

Tendências e Progressos da Eletroquímica e Eletroanalítica no Brasil

CARMEN LÚCIA VOIGT
(Organizadora)

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APRESENTAÇÃO

Nos dias atuais estamos diretamente ligados às tecnologias em desenvolvimento. Muitas vezes não nos damos conta de conhecer todos os processos em evolução e nem mesmo todos os produtos desenvolvidos usados em nosso cotidiano. O conhecimento dos métodos e técnicas utilizados auxilia na compreensão dos sistemas modernos que envolvem a ciência, tecnologia, sociedade e meio ambiente.

Relacionando o desenvolvimento à ciência, está a Química, presente em tudo, por isso chamada de ciência central, sendo uma ponte entre outras ciências. Diversos processos tecnológicos usam diretamente relação com a química para avaliação e avanço em suas pesquisas. Dentre suas diversas áreas, encontra-se a Eletroquímica, que estuda o fenômeno da transferência de elétrons para a transformação de energia química em energia elétrica e vice-versa.

A eletroquímica se faz presente em nossa rotina diária incluindo aparelhos tão utilizados como celulares, relógios e computadores, ou seja, quando falamos nestes dispositivos que possuem uma bateria, desconectados de uma fonte de energia elétrica, que seja oriunda de uma fonte geradora, estamos falando em processos relacionados a essa área da química.

Para desenvolvimento tecnológico, a eletroquímica se divide em eletroanalítica que compreende um grupo de métodos analíticos baseado nas propriedades elétricas de um analito em solução. As técnicas eletroanalíticas podem ser utilizadas em caracterizações entre compostos e validação de novos métodos.

Neste volume, organizado para você, apresentamos estudos eletroquímicos de interação entre nanocarreadores e compostos bioativos, estes apresentam elevado potencial terapêutico, mas dificuldade na obtenção de uma forma farmacêutica estável. Neste contexto estudos bioanalíticos empregam a eletroquímica, surgindo uma nova área que está sendo conhecida como Bioeletroquímica.

Além desta nova área de pesquisa, trazemos técnicas de caracterização usadas no estudo do comportamento de células solares fotovoltaicas. O aumento do interesse por aplicações fotovoltaicas tem estimulado intensivas pesquisas em materiais semicondutores que oferecem maior eficiência de conversão para as células solares. Com isso ocorre avanço tecnológico pensando no meio ambiente.

O meio ambiente também se torna foco quando existe a busca de fontes de energias renováveis, sustentáveis e menos agressivas à natureza. Ainda neste volume apresentamos outro ramo muito interessante da eletroquímica: o uso de eletrodos modificados para processos ambientais, a exemplo da reação de eletro-oxidação de glicerol, um subproduto de biocombustível. A redução eletroquímica do CO_2 utilizando novos eletrodos também se trata de estudo ambiental que mostramos neste volume.

Além disso, trazemos neste volume estudos com eletrodo de disco rotatório, um dos dispositivos experimentais mais utilizados no estudo de sistemas eletroquímicos, quando a influência do transporte de massa está presente, sendo trabalhos realizados

com cálculos e esquema numérico. Também são apresentadas tendências de validação de métodos eletroquímicos que visam validar metodologias eletroanalíticas, visando atestar a confiabilidade dos resultados gerados por estas técnicas.

Com base nestes experimentos, convidamos você a aperfeiçoar seus conhecimentos no que se refere à eletroquímica e seus diversos ramos. Os experimentos oportunizam uma nova visão de materiais, processos e técnicas na área, como desenvolvimento de novos sensores e eletrodos modificados, interações eletroquímicas, estudos de caracterizações eletroquímicas, cálculos numéricos e validação de metodologias. Enfim, deduz-se que a eletroquímica tem relação direta com a ciência, tecnologia, sociedade e meio ambiente, uma vez que pode ser utilizada em todas as áreas, tendo diferentes funções, mas com um bem em comum, desenvolver novos materiais, processos, métodos e técnicas para uso do homem em auxílio no seu cotidiano, influenciando direta e indiretamente toda a sociedade.

Boa leitura.

Carmen Lúcia Voigt

SUMÁRIO

CAPÍTULO 1	1
CARACTERIZAÇÃO ELETROQUÍMICA DA INTERAÇÃO ENTRE COMPOSTOS BIOATIVOS E DENDRÍMERO PAMAM IMOBILIZADO NA SUPERFÍCIE DO ELETRODO DE OURO	
Anna Caroline Lima Candido Marílya Palmeira Galdino da Silva Emanuela Gomes da Silva Fabiane Caxico de Abreu Galdino Thiago Mendonça de Aquino	
CAPÍTULO 2	18
CARACTERIZAÇÃO MOTT-SCHOTTKY EM CÉLULA FOTOVOLTAICA DE SILÍCIO	
Francisco Marcone Lima Vanja Fontenele Nunes Antonio Paulo Santos Souza Ana Fabíola Leite Almeida Francisco Nivaldo Aguiar Freire	
CAPÍTULO 3	26
ELECTRODEPOSITION OF CADMIUM TELLURIDE ON CONDUCTIVE GLASS FOR PHOTOVOLTAIC APPLICATIONS	
Raquele Lima Moreira Francisco Nivaldo Aguiar Freire Ana Fabiola Leita Almeida Francisco Marcone Lima Paulo Herbert França Maia Júnior Manuel Pedro Fernandes Graça	
CAPÍTULO 4	36
NANOPARTÍCULAS DE HEXACIANO FERRATO DE RUTÊNIO PARA REAÇÃO DE ELETRO-OXIDAÇÃO DE GLICEROL	
Deracilde Santana da Silva Viégas Deranilde Santana da Silva Ismael Carlos Braga Alves Isaíde de Araújo Rodrigues Aldalea Lopes Brandes Marques	
CAPÍTULO 5	53
UTILIZAÇÃO DE POLIACRILAMIDA-CO-DIALILDIMETILAMÔNIO COMO IMOBILIZADOR DE CUO NA REDUÇÃO ELETROQUÍMICA DE CO ₂	
Ruan Roberto Henriques Jorge Amim Júnior Ana Lucia Shiguihara Robson Valentim Pereira Kênia Silva Freitas	
CAPÍTULO 6	66
CARACTERIZAÇÃO ELETROCATALÍTICA DO CuO/Cu NA REAÇÃO DE REDUÇÃO DE CO ₂ UTILIZANDO ELETRODO DE DISCO ROTATÓRIO	
Ruan Roberto Henriques	

Robson Valentim Pereira
Kênia da Silva Freitas

CAPÍTULO 7	79
ANÁLISE NUMÉRICA DO EFEITO DE REAÇÕES HOMOGÊNEAS NA CORRENTE LIMITE DO ELETRODO DE DISCO ROTATÓRIO	
Pedro Henrique Moura Leal Oswaldo Esteves Barcia Oscar Rosa Mattos	
CAPÍTULO 8	91
CÁLCULO DA IMPEDÂNCIA ELETRO-HIDRODINÂMICA ATRAVÉS DE SÉRIES NUMÉRICAS	
Oswaldo Esteves Barcia Nathália de Almeida Leite da Silva Flávio Vieira Vasques de Sousa Pedro Rupf Pereira Viana Oscar Rosa Mattos	
CAPÍTULO 9	110
TENDÊNCIAS E PERSPECTIVAS NA VALIDAÇÃO DE MÉTODOS ELETROANALÍTICOS	
Sthéfane Valle de Almeida Andressa Galli Maria Lurdes Felsner	
SOBRE A ORGANIZADORA.....	122

ELECTRODEPOSITION OF CADMIUM TELLURIDE ON CONDUCTIVE GLASS FOR PHOTOVOLTAIC APPLICATIONS

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ABSTRACT: Cadmium Telluride (CdTe) films were deposited on transparent conductor fluorine oxide doped tin oxide (FTO) by electrodeposition in aqueous sulfuric acid solution (H_2SO_4), with cadmium sulfate ($\text{CdSO}_4 \cdot 8/3 \text{H}_2\text{O}$) and tellurium dioxide (TeO_2). The electrodeposition mechanism was previously investigated by cyclic voltammetry. The voltammograms detected CdTe electrodeposition at negative potentials, with cathodic peaks at potentials between -0.7

and -0.3 V and anodic peaks about + 0.75 V. The electrodeposition was made by pulse voltammetry, with current periodic reversion. The films conductivity was verified by the Mott-Schottky, method, whose qualitative analyzes revealed a p-type CdTe. The morphology, the chemical composition and the films structure were analyzed by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD). The films presented composition with tellurium excess, confirming the verified conductivity, and predominant cubic structure. From the obtained films, were built 1cm^2 solar cells, utilizing the n-type CdS as a cell optical window, and lithium perchlorate (LiClO_4) and polyaniline based electrodes. The cell electrical parameters were determined by the construction of current-tension curves. The cell presented short circuit current of 0.134 mA, open circuit voltage of 400 mV and maximum power of 0.0216 mW.

KEYWORDS: cadmium telluride, electrodeposition, cyclic voltammetry, photovoltaic solar cell.

1 | INTRODUCTION

The increased interest for photovoltaic applications has been stimulating intensive researches on semiconductors materials which

offer higher efficiency conversion to the solar cells. The cadmium telluride is a good alternative to the conventional silicon cells due to some of its particularly characteristics.

The cadmium telluride is an II B – VI A class binary semiconductor, with 1.5 eV direct band gap and high absorption coefficient ($> 5 \times 10^5 \text{ cm}^{-1}$) in the visible range of the solar spectrum (LUQUE; HEGEDUS, 2003; CHANDER; DHAKA, 2016). Due to this and others electronic and photochemical properties, it is a material much used in photovoltaic structures and other photoelectronic devices such as photodetectors and light emitting diodes (RAMI *et al*, 1998).

CdTe is a semiconductor that can present conductivity of the p or n type. Obtaining other kind occur by doping processes or by variation of the compound stoichiometry. In the doping, impurities are introduced into the structure, such as indium (In), n type dopant, and gold (Au), p type dopant. Changing the stoichiometry, the Cd excess leads to the p type conductivity, while the Te excess leads to the p type conductivity (LUQUE; HEGEDUS, 2003; UENG; YANG, 2012)

CdTe films can be fabricated through many techniques, such as spray pyrolysis, screen printing, sputtering and electrodeposition (CHANDER; DHAKA, 2016; SOLIMAN *et al*, 2001). Electrodeposition is a very attractive technique to obtain thin films, due to process simplicity and low material and energy spending, besides low cost production to large scale and high automation level (RAMI *et al*, 1998; CHEN; WAN, 1994). Through the application of a range of potentials during the electrodeposition, it is possible to obtain CdTe with different conductivities.

The CdTe films formation mechanism was previously investigated by cyclic voltammetry, to verify the cadmium and tellurium deposition potentials. The films conductivity was verified by the Mott-Schottky method. The morphology and chemical and structural compositions were analyzed by SEM, EDS and XRD.

2 | EXPERIMENTAL ASPECTS

The films were obtained from an aqueous sulfuric acid solution (H_2SO_4), with cadmium sulfate ($\text{CdSO}_4 \cdot 8/3 \text{ H}_2\text{O}$) and tellurium dioxide (TeO_2) at concentrations 0.48 M and 0.06 M, respectively. The solution pH was adjusted between 0 and 1 by H_2SO_4 addition and the temperature was kept constant at 70 °C, through thermostatic bath (CHEN; WAN, 1994).

The electrodeposition was realized by pulse, with current periodic reversion. In this method, one cycle is formed by a cathodic current pulse (j_c), followed by an anodic current pulse (j_a). Each pulse is applied in a specified potential, with controlled time. The cycle experimental parameters can be seen at Table 1. For the films formation, 30 cycles were performed.

Cathodic Potential (V)	Cathodic Time (s)	Anodic Potential (V)	Anodic Time (s)
- 0.6	25	+ 0.2	10

Table 1 Pulsed electrodeposition experimental parameters.

The films obtained were chemically treated in cadmium chloride solution ($\text{CdCl}_2 \cdot 2\text{H}_2\text{O}$) 0.03 M in ethylic alcohol for 20 minutes, with previously annealing at 350 °C for 40 minutes in muffle oven.

The scanning electron microscopy and the EDS analysis were realized in SEM Quanta 450 FEG – FEI. The X-ray diffraction patterns were obtained from a D8 Advanced Bruker diffractometer.

All the electrochemical measures (cyclic voltammetry, pulsed electrodeposition and Mott-Schottky method) were executed using an AUTOLAB PGSTAT302N Metrohm potentiostat, with an electrochemical cell containing three electrodes: reference electrode (Ag/AgCl), work electrode (FTO conductive glass substrate, with resistance $17\Omega/\text{cm}^2$) and a platinum (Pt) counter electrode (CHEN; WAN, 1994).

3 | DISCUSSION AND RESULTS

3.1 Pulsed Electrodeposition

Figure 1 represents the generated graphic during electrodeposition, from which it is possible to observe the pulses symmetry and its respective cathodic ($j_c = - 0.06$ A) and anodic ($j_a = + 0.025$ A) current. At the end of the electrodeposition process, the films were as shown in Figure 2.

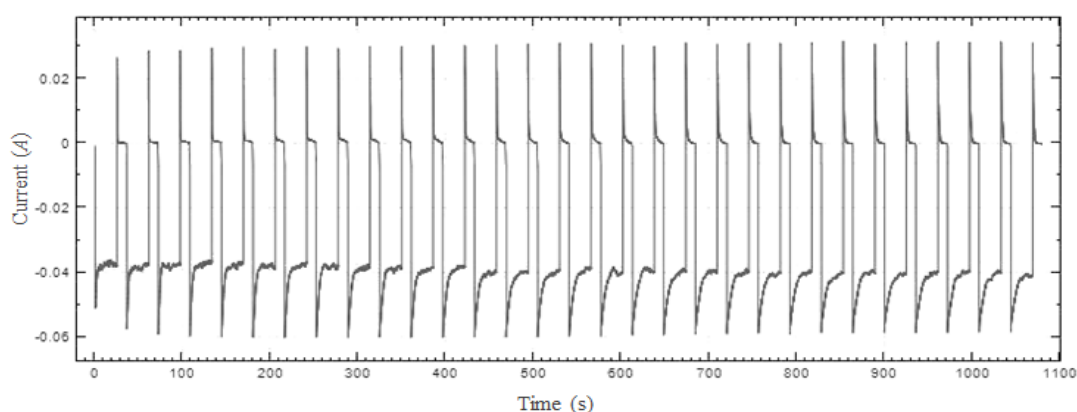


Fig 1. Pulsed Electrodeposition with current reversal graphic.

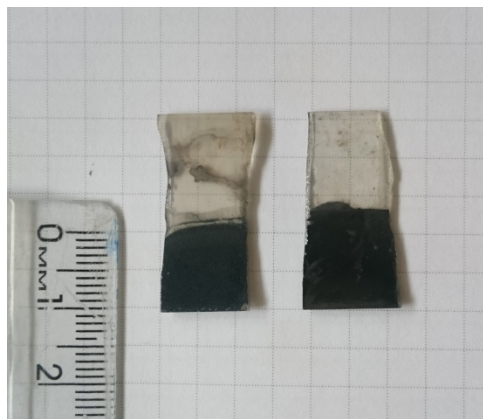


Fig 2. CdTe films on FTO glass substrate. Film area: 1 cm².

3.2 Cyclic Voltammetry

The cyclic voltammetry was performed in an electrochemical solution with 0.48 M CdSO₄ · 8/3 H₂O and 0.06 M TeO₂, at constant temperature of 70 °C. The -1.0 V to +1.0 V potentials were scanned, with a 100 mV/s rate. Figure 3 shows the voltammogram generated after three cycles, each cycle starting a scan from a different potential, so that there is no overlap of the curves. The scanning potentials for cycles (a), (b) and (c) were 0.06 V, 0.04 V and 0.02 V.

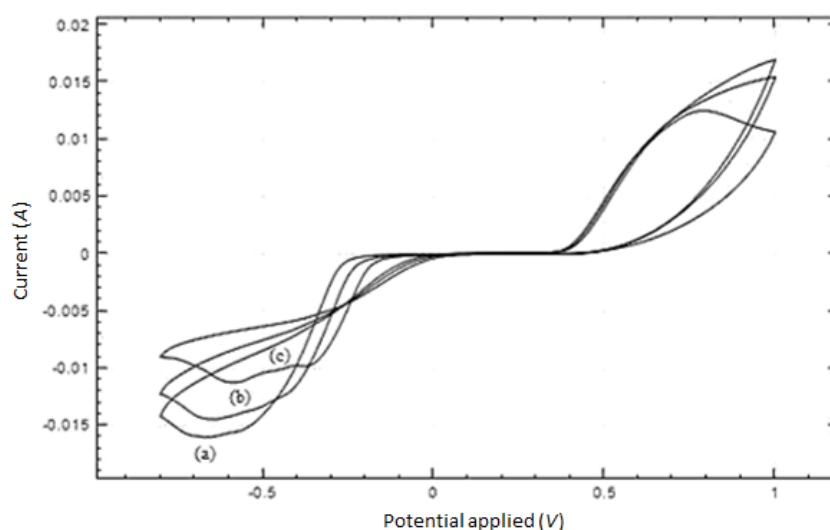
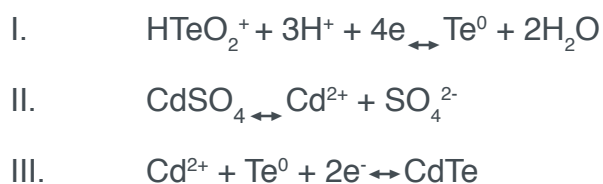


Fig 3. Electrochemical solution cyclic voltammogram with 0.48 M CdSO₄ · 8/3 H₂O and 0.06 M TeO₂, bath at 70 °C.

In the third cycle (c cycle) the peaks related to deposition potentials are more visible, indicating a greater amount of deposited species than in the first two cycles. It is possible to observe, from the c cycle, two cathodic peaks presence at the -0.35 V and -0.6 V potentials, which represent the tellurium and cadmium reduction, respectively. There is an anodic peak, at the potential about +0.75 V, indicating the species oxidation in the work electrode surface. The observed potentials acted accordingly to cyclic voltammograms obtained by Chander and Dhaka (2016), Chen and Wan (1994) and

Yang, Chou and Ueng (2010). The CdTe film formation happens according to the following reactions:



3.3 Mott-Schottky

Capacitance measures utilizing Mott-Schottky analysis were performed to determine the semiconductors properties of the electrodeposited CdTe films. The electrochemical measures were equally executed in AUTOLAB PGSTAT302N potentiostat, realized in the same three electrodes cell used in the electrodeposition, in a NaCl 1 M solution.

The Mott-Schottky method is based on capacitance measures as a function of the applied potential. The capacitance measures offer information about the charges distribution inside the semiconductor, which gives the p or n type semiconductivity. The used equations for the method are Eq. 1, to n type semiconductors, and Eq. 2, to p type semiconductors:

$$\frac{1}{C_{sc}^2} = \frac{2}{\epsilon_0 \epsilon_r N_D A^2} \left(E - E_{FB} - \frac{KT}{e} \right) \quad (1)$$

$$\frac{1}{C_{sc}^2} = \frac{2}{\epsilon_0 \epsilon_r N_A A^2} \left(E - E_{FB} - \frac{KT}{e} \right) \quad (2)$$

Where ϵ_r is the film relative dielectric constant, ϵ_0 is the free space permittivity ($8.854 \times 10^{-14} \text{ F cm}^{-1}$), e is the electron charge ($1.602 \times 10^{-19} \text{ C}$), A is the film area (cm^2), N_A and N_D are the charge donors density (cm^{-3}) and the charge acceptors density (cm^{-3}), E_{FB} is the flat band potential (V), K is the Boltzmann ($1.38 \times 10^{-23} \text{ J K}^{-1}$) constant and T the absolute temperature (in Kelvin) (REN *et al*, 2015).

The electronic conduction behavior can be analyzed through the method plotted curve. The Mott-Schottky graphic qualitative analyzes allows the conductivity characterization in p or n type, from the presented inclination. A positive inclination relates to the n type conductivity, while a negative inclination relates to the p type conductivity (REN *et al*, 2015; LIU, 2014), as the one which was verified to the electrodeposited CdTe films (Figure 4).

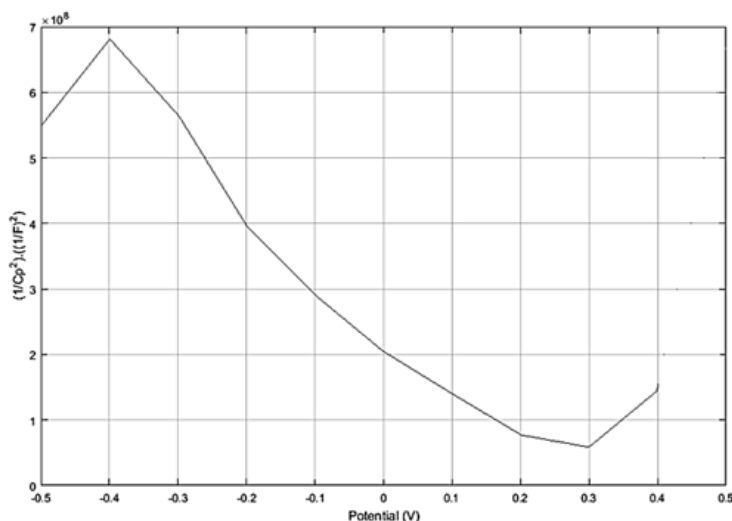


Fig 4. Mott-Schottky curve. Negative inclination indicates p type semiconductivity.

For CdTe, a p type conductivity indicates Te excess in the sample [1], fact proved by the following EDS analysis.

3.4 Chemical and Morphology analysis (SEM and EDS)

The CdTe electronic microscopy electrodeposited at -0.6 V is presented in the Figure 5.

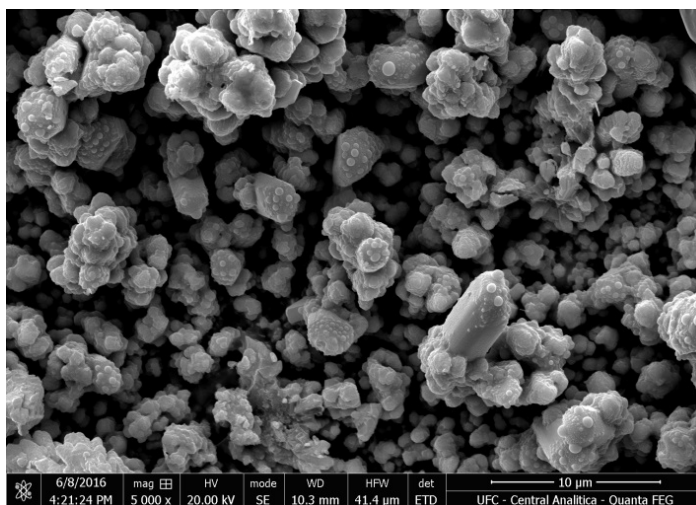


Fig 5. CdTe film SEM obtained at - 0.6 V, 70 °C. 5.000x magnitudes.

It is possible to observe a nodular morphology, with agglomerates similar to a “cauliflower” (DHARMADASA *et al*, 2014) and many voids, which will directly influence device performance. However, the film came out without cracks.

The chemical composition of the film was analyzed by EDS, whose spectrum (Figure 6) revealed the presence of cadmium and tellurium ions, the main film components, besides residual species from the reagents involved in the FTO substrate formation, as chlorine and tin (the fluorine doped tin oxide ($\text{SnO}_2:\text{F}$) is obtained from an ammonium fluoride (NH_4F) and tin chloride (SnCl_2) solution (GORDILLO *et al*, 1994).

The gold present in the sample is a contribution from the metallization stage, realized on the sample previously to the microscopy analyses. The film was formed by Te 71.7% and Cd 19.9%, which assigned type p conductivity, as previously verified by the Mott-Schottky method.

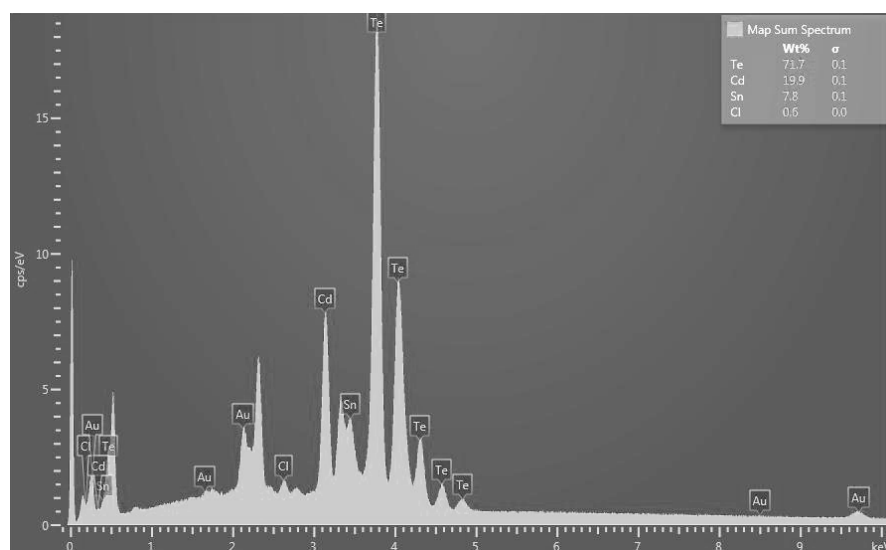


Fig 6. CdTe film EDS obtained at -0.6 V, 70 °C.

3.5 X-Ray Diffraction

The electrodeposited CdTe film diffractogram on FTO substrate glass is presented in Figure 7.

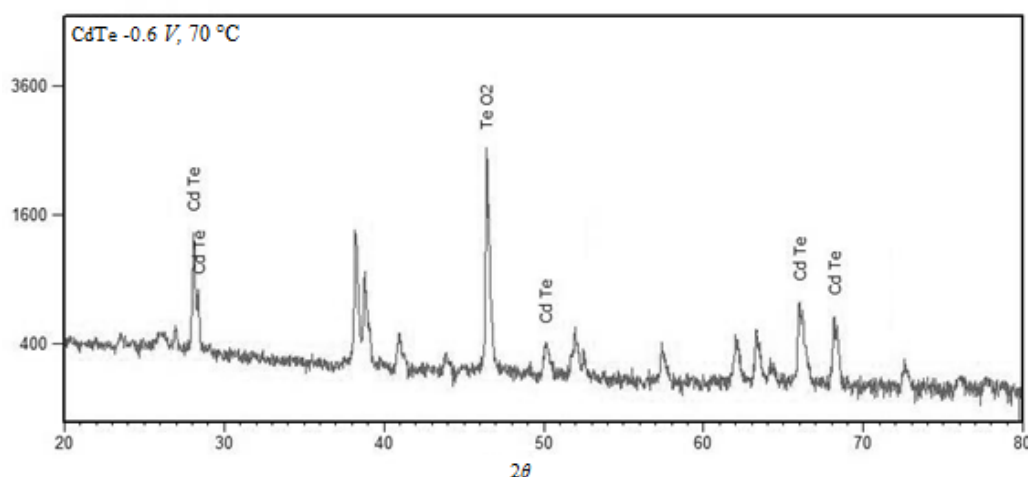


Fig 7. X-ray pattern of the CdTe sample electrodeposited, at -0.6 V and annealed at 350 °C.

The crystallographic file identified the CdTe as present in the compound as of cubic phase, with net parameters about $a = b = c = 6.3200$ Å; $\alpha = \beta = \gamma = 90^\circ$. The higher intensity peak corresponds to the CdTe located at $2\theta = 28^\circ$, approximately, indicating strong preferential orientation along the reflection plane (111), referring to the cubic structure [CHANDER; DHAKA, 2016; SOLIMAN *et al*, 2001; TREFNY *et al*, 1993). The other present peaks are due to noises and other present species in the

sample, as the peak at $2\theta = 57^\circ$, which corresponds to the TeO_2 .

3.6 Device assembly and electrical characterization

To prove the films applicability to photovoltaic purposes, devices were built from the CdTe obtained. Considering the p type semi conductivity achieved, cadmium sulfide was utilized as the cell optical window. CdS is an n type intrinsically semiconductor, much used with the CdTe, forming heterojunction CdTe/CdS cells (TREFNY *et al*, 1993; LI; LIU, 2015; SIVAKUMAR; HARIHARAN; ABITH, 2012). A lithium perchlorate (LiClO_4) and polyaniline based electrode chitosan dispersed was introduced in the structure, in a way that the device presented the glass configuration FTO/CdTe/electrolyte/CdS/FTOglass, as represented in Figure 8.

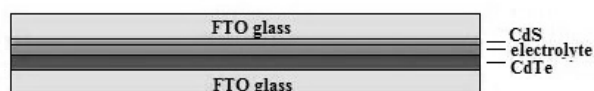


Fig 8. Schematic representation of the prototype cell.

The tests under illumination were realized with the device exposed to a 105 mW/cm^2 irradiation, corresponding to the utilized led maximum power, generating a characteristically cell I - V curve, Presented in Figure 9.

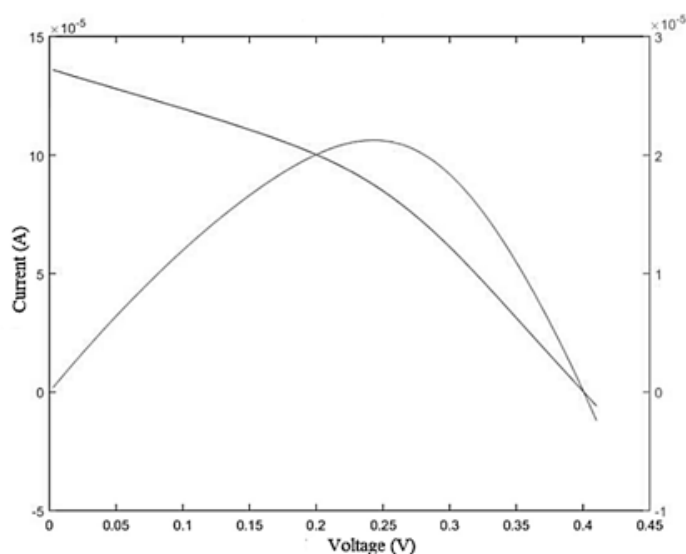


Fig 9. Cell characteristic I - V curves.

The device electrical parameters were estimated from the I - V curve generated data, obtaining the short circuit value of 0.134 mA (I_{sc}), open circuit voltage of 400 mV (V_{oc}) and maximum power of 0.0216 mW (P_{max}).

In 2002, a group from the National Renewable Energy Laboratory (NREL) obtained the maximum efficiency record of 16.5% to laboratory scale CdTe/CdS cells. Such cells has CdTe and CdS layers deposited by sublimation in reduced space and chemical bath, respectively, and presented- under 100 mW/cm^2 illumination- the parameters V_{oc}

= 850 mV e $I_{sc} = 25.88$ mA (LEE, 2011).

It is possible to observe, from this data, that the fabricated cell presented current low value and a V_{oc} 450 mV lower, which can be related to the presence of cell intern resistances and to current escapes.

The intern layers and interphases that compose the cell can offer resistance to the current passage, resulting in electrical losses. The current shunts can occur due to tunnels through the junction imperfections, short circuit due to layers holes (as the voids present in the CdTe films, observed by SEM) and conduction by surface.

The open circuit voltage is related to the semiconductor Fermi level position, in relation to the electrolyte potential, with maximum voltage valued provided by the cell, being the difference between both. This way, the semiconductor/electrolyte interphase behavior must have contributed to the V_{oc} reduced value.

The obtained values confirm, then, the cell photovoltaic behavior, with the necessity, however, of improving the components layers, to elevate the electrical parameters and, as a consequence, the device efficiency conversion.

4 | CONCLUSIONS

The pulsed electrodeposition with current reversion made possible to obtain compact and partially uniform CdTe films, presenting itself as an efficient technique to form thin semiconductors films. The voltammetric experiments allowed observing the CdTe formation region. The XRD revealed a cubic structure CdTe. The nodular morphology was observed by SEM and the p type semi conductivity was proved by the Mott-Schottky method and the EDS analysis, which presented the film composition as being 71.7% Te and 19.9% Cd. The cell electrical parameters proved the film applicability to photovoltaic purposes, indicating, however, the necessity of reducing the intern resistances, improving the cell components and interphases, to elevate the device parameters and efficiency values.

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