Cleberton Correia Santos
(Organizador)

Estudos Interdisciplinares nas Ciências e da Terra e Engenharias 2

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APRESENTAÇÃO

O livro "**Estudos Interdisciplinares nas Ciências Exatas e da Terra e Engenharias**" de publicação da Atena Editora apresenta em seu 2º volume 35 capítulos relacionados temáticas de área multidisciplinar associadas à Educação, Agronomia, Arquitetura, Matemática, Geografia, Ciências, Física, Química, Sistemas de Informação e Engenharias.

No âmbito geral, diversas áreas de atuação no mercado necessitam ser elucidadas e articuladas de modo a ampliar sua aplicabilidade aos setores econômicos e sociais por meio de inovações tecnológicas. Neste volume encontram-se estudos com temáticas variadas, dentre elas: estratégias regionais de inovação, aprendizagem significativa, caracterização fitoquímica de plantas medicinais, gestão de riscos, acessibilidade, análises sensoriais e termodinâmicas, redes neurais e computacionais, entre outras, visando agregar informações e conhecimentos para a sociedade.

Os agradecimentos do Organizador e da Atena Editora aos estimados autores que empenharam-se em desenvolver os trabalhos de qualidade e consistência, visando potencializar o progresso da ciência, tecnologia e informação a fim de estabelecer estratégias e técnicas para as dificuldades dos diversos cenários mundiais.

Espera-se com esse livro incentivar alunos de redes do ensino básico, graduação e pós-graduação, bem como outros pesquisadores de instituições de ensino, pesquisa e extensão ao desenvolvimento estudos de casos e inovações científicas, contribuindo na aprendizagem significativa e desenvolvimento socioeconômico rumo à sustentabilidade e avanços tecnológicos.

Cleberton Correia Santos

SUMÁRIO

CAPÍTULO 35

PHOTOELECTROCATALYSIS PROPERTIES OF $CUWO_4$ POROUS FILM UNDER POLYCHROMATIC LIGHT

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ABSTRACT: Triclinic CuWO4 structure particles were deposited onto transparent conducting glass electrodes to prepare porous photoelectrode with 1.0 cm^2 of area, which was then used as photocatalysts for Rhodamine B (RhB) degradation. After 165 min under polychromatic irradiation with the solar simulator, the $CuWO_4$ porous film promoted 35.4% of RhB removal. Superior efficiency was reached when $CuWO_4$ photoelectrode was biased at + 0.7 V, with RhB removal corresponded to 45.7%. Applying potential bias on electrode minimizes the recombination of photogenerated charge carriers, resulting in higher performance for organic pollutant degradation.

KEYWORDS: *$CuWO_4$, Visible light, photoelectrocatalysis, Rhodamine B.*

1 I INTRODUCTION

The release of hazardous compounds into the water from the urban and the industrial sectors has been a problem due to the presence of a large variety of chemical species at different concentrations (Youssef et al. 2018; Bello and Raman 2018). Synthetic dyes belong to a notable group of pollutants because of their high toxicity and carcinogenic properties. Several industries, including textiles, food technology, cosmetics, in photoelectrochemical cells and pharmaceuticals produce high concentrations

of dyes in water resources (Xie et al 2018; Verma and Samanta 2018; Alcocer et al. 2018). The discharge these colored compounds in the environment raises much concern because of the toxic effects, carcinogenic and mutagenic. Furthermore, the presence of these contaminants in wastewaters can inhibit the penetration of sunlight by consuming dissolved oxygen and thereby compromising many aquatic organisms (Ismail et al. 2018; Mahmoud et al. 2017).

Currently, advanced oxidation processes (AOPs) are one of the most innovative solutions for decontamination of organic pollutants hardly biodegradable. Among them, heterogeneous photocatalysis (HP) using semiconductor oxides emerges as a promising wastewater treatment technology (Osman, Su and Ma 2017; Filho et al. 2018). In general, photocatalytic reactions are initiated by the excitation of the photocatalytic surface with sufficient energy, resulting in generation of an electron (e^-) / hole (h^+) charge pair on semiconductor surface. After that, the series of reaction mechanism involves the production of highly reactive hydroxyl radicals (HO•) that are effective for totally removing organic compounds (Costa at al. 2018; Oliveira et al. 2010).

Among semiconductors, copper tungstate $CuWO_4$ crystals has attracted increasing interest as photocatalyst for the degradation of some refractory organic pollutants (Dong et al. 2019). This semiconductor can harvest visible-light ($l > 500$ nm) and is chemically stable in a large pH range (Wu et al. 2019). However, $CuWO_4$ has proved photocatalytic activity low for applications involving the catalytic material dispersed in suspension in the reaction medium, due to the rapid recombination of photogenerated hole-electron pairs and the slow carrier mobility in the bulk. Thus, it is desirable to improve the photocatalytic activity this material (Li et al. 2018; Xie et al. 2016) Moreover, an alternative strategy consists in retard recombination of the charge carriers through the application of a positive potential bias to the material supported as a film. In this condition, the HP system can be called of electrochemically assisted (EHP), which increased degradation efficiency (Costa et al. 2018).

In general, the literature has reported that the presence of the porous structure in the photoelectrodes provides in increased interfacial area between the electrolyte and the film, which can facilitate overcoming possible charge separation limitations (Kim et al. 2011; Hagfeldt and Graetzel 1995) Furthermore, the porous structure can provide a better diffusion of the electrolyte and minimization the recombination of photogenerated charge carriers before participating in the electrochemical reaction (Pilli at al. 2011).

In the last years, our group have recently shown the co-precipitation-hydrothermal synthesis of $CuWO_4$ porous film and their structure and photoelectrochemical properties (Lima et al. 2017). The study show that the $CuWO_4$ porous film is a promising candidate to be applied as a photoanode in photocatalytic processes under irradiation by visible light. Furthermore, structural refinement and

photocatalytic properties of $CuWO_4$ crystals (Souza et al. 2017; Souza et al. 2014) Therefore, in this communication, we have evaluated the effect of applied potential on photocatalytic performance of $CuWO_4$ porous film were employed to investigation to photodegradation rate of RhB dye in aqueous solutions using the HP and EHP process.

2 I EXPERIMENTAL DETAILS

2.1 Preparation of CuWO4 porous films

The synthetization of copper tungstate ($CuWO_4$) and preparation of photoelectrode have been reported by the authors' previous paper (Lima et al. 2017). Briefly, to prepare $CuWO_4$ photocatalyst, 2.0 mmol of copper nitrate trihydrate [$Cu(NO_3)_2.3H_2O$] and 2.0 mmol of sodium tungstate dihydrate ($Na_2WO_4.2H_2O$) were dissolved in 100 mL of deionized water to form the suspension. In following, the suspension was heated in a Teflon lined autoclave at 200ºC±5 for 8h. The suspension prepared with CuWO4 nanocrystals and polyethylene glycol (PEG, *MW* ≅ 20.000) is coated onto fluorine doped tin oxide (FTO) coated glass substrate with surface resistivity ~ 7Ω/sq (TCO22-7, Aldrich, _7 V/sq) using doctor-blade method. Finally, the $CuWO_4$ porous films were obtained after heating at 500 ºC for 30 min in a conventional muffle furnace.

2.2 Characterization of CuWO4 film

The CuWO4 porous films was examined by X-ray diffraction (XRD) in order to evaluated their crystalline structure. XRD patterns were obtained using a LabX XRD-6000 diffractometer (Shimadzu, Japan) with Cu-Ka radiation (l= 0.15406 nm) in the 2q range from 10º to 70º with a scanning rate of 1º/min. Morphological observations of the $CuWO_4$ porous films deposited on the FTO-glass was performed by Field Emission Scanning Electron Microscopy (FE-SEM; Carl Zeiss, Model Supra 35-VP, Germany), using a voltage of 2 kV.

2.3 Photocatalytic activity measurements

For photocatalytic activity investigation, $CuWO_4$ porous films as electrode with 1.0 cm^2 was utilized as work electrode in a photoelectrochemical cell (PEC) configured with three electrodes. To complete the PEC, platinum (Pt) wire and Ag/AgCl electrode were utilized as counter electrode and reference electrode, respectively. In the PEC, 10 mL of [9-(2-carboxyphenyl)-6-diethylamino-3-xanthenylidene]-diethylammonium chloride, also known as tetraethylated Rhodamine or Rhodamine B (RhB; 95%, Sigma-Aldrich, with a λmax = 543 nm) solution with initial concentrations (*C*0) of 1.0 mmol L^{-1} was utilized for studies of photodegradation. Na_2SO_4 0.1 mol L^{-1} was utilized as supporting electrolyte. The cell was irradiated with a polychromatic metallic vapor discharge lamp (HQI-TS NDL), with a nominal potency of 150 W. The system

temperature was maintained at 24 ± 5 ºC.

The reaction kinetics for degradation of RhB dyes was investigated for HP and EHP configuration for $CuWO_4$ porous films as electrode no polarized and under bias voltage of +0.7 V, respectively. Before the irradiation, the system was maintained in the dark for 30 min to reach complete adsorption–desorption equilibrium. The systems were not stirred and not bubbled with any gas in during the measurements. The variations in the maximum absorption band of pollutant were monitored by ultraviolet (UV–vis) spectroscopy using a UV-2600 (Shimadzu) spectrophotometer at the characteristic wavelength.

3 I RESULTS AND DISCUSSION

3.1 Morphological and structural characterization of the CuWO4 photoelectrode

Details about the structure and degree of crystallinity of the $CuWO_4$ porous films was obtained from XRD studies. Fig. 1 shows the XRD patterns of $CuWO_4$ film prepared at annealing temperature at 500ºC for 30 min and the FTO-glass, respectively. As can be seen the diffraction peaks from the $CuWO_4$ film were perfectly indexed to the wolframite-type triclinic structure (Inorganic Crystal Structure Data (ICSD) card No. 16009). The signals indexed at 2θ equal to 38.28°, 44.45°, 64.80° and 77.80° are attributed to the conductive layer of the SnO_2:F phase of the FTO-glass used as a substrate (Costa et al. 2018).

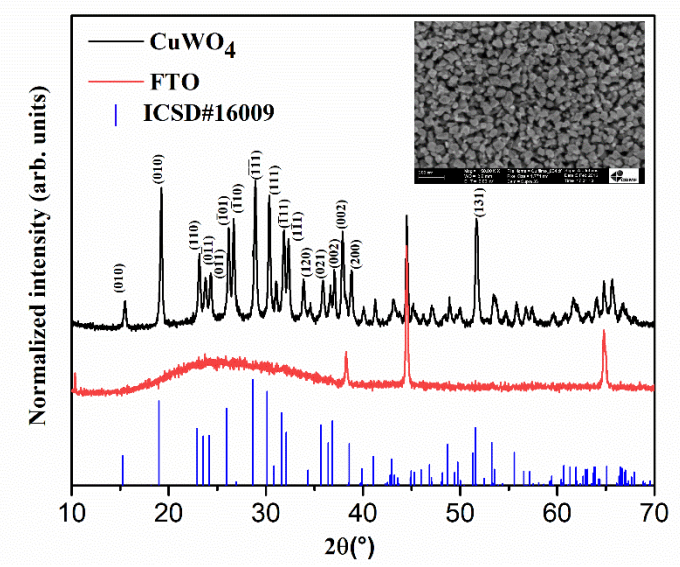

Fig. 1: XRD patterns of the FTO substrate and $CuWO_4$ film annealed at 500 °C for 30 min, respectively. The vertical bars represent the ICSD card No. 16009 for the $CuWO_4$ phase with triclinic structure. The insert shows the FE-SEM image of the surface of the $CuWO_4$ film.

The surface morphology of the annealed $CuWO_4$ porous films is illustrated in Fig. 1 (insert). FE-SEM micrographs showed that the $CuWO_4$ porous films deposited

on glass-FTO exhibit an agglomerated particle with a porous structure and average size distribution ranging from 60 to 130 nm. The design of porous electrodes offers high surface area, which can be significantly increases the photocatalytic activity (Patel et al. 2018).

3.2 Evaluation of CuWO4 film in RhB oxidation

In order to evaluate the possible application of porous $CuWO_4$ electrode for RhB photoelectrocatalytic degradation an energy diagram for the dyelsemiconductor interface was assembled, considering the positions of conduction band (CB) and valence bands (VB) for the semiconductor and the lowest unoccupied molecular orbital (LUMO) and highest occupied molecular orbital (HOMO) energies for the RhB dye. In a previous study (Lima at al. 2017), we described the construction of a surface band diagram for the porous $CuWO_4$ electrode using a combining of flat band potential (Efb) and band gap energy (EBG) derived from photoelectrochemical and UV-Vis spectroscopy analysis, respectively. Assuming that the EBG is associated to the photocurrent onset, the Efb for the $CuWO_4$ electrode was estimated as 0.34 V *vs* (Ag/AgCl at pH 5.6). This value provides an approximation of the CB edge for the n-type semiconductor. In addition, our experimental UV-Vis spectroscopy data showed an EBG of about 2.45 eV for $CuWO_4$. Thus, the value found was related to valance band edge, since the difference between CB e VB may be approximate to EBG of the semiconductor. In both cases, the edge values were estimated through on the vacuum scale [E(eV) = 4.5 e_{ERHE}(V)], where *e* is the elementary charge and ERHE is the potential with respect to the reversible hydrogen electrode (Bard and Faulkner 20111). The CB and VB edges for $CuWO_4$ porous films as electrode correspond respectively to -5.4 eV and -7.8 eV as represented in Fig. 2(c).

For organic compounds, the oxidation potential value can be attributed to the HOMO level (Andrade at al. 2005). Fig. 2(a). shows the cyclic voltammogram (CV) containing 1.0 mmol L^{-1} of RhB (0.1 mol L^{-1} Na_2SO_4, as supporting electrolyte) registered at 20 mV s^{-1} in the dark using $CuWO_4$ porous films working as electrode. In the potential range between +0.1 to +1.2 V *vs* (Ag/AgCl at pH 5.7), the electrode exhibited anodic peak at around 0.95 V that can be attributed to the oxidation of RhB on $CuWO_4$ film surface. The HOMO energy for RhB derivative is 6.0 eV. The HOMO-LUMO energy gap for RhB dye can be associated with its absorption in UV-Vis region (Fu at al. 2005). Fig 2(b). illustrates the absorption spectrum obtained for a solution containing RhB dye dissolved in 0.1 mol L^{-1} Na_2SO_4 electrolyte. The gap between the HOMO and LUMO energies was determined from maximum absorption band of dye with wavelength of 553 nm. The LUMO energy level is approximately eV, given by the mathematical relationship, E (eV) = 1240 / λ (nm).

The energy diagram for the semiconductor/RhB interface in aqueous solution is illustrated in Fig 2(c). The result indicates that dye can be oxidized by photogenerated holes at the semiconductor surfaces, since the HOMO potential of the dye is sufficiently

positive compared to the valence band edge of semiconductor. In this condition, the electrons photo-generated by absorption of photons from the HOMO energy level of RhB dye are transfer to the conduction band of the semiconductor, where they can be picked up by the dissolved oxygen in water producing various species with strong oxidative capacity. Furthermore, this study shows that RhB dye could be degraded within a certain wavelength range of visible light over $CuWO_4$, especially sunlight (Fu at al. 2005; Chang at al. 2012).

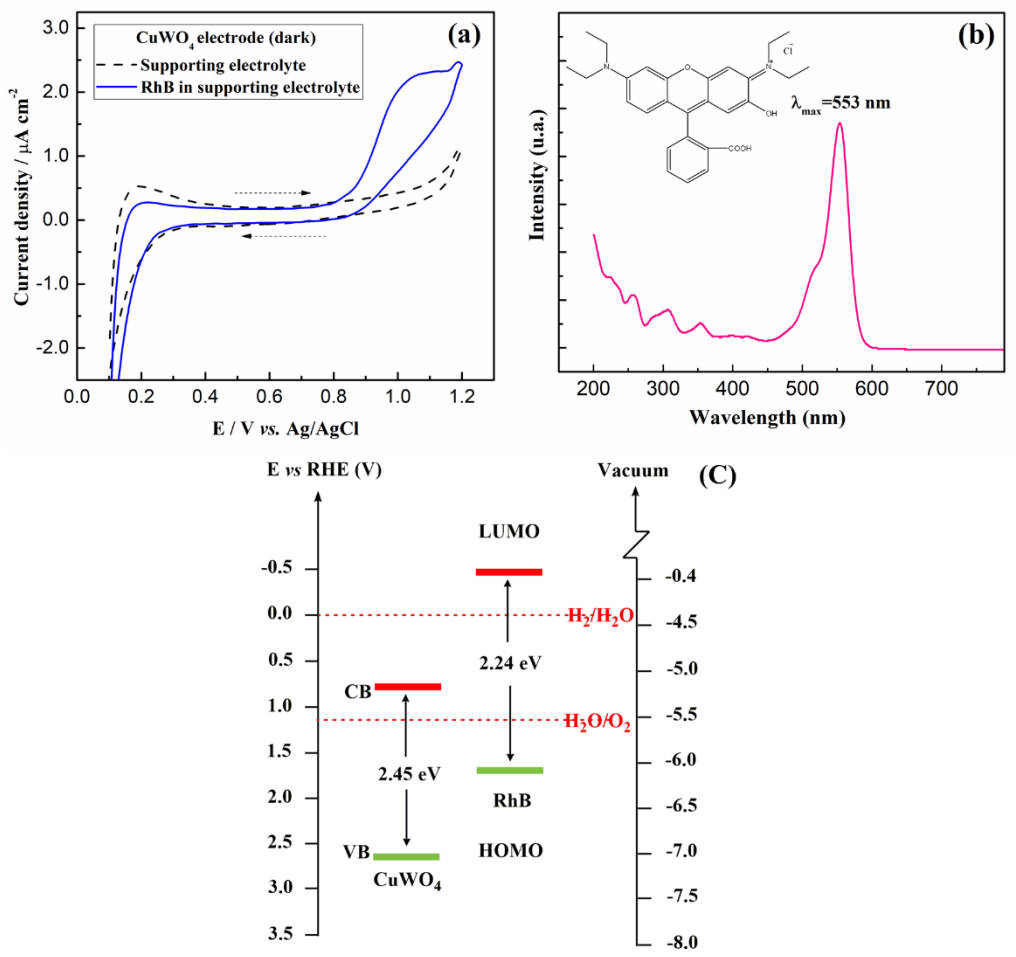

Fig. 2: (a) Cyclic voltammograms (20 mV s^{-1}) for $CuWO_4$ electrode in the dark, in 0.1 mol L 1 Na2SO4 aqueous solution and in supporting electrolyte containing RhB dye, (b) Absorption spectrum obtained for a solution containing RhB dissolved in 0.1 mol L^{-1} Na_2SO_4 and (c) Energy diagram for the semiconductor/RhB dye interface in aqueous solution, considering the highest occupied and the lowest unoccupied molecular orbitals, as well as the valence and conduction band edges for $CuWO_4$ electrodes.

3.3 Photocatalytic activity of $CuWO_4$ porous film as electrode for degradation of RhB dye

As described early, the photoelectrocatalytic activity of the porous $CuWO_4$ electrode, with immobilized particles density ca. 1.7 ± 0.1 mg cm^{-2}, was evaluated from RhB degradation using systems in HP and EHP configurations. For comparison effect, an RhB solution irradiated without photocatalyst, just the FTO-glass, was also investigated (photolysis). For HP configuration, none potential was applied, while

EHP configuration was studied for by applying a bias of + 0.7 V in order to suppress the electron-hole (e^-/h^+) recombination. Fig. 3. shows the degradation efficiency of the RhB aqueous solution, and linear sweep voltammetry of a $CuWO_4$ film obtained at 1 mV/s under chopped illumination.

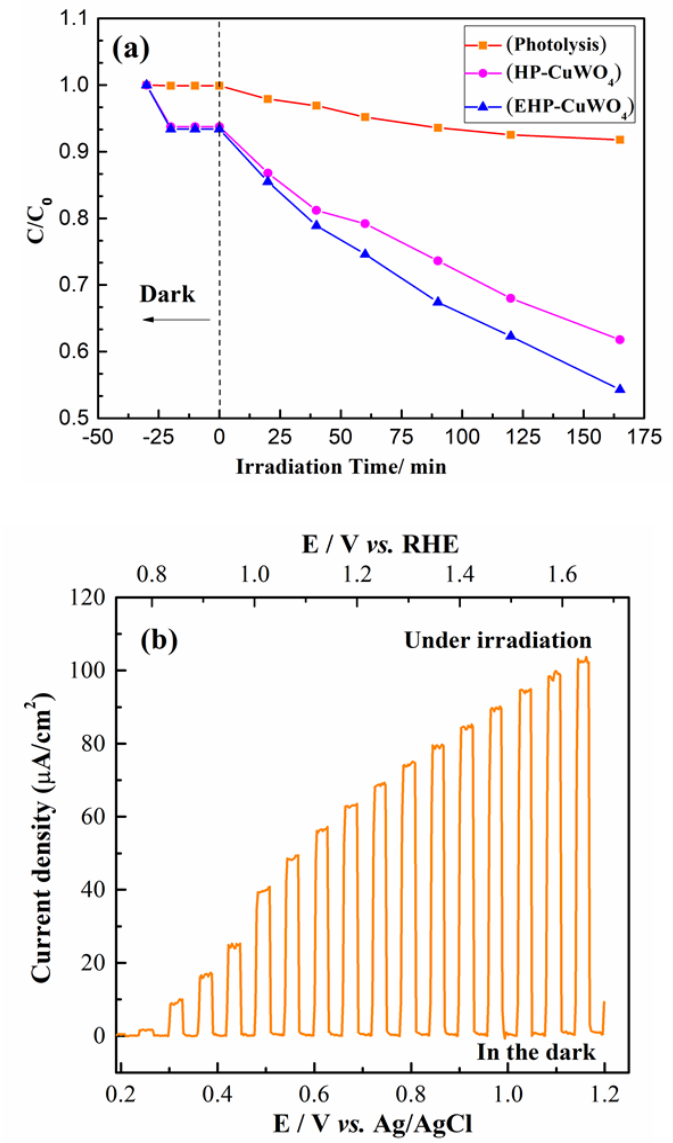

Fig. 3: (a) Degradation efficiency of RhB aqueous solution under polychromatic irradiation by photolysis, heterogeneous photocatalysis (HP) and electrochemically-assisted HP (EHP) using $CuWO_4$ electrodes and (b) Linear sweep voltammetry curves of $CuWO_4$ under chopped light illumination in an 0.1 mol L^{-1} Na2SO4 aqueous solution.

As shown in Fig. 3a. after 30 min adsorption in the dark, only small amounts of RhB were adsorbed and, for photolysis condition, only 8.3% of the dye was removed. Meanwhile, the HP and EHP process exhibited the highest photocatalytic activity for the decomposition of RhB. The extent of degradation of the dye reached 35.5% and 45.5%, respectively within 165 min. As expected, the electrochemically-assisted system exhibits superior efficiency due to its capacity for retard the recombination of electron-hole at the surface of the semiconductor through a gradient of potential generated by the biased electrode. In addition, the electrical bias increases the lifetime

of photogenerated charges leading to higher photocatalytic efficiency, which can be confirmed by photocurrent measurements (Garcia-Segura and Brillas 2017; Tang at al. 2019). As seen in Fig. 3b., the $CuWO_4$ electrode shows higher photocurrent density (68 mA cm^{-2} at 0.73 V *vs.* Ag/AgCl) at more positive potentials, indicating that applied bias facilitated the extraction of photoinduced e^-CB by the external electrical circuit, thereby yielding an efficient separation of the charge carriers.

4 I CONCLUSIONS

In summary, we have obtained with success the $CuWO_4$ porous film as electrodes deposited onto FTO-glass substrate. According to XRD patterns exhibited triclinic structure and FE-SEM images shown the microstructure porous with a uniform pore distribution. The electrode exhibited superior photocatalytic activity of 49% of degradation of RhB dye using EHP process, after external connection to a Pt counter-electrode and application of a potential bias of 0.7 V with a potentiostat. The applied potential suppresses the recombination of charge carriers and enhances the photocatalytic activity of the $CuWO_4$ porous film as electrodes for degradation of RhB dye and other organic molecules.

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