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STRUCTURAL AND MORPHOLOGICAL CHARACTERIZATION OF DYE-SENSITIZED SOLAR CELLS USING NATURAL DYES AS SENSITIZERS

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Abstract: *The search for alternative forms of energy turns out to be a crucial point for the development of a technologically advanced society committed to environmental sustainability. Amongst those, solar power is a particularly promising solution to the world's energy needs. Recent researches on finding an alternative energy resource for the next generation addresses the design of efficient photovoltaic cells. From the variety of such devices, we focus on the production of dye-sensitized solar cells (DSSC's). However, the construction of low cost, highly efficient and stable DSSC's defines a challenge for practical applications. At the Laboratory of Experimental and Applied Physics (LaFEA - CEFET/RJ) we have been able to produce DSSC's using organic dyes extracted from different fruits and vegetables to be used as sensitizers. Photo-anodes Titanium Dioxide (TiO_2) films with different thicknesses were prepared in order to get proper morphological surface profile. The cathode films were composed by a thin layer of graphite, applied over a Transparent Conductive Oxide (TCO) Indium Tin Oxide (ITO) coated glass. The resulting films were characterized by Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) measurements, and the composition and superficial characteristics of the films were also studied. The photovoltaic performance and electrochemical impedance studies were carried out to understand the effect of the variation of the thickness of photo-anode films for different dyes. In a comparison between solar cells with two different thicknesses, it was found that dye-sensitized solar cell prepared with the thick photo-anode film shows relatively more short-circuit current (Isc = 0.4 mA) and open-circuit voltage (Voc = 400 mV) than the solar cell designed using thin coating of photo-anode film (Isc = 0.3 mA, Voc = 225 mV). Also, the maximum short-circuit current of 4 mA was observed in a sample with 10 x 10 cm^2 of area, designed for large applications. As a matter of fact, the project we follow herewith meets the expectation of developing and characterizing low-cost solar cells, that may turn out to be economically viable. Future assembling in panels or small power modules, resulting in highly-stable and efficient DSSC's are challenges to be achieved as requirements for large scale power supply provided by this kind of solar cells.*
Keywords: *solar cells, organic dyes, thin films*

1. INTRODUCTION

The search for alternative forms of energy turns out to be a crucial point for the development of a technologically advanced society committed to environmental sustainability. Amongst those, solar power becomes an interesting choice for clean, renewable and non-polluting source, as photovoltaic cells are employed to convert solar energy into electric power. Over the last few decades, several kinds of solar cells have been the subject of intense research, and different materials were used for this purpose. Recently, photoelectrochemical dye-sensitized solar cells, also known as dye-sensitized solar cells (DSSC's) or Grätzel cells (O'Regan and Grätzel, 1991), have been proposed as a low-cost and efficient alternative to conventional silicon-based solar cell technology.

A Typical DSSC consists of the following main components: two glass substrates covered by a conductive and transparent layer (TCO Film - Transparent Conductive Oxide), a dye-sensitized semiconductor nanostructured photo-anode, an electrolyte with a redox couple and a conductive counter-electrode.

The photovoltaic conversion in this type of device is based on interference with recombination processes which tend to bring the electrons to a non-excitation condition in the conduction band, since these electrons in the conduction can recombine with impurities or holes contained in the semiconductor material and return to the valence band (Kirchartz

et al., 2015). The charges' collection in the device must occur before its recombination, in order to make a more efficient solar cell.

The semiconductor material (TiO_2) of the Grätzel cell works as a receiver of the electrons generated by light in the excited dye, since these electrons, after being generated in the dye, are injected into the conduction band of TiO_2, resulting in a current flow in the film.

The occupation of the gap that is in the oxidized dye after the electron being injected into the conduction band of TiO_2 is carried out by the reaction with the redox couple (I_3^- / I^-) that exists in the electrolyte because the iodide (I^-) oxidizes generating triiodide (I_3^-), leading to the occupation of the gap generated in the dye. After this chemical reaction, the electrolyte undergoes a reduction reaction on the counter electrode of the solar cell. Figure 1 illustrates the principle of a basic operation.

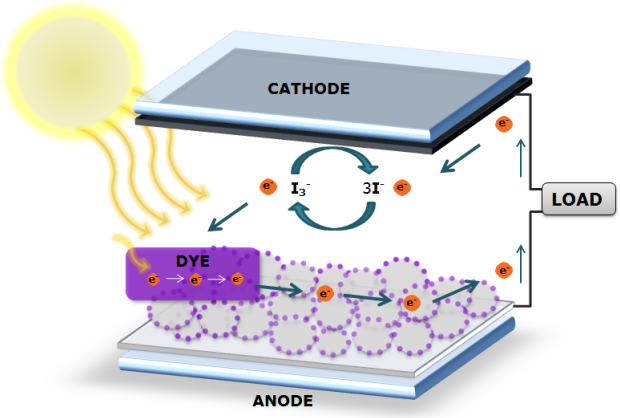


Figure 1: **DSSC's working principle.**

The dye is the main element of the DSSC's and performs the function of reception of the photons and sensitization of the anode, so that it must absorb the maximum amount of radiation throughout the solar spectrum, strongly bind to the semiconductor surface, have a suitable redox potential and be stable for several years of sun exposure. However, most semiconductor polymers have energy gap larger than 2.0 eV (620 nm), which limits absorption of solar photons at around 30% (Nunzi, 2002).

Amongst the natural dyes, shows better efficiency those which contains flavonoids and anthocyanins, which can be found in flowers, leaves and fruit. Such dyes have absorption in the visible light range, in the range of 520-560 nm. The adsorption of anthocyanins on the surface of TiO_2 is only possible through Van de Walls forces, and occurs by linking two oxygen atoms of anthocyanin with ion Ti^{4+} lying in surface of TiO_2 with two positive links unfilled (de Araujo, 2012). The main electrochemical processes that occur in the DSSC's are the separation and recombination of charges.

The electrolyte consists of iodine / triiodine and has the function of electrons' transport between the electrodes. In the DSSC's, iodine ions (I^-) fill the holes of the pigments and are converted into triiodine (I_3^-) in nanoporous surface. The reverse process occurs at the positive electrode, when it receives electrons that complete the cycle through the external circuit.

Polymer electrolytes are described as solid ionic conductors prepared by the dissolution of salts in a suitable high molar mass polymer containing polyester units (Vincent, 1987). The role of the polymer is to act as a stiffener for the solvent, creating a three-dimensional network, where cations and anions move freely in the liquid phase. In this work, we used a mixture of iodine and polyvinylpyrrolidone (PVP).

In this work, we propose a design for manufacturing photon-to-electron conversion DSSC's using the semiconductor oxides TiO_2 and natural dyes as sensitizers, extracted from avaiable fruits and vegetables.

2. EXPERIMENTAL PROCEDURE

Once studied the main features of the DSSC's, it was defined the assembly of the same, as well as the semiconductor films and dyes used. The components used for the synthesis of the dye-sensitized solar cells include: dyes ; the nanocrystalline semiconductor (TiO_2); the electrolyte (iodine solution + PVP); two glass electrodes with a conductive and transparent layer (film ITO) and a catalyst (graphite).

For the substrates were used glass plates of 2 x 2 cm and 10 x 10 cm coated with indium tin oxide (ITO film). Initially, we cleaned the glasses with isopropyl alcohol, and then to submit a new industrial cleaning with high purity detergent Triton X-100 and double distilled water. We used an ultrasound cleaning device for about 20 minutes and then the glasses were dried using hot air. Then, with the help of multimeter, we identified which side of the plates contained the surface where the conductive film ITO was deposited.

The TiO_2 films were prepared with 2 g of TiO_2 mixed with 10 ml of ethanol, 1 ml of HNO_3 (nitric acid) and 1 ml of Triton X-100. The deposition of the paste was performed by spreading and spontaneous adhesion by technique known as "doctor blade" (Dinh *et al.*, 2011). In this technique, TiO_2 emulsion is applied and carefully spread over the

semiconductor substrate using a glass rod. After the preparation, the material was subjected to an thermal treatment at (*annealing*) 450 °C for 30 minutes using a muffle oven. Samples with two different thicknesses were prepared, and also, a sample with 10 x 10 cm, with different dyes.

Graphite coated counter electrodes were prepared by the following procedures: the graphite paste was prepared by using commercially available graphite powder of 1 g with a few drops of Triton X-100 and HNO_3 solutions. The as-prepared paste was then coated on the conducting ITO substrate by Doctor Blade technique. The prepared samples were annealed at 450 °C for 30 minutes. The surface morphology of cathode films was analyzed by AFM instrument. The thickness on the graphite coating was maintained constant for all DSSCs.

In this work, we used two different dyes extracted from jambul and jabuticaba skins. The fresh fruits were cleaned and had their bark removed. The 100 ml of ethyl alcohol was added and mixed until they form a kind of gel. The solution was then filtered and stored in a closed container and refrigerated.

The anode was then dipped into the solution with the dye for 20 to 30 minutes, and once the absorption could be observed by color change of the semiconductor film, the formed anode glass with the semiconductor oxide and applied dye was dried using hot air. These sampleswere characterized using spectroscopy in Fourier transform infrared (FTIR).

The electrolyte was first prepared as follows: a solution of KI (Potassium Iodide) was mixed with 0.1 M Iodide, 0.1 M in a ratio of 1:1 to obtain 20 ml. For the manufacture of the polymer electrolyte, the same solution was mixed with 100 mg of PVP. These solutions were stored in the dark in order to avoid degradation of the electrolyte and used during assembly of various cells manufactured later.

For the assembly of photovoltaic cells, we join the two ready electrodes (anode and cathode). The two surfaces were joined in lagged manner for obtaining the positive and negative terminals of the solar cell. The electrolyte was then applied between the two faces and the cell was sealed with metal clips which were subsequently removed and replaced with transparent tape.

Figure 2 shows the different steps for the preparation and assembly of the DSSC's. In Fig. 2a we can see the TiO_2 films with two different thicknesses (samples A and B). Figure 2b depicts the graphite films cathodes, as Fig. 2c shows the anodes with the dyes adsorbed. Finally, Fig. 2d shows the final assembly of the DSSC's, with the photo-anode and cathode films sandwiched, by facing each other for solid-state DSSC's.

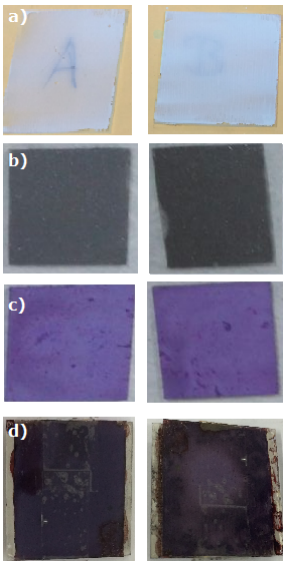


Figure 2: **Steps for preparatin and assembly of the DSSC's.**

3. RESULTS

In order to understand the thickness dependent microstructure of TiO_2 anode films, the samples A and B were subjected to microstructural analysis by SEM (Scanning Electron Microscopy) and AFM (Atomic Force Microscopy) techniques.

The Scanning Electron Microscope (SEM) is a type of electron microscope that produces images by an electron beam that focuses on the sample, scanning it (Vernon-Parry, 2000). These electrons interact with the atoms of the sample, so we can determine and display its composition and topography from the image obtained. SEM can produce images with a magnification of 10 to 500,000 times, including color images, which greatly contributes to a more complete analysis of the surface morphology of a given sample (Midgley, 2006).

Figure 3 shows the images generated in SEM with magnifications of 100x, 1000x and 2500x for samples A and B.

We can see similar structures in samples A and B, with sample B presenting the more uniform structure of the mesoporous TiO_2 through the distribution of fine grains of TiO_2 throughout the film, as we can see in the higher magnification image. These pores are important for the chemical mechanism of absorption of dye molecules by the semiconductor oxide

as the dye is adsorbed to these nanopores, which thus guarantee the sensitization of the photosensitive anode.

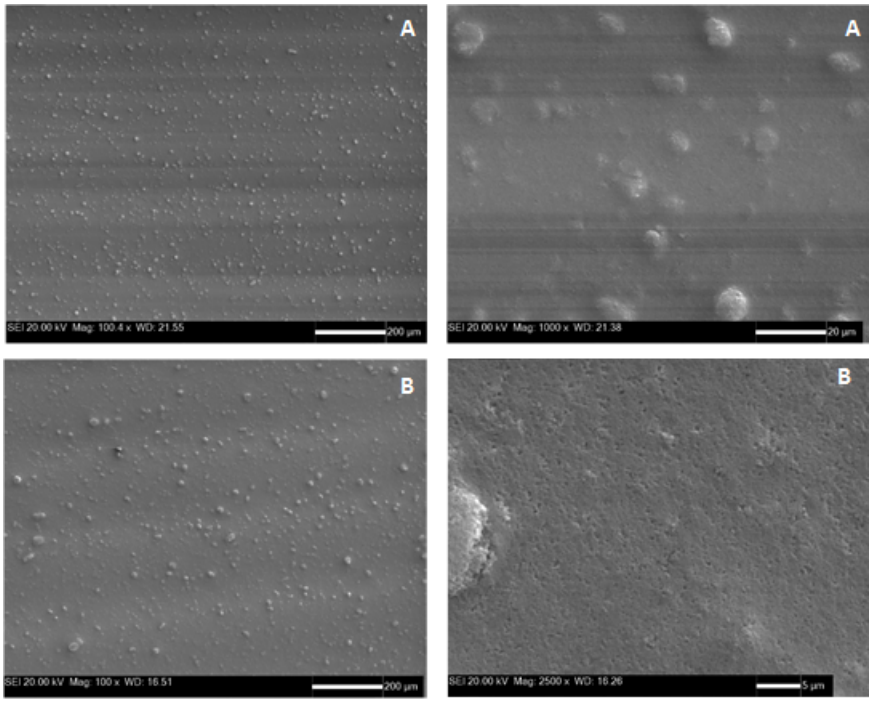


Figure 3: **SEM images for the samples A and B.**

Details of the structure and morphology of the films could be verified using Atomic Force Microscope (AFM). This analysis is essential for characterizing thin films, as it gives us an idea of the thickness, roughness and performance of semiconductor thin films.

In Fig. 4, we see the images generated in the AFM for different thin films produced, for the samples A and B. In Fig. 4a we see a surface image of a thiner film composed of TiO_2 nanostructured, with mean square roughness (RMS) of 36.7 nm, and the average size of TiO_2 nanoparticles is approximately 27.9 nm. Figure 4b Figure shows a thicker film composed of TiO_2 with RMS roughness of 103.0 nm and 75.0 nm nanoparticles. These values, as shown in Table 1, indicate a profile for the samples and validate the " doctor blade " fabrication of thin films.

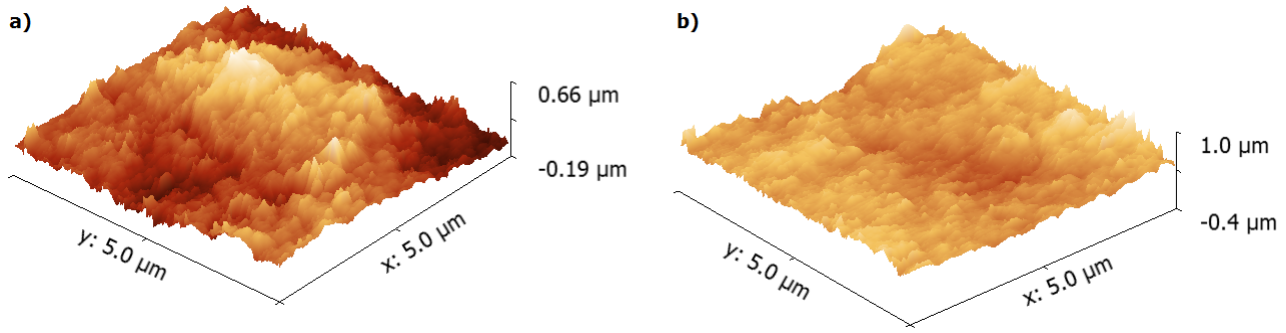


Figure 4: **AFM images for the samples A and B.**

Table 1: RMS roughness and particle size for the samples A and B.

AFM Results		
Sample	**RMS Roughness (nm)**	**Particle Size (nm)**
A	36.7	27.9
B	103.0	75.0

As can be seen in recent studies (Ye *et al.*, 2015a), many natural pigments such as anthocyanins, chlorophyll, carotene and tannins have been used as sensitizing dyes in theDSSC's. Amongst them, the anthocyanins are shown as natural sensitizers with better performance (Hemalatha *et al.*, 2012). To find out which dyes would be most appropriate, the analysis of the sensitized anodes was performed using Fourier Transform Infrared spectroscopy technique (FTIR). The FTIR was used to obtain the spectra of absorption, emission and photoconductivity of the DSSC's, revealing to us its composition. Infrared spectroscopy allows us to identify the presence of various functional groups in the structure of

an organic compound. This is possible due to the interaction of atoms and molecules with electromagnetic radiation at specific vibration frequencies, which vary with the structure and composition of the sample, defining the vibration modes. Infrared is then used to scan such frequency range. The FTIR spectra for jambul dye with the different thicknesses is shown in Fig. 5.

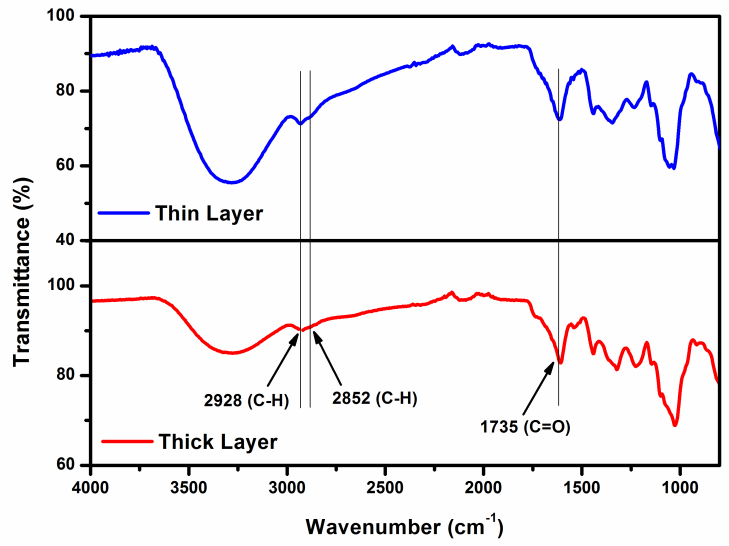


Figure 5: **FTIR spectra for the samples A and B.**

In the FTIR spectra obtained for both samples we can identify the same peaks, with some differences in amplitude, showing us the differences in the absorption of the dye in each case. Also, the FTIR spectrum in Fig. 5 exhibits most of the characteristic peaks of the anthocyanin. The two peaks at 2928 and 2852 cm^{-1} correspond to asymmetric and symmetric C-H stretching modes, respectively. The spectrum also contains a peak at 1735 cm^{-1} corresponding to characteristic C=O stretching vibration of anthocyanins (Shanmugam *et al.*, 2013).

The FTIR still serves us as a good tool for indirect measurement of the thickness of the films, since there is no availability of more accurate tools to carry out this measure, such as a profiler. Through the absorbance spectrum (Fig. 6), we can see the peaks of H_2O present in the wave number range between 2927 and 3282 cm^{-1}. the band area being calculated for each sample, column density (*N-Value*) and knowing the strength of integrated band (*A-Value*), we can then calculate the thickness of the films (Fulvio *et al.*, 2009). The value used for the density of the TiO_2 was 0.94 g / cm^3. Table 3 shows the obtained results.

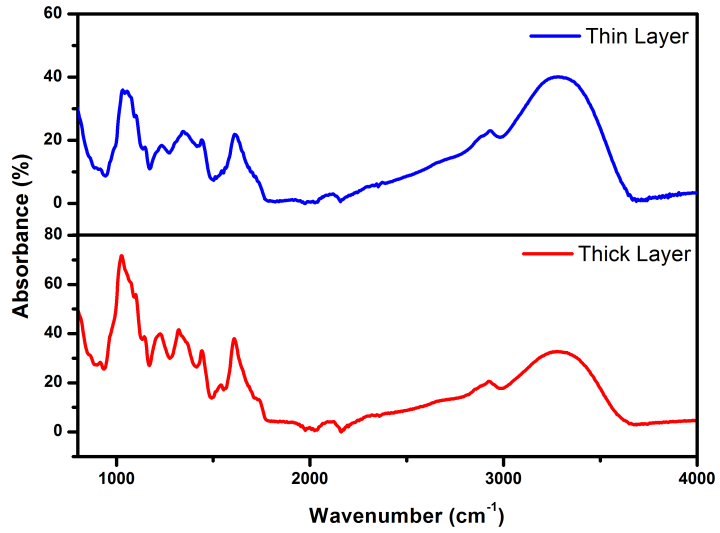


Figure 6: **FTIR absorbance spectra for the samples A and B.**

The area under the H_2O peak ($\mathbf{a}_\nu$) was determined for calculation of the initial column density (N_0) of all samples, according to the Equation 1. The value of the band strength for H_2O, A (cm/molecule), of the respective sample vibrational mode, was 2 x 10^{-16} cm/molecule (Pilling *et al.*, 2011).

$$N_0 = \frac{2.3}{A} \int \mathbf{a}_\nu \, d\nu \; [molecule\ cm^{-2}] \tag{1}$$

Considering an average density for the TiO_2 film with H_2O of about 4.3 g/cm^3, the sample thickness (μ) can be obtained by the Equation 2.

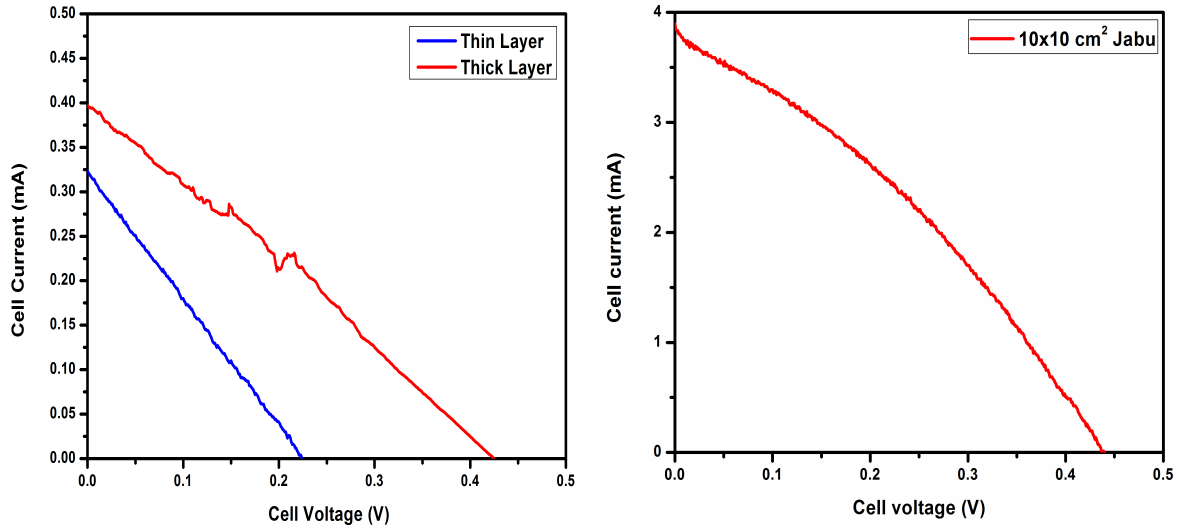
$$\mu = \frac{N_0}{6.02 \times 10^{23}} \frac{M}{\rho} \times 10^4 \; [\mu m] \tag{2}$$

where N_0, M, and ρ are the initial column density, in units of molecule/cm^2, the molar mass, in units of g/mol and density, in units of g/cm^3 for the considered species, respectively. The thicknesses of 3.47 and 11.51 μ m were found for the samples A and B and C, respectively, as can be observed in the Table 3.

Table 2: **Thickness calculations for the samples A and B.**

Sample	*A*-Value (cm/molecule)	*N*-Value (molecule/cm^2)	a_ν (cm^{-1})	Thickness ($_\mu$m)
A	2.00 x 10^{-16}	112.24 x 10^{17}	975.99	3.47
B	2.00 x 10^{-16}	372.53 x 10^{17}	3239.44	11.51

Figure 6 shows the photovoltaic characteristics of the DSSCs for two samples with jambul fruit dye molecule (samples A and B) and another one with jabuticaba fruit dye (sample C). The potential-current curve was measured under direct illumination of sunlight, with an irradiance of 800 W/m^2. It can be observed that the samples A and B show the typical I-V characteristics with 225 and 425 mV open-circuit voltages (V_{oc}), whereas sample C shows the linear decrease of cell current with maximum open-circuit voltage of 435 mV, as shown in Table 3.



(a) **IV curves for DSSC's with different thicknesses.** (b) **IV curve for a DSSC with jabuticaba dye.**

Figure 7: **Current vs. voltage curves for DSSC's with different dyes.**

Table 3: **Voltage vs. current values of the samples with different sensitizers.**

Dye	Isc (mA)	Voc (mV)
Jambul - A	0.33	225
Jambul - B	0.40	425
Jabuticaba - C	3.90	435

We can observe by the I-V curves that sample C shows higher values of V_{oc} and I_{sc}, despite of samples A and B. In general, the decrease in V_{oc} of DSSC's can be attributed to many reasons. In general, photoelectrodes for DSSC's are often resulting the low performance due to important recombination between the injected electrons and triiodides or direct contact of the triiodides to the anode substrate. In this case, decreased V_{oc} of sample A can be mainly contributed to an inequal distribution of the TiO_2 film over the substrate. And also, this can contribute to the reduction of the average optical power density within the film and more charge recombination sites are produced.

4. CONCLUSIONS

The main purpose of this work was to develop and study the functional characteristics of the DSSC's. For this, Doctor Blade technique has been used to prepare the photo-anode and cathode films. For the photo-anodes, we used two different dyes, extracted from jabuticaba and jambul skins. Also, we prepared samples with relatively different thicknesses, for the jambul samples. The thickness dependent surface microstructure of the photo-anode film was also studied, by the structural analysis of the produced thin films, with the SEM and AFM microscopy techniques. It was found that increasing the thickness of TiO_2 film affects the continuity of microstructure, which results in of increasing the

open-circuit voltage. The increasing of thickness creates paths in the meso-porous TiO_2 layer and those have an effect of increasing the photovoltaic performance of DSSCs. The photovoltaic performance results indicate that the films with semitransparency and continuous microstructure are the best candidates for dye-sensitized solar cells.

Also, comparing the photo-current of the sample C with the samples A and B are decreasing linearly with potential, which can be attributed to more charge recombination and electron scattering effects at the photo-anode. The extracts of jabuticaba skin act as good sensitizers and efficiently promote electron transfer across the dye/semiconductor interface. However, thickness of the photo-anode films and electrolytes playing the important role in efficient performances of DSSCs. In our case, it was found that the optical transparency as well as relatively thick photo-anodes are important parameters for increasing the efficiency of DSSCs.

FTIR technique was used to obtain an infrared spectrum of absorption, emission and photoconductivity of the DSSC's, showing us the compounds characteristics. As one can read from recent works (Ye *et al.*, 2015b), several natural pigments such as anthocyanin, chlorophyll, tannin, and carotene have been successfully used as sensitizers in DSSC's. The FTIR analysis showed us that some fruits containing high concentration of anthocyanidin molecules exhibit better efficiency as a dye, and should be targeted for the best thin film preparation technique. Amongst the tested dyes, jabuticaba juice presented the highest values of conversion, with improved values of V_{oc} (Open-circuit voltage) and long stability. In fact, for dye molecule to be excellent sensitizer, it must possess several carbonyl (C=O) or hydroxyl (-OH) groups capable of chelating to the semiconductor photoanode sites on the thin film surface (Luo *et al.*, 2009).

Despite of the fact that this kind of solar cells present lower efficiency compared to traditional sillicon solar cells, the technology based on solar cells sensitized by dyes (DSSC's) is an attractive method for power generation, due to its low cost, and the possibility of using lighter materials, which greatly facilitates the installation of modules and can be applied not only in remote locations, but also for use in the urban area, as well as a source of renewable energy, low cost and easy application. The use of organic dyes as sensitizers to the anodes enables the absorption of light in several wavelengths, which gives greater possibilities, since they can be used in the dark or closed environments.

However, the efficiency of the device needs to be increased in order to enable its use on a large scale. We must optimize the process of manufacture of the photoelectrodes, the encapsulation of the electrolyte, the absorbtion capacity of the dyes and the interaction with the semiconductor oxides, in order to reduce the device losses. If these problems could be solved, this type of technology will certainly be widely used in the world and the DSSC's will become a viable device as a source of usual electricity, both in small and in large scales.

This work, therefore, involves the development and electrical, structural and morphological characterization of dye-sensitized solar cells, with low cost and economically viable, which makes possible, in the future, an extension of the studies on the device and the assembly of panels or small modules, using the cells produced in our laboratory.

5. ACKNOWLEDGEMENTS

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