopy was performed in the potentiostat galvanostat Biologic using the EC-Lab/BT-Lab Software program, for this technique a specific working area of 1 cm^2 was delimited to the samples, this was done with the help of adhesive. A 0.1 M K₂SO₄ solution was prepared to assemble the cell, then a graphite bar was used as counter electrode, Ag/AgCl as reference electrode and also the ITO with Cu electrodeposits and inked at different times as working electrode; once connected to the potentiostat, the PEIS technique was selected within the program, and then the OPC of each of the samples was evaluated. Finally, frequency parameters ranging from 0.1 MHz and 1 mHz were proposed. Figure 1 shows the UV spectrum of copper at different times, which was obtained using the technique described above

3. Results and Discussion.

3.1. Sensitization of the Copper Oxide Electrodeposit

In the case of the longer exposure time of the oxide with the undiluted dye, a higher absorbance is obtained with an approximate value of 3, this highest point is at 600 nm and belongs to the exposure time of the dye of 4 h, compared to the spectra obtained from the dye. The groups present in the carotenoid molecule bind with the copper oxide film and this generates an alteration that is attributed to the decrease and the fact that the absorbance is shifted to the right, this not only happens with copper compounds, it also happens with TiO_2 that as presented in his work Maciej Zalas [17], also when in contact with the compounds of the carotenoid molecule do not present the same absorption as the dye alone.

3.2. XDR of Copper Electrodeposits

First, to the ITO spectra correspond $2\theta = 21.43$, 30.4, 35.9 and 60.25 which appear in the crystallographic chart PDF-Card: 01-071-2195 and then the copper peaks which are $2\theta = 43.35$, 50.49 and 74.20 according to data taken from Wyckoff R. W. (1963) [18], these data indicate that ITO and copper have a cubic crystal structure with orientations (211), (222), (400), (622) and (111), (200) and (220) respectively. In the case of the spectrum of the inked deposit it is observed



Figure 1. UV-Vis spectra of copper films inked at different times.

that there are no changes in terms of the characteristic peaks of the mentioned materials, it is only observed that there is a small edge at the beginning of the x-axis, this is due to the presence of a coloring compound, it is observed that the copper deposit does not contain impurities since the peaks of copper and ITO are very well defined [19]. This is reflected in Figure 2.

3.3. FTIR of Copper Electrodeposits

It can be seen in **Figure 3(a)** that ITO and copper are present in peaks 1230.58461 and 709. 804 respectively, it indicates that the bonding of both is present, this can be compared with the work done by Afrizal A. and coobblers [20] who



Figure 2. Diffractogram of the copper deposit sample without dye and of the copper deposit sample with dye for 4 hrs.



Figure 3. (a) FTIR spectrum of copper deposit on ITO without dye; (b) FTIR spectrum of Cu electrodeposit on ITO with dye 4 h.

worked with ITO and the work of Wang L. and co-cobblers [19] show a spectrum showing the characteristic Cu peaks, in this case Cu-O bonds are also shown since the deposits were in contact with air therefore must have a bond present. **Figure 3(b)** shows a change in the peaks present from the appearance of the C=C, C=O and O-H molecules, since the peaks that appear on the left side of the spectrum show the presence and stretching of these molecules since the signals are not as intense. On the right side it is observed that the peaks of ITO and Cu are affected by the presence of the molecules already mentioned since it is known that the solvent of the dye is ethanol, likewise also the carotenoid molecules contain these same groups therefore the Cu-O bond is affected as well as the one that exists in the ITO-Cu dye.

3.4. Electrochemical Impedance Spectroscopy of Copper Electrodeposit

The partial results of this technique are shown in the Figure 4(a) and Figure 4(b), where the copper electrodeposits with the lowest resistance to charge transfer would be the 2 h in dye since the smaller the diameter of the semicircle the lower this resistance, what follows is to repeat the test once again in other samples under the same conditions to those already performed for comparison, once this is done we will proceed to perform the analysis by an equivalent circuit that will help to understand each part of the cell as a component of an electrical circuit and quantitatively obtain the resistance to charge transfer.

From the graphs obtained from this characterization it can be observed that by itself the ITO is a very good conductor, that is, its resistance to charge transfer is minimal, as for the sample with copper it is observed that the resistance increases as the diameter of the semicircle increases, for the two samples that already have dye the resistance decreases, which agrees with the data in **Table 1**, according to the chosen circuit, with the data collected we can verify that the dye does help the charge transfer.

The circuit used was obtained according to the work of Sacco A. [21] in which the use of resistors and capacitors to obtain these data is explained in detail, since the basic electron transfer processes occurring in DSSCs are divided into 6, the photoexcitation of the dye (process 1) results in the injection of an electron into the conduction band of the oxide (process 2), leaving the dye in its oxidation state. The injected electron travels through the semiconductor reaching the conductive substrate (process 3), thus are flowing into the external circuit. The dye returns to its initial state by electron transfer from the electrolyte (process 4). The ions formed by oxidation flow through the electrolyte to the cathode, where the regenerative cycle is completed by the transfer of electrons to reduce (process 5). Therefore, this process is compared with a circuit that serves us to obtain the desired results, as Sacco mentions in his work there is a method called TLM which is a linear transmission model that takes into account different components the main ones are the resistances to charge transfer that exist between the different interfaces, in this case the one we are most interested in is the one between the dye and the oxide, therefore the circuit used has those components. Figure 5 shows the Nyquist plot for the copper electrodeposit with 2 h of dye. B) Nyquist plot for copper electrodeposit with 4 h of dye.



Figure 4. (a) Nyquist diagram of the ITO; (b) Nyquist diagram for the copper electrode-posit without dye.

Table 1. Resistance an	d circuit for	the samples
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Sample	Circuit	Resistance (Ohm)
White	C1/R1	0.2117
Cu	C1/R1 + C2/R2	1.327
2 hrs	C1/R1 + C2/R2	0.3031
4 hrs	C1/R1 + C2/R2	0.5580



Figure 5. (a) Nyquist plot for copper electrodeposit with dye for 2 h; (b) Nyquist plot for copper electrodeposit with 4 h of dye.

3.5. SEM of the Copper Electrodeposit Samples at ITO

Figure 6(a) and **Figure 6(b)** shows the morphology of the Cu deposit in ITO, which is mostly spherical except for certain areas where elongated morphologies are observed, this is due to the potential and deposition time at which they were performed, it is also observed that the deposit is not uniform, since there are certain areas that appear without particles but this is due to the fact that in the electrodeposition a uniform deposit cannot be controlled due to the solution used during the process. **Figure 7(a)** shows a more uniform and brighter deposit, this is due to the appearance of the dye that is observed as a viscous mass that



(b)

Figure 6. Micrographs of Cu electrodeposit on ITO seen (a) $2500\times$; (b) $5000\times$ magnification, with secondary electrons, at a working distance of 8.4 mm.

covers especially in certain areas, the fact that the sample shines more than in the sample without dye indicates that when bombarding this sample with electrons this sample with dye reacts reflecting due to the fact that the sample with dye is more conductive. It can be seen in **Figure 7(b)** the coating containing the dye, which gives the sample the ability to absorb electrons and retain them. In





Figure 7. Micrograph of Cu electrodeposit on ITO with dye at 2 hr, viewed at (a) $1000 \times$ magnification, with secondary electrons, at a working distance of 8.5 mm; (b) $5000 \times$ magnification, with secondary electrons, at a working distance of 7.4 mm.

general, a change can be observed regarding the presence of the dye that appears as a viscous mass, moreover, based on the works of Rahman A. S. [22] and Wang L. [19], who present electrodeposits controlled only by the potential, it can be observed that they had very similar morphologies.

4. Conclusion

The dye increases the light absorbance due to the groups present in the carotenoid molecules compared to the absence of dye. The electrodeposits are formed by copper and copper oxide in the shape of a cube with spherical and elongated morphology. The resistance to energy transfer decreases in the presence of the dye.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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