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A GERAÇÃO DE NOVOS CONHECIMENTOS NA QUÍMICA 2

Eleonora Celli Carioca Arenare
(Organizadora)

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APRESENTAÇÃO

A proposta implícita nessa coletânea fundamenta-se numa valorização eclética da pluralidade e diversidade, que reúne pesquisas que envolvem diversas linhas de abordagem, destacando-se por meio de tendências de estudos envolvendo a Ciência "Química". Tendo como propósito principal disseminar e divulgar no meio acadêmico, envolvido com tal Ciência, informações provenientes de estudos e pesquisas desenvolvidas pela comunidade acadêmica contemporânea.

O e-book "A Geração de Novos Conhecimentos na Química", está dividido em dois volumes, totalizando 46 artigos científicos, destacando-se temáticas pesquisadas e discutidas por estudantes, professores e pesquisadores. Os quais evidenciam, artigos teóricos e pesquisas de campo, abrangendo a linha de Ensino e diversas outras linhas de estudo, que se desenvolveram por meio de pesquisas laboratoriais.

O volume I aborda tendências, envolvidos com a área de Ensino de Química, os quais dão ênfase as seguintes abordagens: Ensino Remoto, Experimentação, Concepções Pedagógicas, Bioinformática, Contextualização, Jogos Lúdicos, Redes Sociais, Epistemologia, Formação de Professores, Habilidades e Competências e Metodologias utilizadas no processo de Ensino e Aprendizagem.

O volume II aborda temáticas de cunho experimental, desenvolvidas e comprovadas por meio das análises desenvolvidas em diferentes universidades brasileiras, dando ênfase à: Química Inorgânica, Eletroquímica, Química Orgânica, Química dos Alimentos, Quimiometria, Química Analítica, Química Biológica, Nanoquímica e Processos Corrosivos.

A coletânea é indicada para àqueles (estudantes, professores e pesquisadores) envolvidos com a Ciência "Química", que anseiam por intermédio de informações atualizadas, apropriarem-se de novas informações, correlacionadas a pesquisas acadêmicas, tendo desta forma, novas bases de estudo e investigação para a aquisição e construção de novos conhecimentos.

Excelente leitura!

Eleonora Celli Carioca Arenare

SUMÁRIO

SUMÁRIO

CAPÍTULO 16

RHODAMINE B PHOTODEGRADATION OVER Ag_3PO_4/SBA-15 UNDER VISIBLE RADIATION BASED ON WLEDS LIGHT

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ABSTRACT: Rhodamine B (RhB) is a dye belonging to the class of xanthene, very soluble in water and with great chemical stability, causing adverse effects to the natural resources whenever discarded without previous treatment. This work was developed to evaluate for the first time the photodegradation of RhB dye in an aqueous solution containing pure silver phosphate (Ag_3PO_4) and supported in SBA-15 mesoporous silica as catalysts, irradiated by white light-emitting diodes (WLEDs). The results of the structural investigation realized by Raman and infrared spectroscopy revealed that both the pure crystals of Ag_3PO_4 and the solids impregnated Ag_3PO_4/SBA-15 (%), presented active modes characteristic of the crystalline phase, as well as high degrees of purity and organization at short range. In addition, it was possible to observe for the composites of Ag_3PO_4/SBA-15, some active modes in the infrared referring to the amorphous phase of the SBA-15 molecular sieve. The samples of pure Ag_3PO_4 and Ag_3PO_4/SBA-15 (5, 10, and 20%) showed indirect bandgap energies of 2.40, 2.33, 2.34, and 2.39 eV, respectively, indicating that these samples are capable of using visible electromagnetic radiation in photocatalysis processes. The photocatalytic tests showed that RhB was photodegraded in 97.35% using pure Ag_3PO_4 and 83.81, 89.79. and 96.98% using Ag_3PO_4/SBA-15 in proportions 5, 10, and 20% under irradiation the WLEDs light for 80 minutes, as well as a good catalytic activity during the three reuse cycles.

KEYWORDS: Photocatalysis, Silver phosphate, Ag_3PO_4/SBA-15, Rhodamine B dye, WLEDs.

FOTODEGRADAÇÃO DE RODAMINA B SOBRE Ag_3PO_4/SBA-15 SOB RADIAÇÃO VISÍVEL BASEADA EM LUZ DE WLEDS

RESUMO: A rodamina B (RhB) é um corante pertencente à classe dos Xantenos, bastante solúvel em água e com grande estabilidade química, causando efeitos adversos aos recursos naturais sempre que descartado sem tratamento prévio. Este trabalho foi desenvolvido com o objetivo de avaliar pela primeira vez a fotodegradação do corante RB em solução aquosa, contendo fosfato de prata (Ag_3PO_4) puro e suportado em sílica mesoporosa SBA-15 como catalisadores, irradiados por diodos emissores de luz branca (WLEDs). Os resultados da investigação estrutural realizada por espectroscopia Raman e de infravermelho revelaram que tanto os cristais puros de Ag_3PO_4 quanto os sólidos impregnados Ag_3PO_4/SBA-15 (%), apresentaram modos ativos característicos da fase cristalina, bem como alto grau de pureza e de organização a curto alcance. Além disso, foi possível observar para os compósitos de Ag_3PO_4/SBA-15, alguns modos ativos no infravermelho referentes a fase amorfa da peneira molecular SBA-15. As amostras de Ag_3PO_4 puro e Ag_3PO_4/SBA-15 (5, 10 e 20%) apresentaram energias de banda proibida indiretas de 2,40, 2,33, 2,34 e 2,39 eV, respectivamente, indicando que essas amostras são capazes de utilizar radiação eletromagnética visível em processos de fotocatálise. Por fim, os testes fotocatalíticos mostraram que o RB foi fotodegradada em 97,35% usando Ag_3PO_4 puro e 83,81, 89,79 e 96,98% utilizando Ag_3PO_4/SBA-15 nas proporções 5, 10 e 20% sob irradiação a luz de WLEDs por 80 minutos, bem como boa atividade catalítica durante os três ciclos de reutilização.

PALAVRAS - CHAVE: Fotocatálise, Fosfato de Prata, Ag_3PO_4/SBA-15, Corante rodamina B, WLEDs.

1 I INTRODUCTION

The growth of demographic and industrial indexes has been causing negative contributions to human health due to the increase of air, soil, and water pollution, being one of the current environmental concerns (MELO 2009; JUMA et al. 2014; INYINBOR et al. 2018). Among the various types of pollutants targeted to nature, stand out the dyes and pharmaceuticals, toxic and stable compounds, which even in small concentrations are quite harmful to living beings, can cause endocrine changes and cancer (CERVANTES et al. 2009; RODRIGUES-SILVA et al. 2014; SHARMA et al. 2017).

Dyes are a group of organic substances synthesized by the man or obtained through of natural routes that have proven to cause significant adverse environmental effects, enabling the weakening or even destruction of aquatic biomes (ALCÂNTARA and DALTIN 1996; GÜRSES et al. 2016). The contamination of these resources by the incorrect discard of the effluent can generate imbalance to the ecosystem, because its action causes a deficiency in oxygenation capacity and the process of photosynthesis, by impediment the penetration of light (BHATNAGAR and JAIN 2005; DALPONTE et al. 2016; SHARMA and BHATTACHARYA 2017).

Thus, these polluting industrial wastes are classified according to their chemical structures. The most frequently used, for example, are synthetic azo dyes that have high toxicity, as they can give rise to amines that are as or more toxic than the compounds that gave rise to them, when discarded in nature without previous treatments (ALCÂNTARA and DALTIN 1996; GUARATINI et al. 2000; AJMAL et al. 2014).

Among this class of pollutants, stands out the Rhodamine B (RhB), which belongs to the family of xanthene and is highly soluble in water. In addition, RhB is directed to the dyeing of cotton and paper and also used in laboratories (dyeing of biological cells) and in industry for traces of water bodies (BENKHAYA et al. 2017; CHEN 2007; HEITZ 1982; JAIN et al. 2007; RICHARDSON et al. 2004).

The treatment of these effluents by traditional methods, microbiological and incineration does not meet the efficiency required by the legislation, pointing to the need for remediation of this problem, through processes capable of mineralizing these pollutants (MORAIS et al. 2006; AQUINO et al. 2013; TERAN 2014; AHMAD et al. 2015; RAJASULOCHANA and PREETHY 2016). For this, Advanced Oxidation Processes (AOPs) are under development; which are physical-chemical processes that when applied to the treatment of wastewater are capable of producing changes in the chemical structures of contaminants; and thus, AOPs are regarded as a promising solution to this environmental problem (POYATOS et al. 2010; BETHI et al. 2016).

Such processes are based on the production of radicals, in which the hydroxyl species gains relevance for being with high reactivity and small selectivity, which attack most of the organic and inorganic molecules producing species like CO2, H2O, and inorganic salts after a series of intermediate reactions (MIKLOS et al. 2018; ARAÚJO et al. 2016; CUERDA-CORREA et al. 2020; GUO et al. 2020). AOPs are classified into homogeneous or heterogeneous processes. Heterogeneous photocatalysis (HP) differ of homogeneous photocatalysis by the presence of a catalyst semiconductor in the solid state, characterized by the presence of a valence band (VB), conduction band (CB), and the region between the bands called forbidden energy gap (XU et al. 2014; MOREIRA et al. 2017; ANJALI and

SHANTHAKUMAR 2019; ORTIZ et al. 2019). The search for these new semiconductors able to be active under visible radiation is still an arduous task, although there are already studies directed to TiO2:N, ZnO e Fe2O3 (FUJISHIMA and HONDA 1972; REHMAN et al. 2009; CANDAL and CRUZ 2014).

In view of this investigation, in 2010 was uncovered a new catalyst semiconductor, the silver phosphate (Ag3PO4), have high photo-oxidative capabilities for water splitting and dye degradation under visible light irradiation due to its absorption near ≤ 530 nm and bandgap energy (EBG) ranging around 2.36 eV (indirect) to 2.43 eV (direct) and with the quantum efficiencies of up to 90% at wavelengths greater than 420 nm (DHANABAL et al. 2015; YI et al. 2010; BI et al. 2011).

Synthesis methods most used for obtaining Ag3PO4 are: co-precipitation and simple ion exchange methods, which use aqueous solutions of AgNO3 or silver amine complexes and phosphate sources (Na3PO4, Na2HPO4 and NaH2PO4) (DONG et al. 2016; DHANABAL et al. 2015; WANG et al. 2012; AMORNPITOKSUK et al. 2013; HUANGA et al. 2015). It is worth mentioning that different temperatures of syntheses or thermal/hydrothermal treatments can be used to obtain this material and that these synthesis methods are used as an initial step for the formation of composites and heterostructures based on the addition of Ag3PO4 to the surface of supports (CRUZ-FILHO et al. 2019; COSTA et al. 2018; KRUNGCHANUCHAT et al. 2017). In particular, these parameters can result in Ag3PO4 of different morphologies, ranging from irregular to spherical, cubic, tetrahedral and dodecahedral (FILHO et al. 2019; WANG et al. 2012; COSTA et al. 2018; KRUNGCHANUCHAT et al. 2017; Al KAUSOR et al. 2019).

Ag_3PO_4 is usually a yellow material with low water solubility (0.02 g/L at 25 °C), considered ideal for processes for removal of organic contaminants since it is nontoxic compound. This one still has a body-centered cubic structure composed of [PO_4] tetrahedral with 6 pairs of deformed [AgO4] tetrahedral at each cubic face. (BAĎUROVÁ et al. 2017; LEBOGANG et al. 2019). However, Ag3PO4 suffers reduction reactions when irradiated, because photo induced electrons in the conduction band (CB) can combine with Ag+, promoting the formation of metallic silver (Ag^0) on the surface of the catalyst; that, depending on the proportion may favor or disadvantage its application in photocatalytic processes (YAO et al. 2012; SHARMA et al. 2015; GE and LI 2017; LIU et al. 2019).

Based on this negative characteristic, photocatalytic tests are being developed with the use of a system with radiation under white light-emitting diodes (WLEDs), which provides the decrease of the photoreduction effect, thus increasing the lifetime of the material (SVOBODA et al. 2018; CRUZ-FILHO et al. 2019). These systems are known as photoreactors and require an efficient and durable light source. LEDs correspond with these characteristics, as they have high energy efficiency and versatility in making the project more flexible concerning to intensity and wavelength (JO and TAYADE 2014; CASADO et al. 2017).

In this context, other studies on the optimization and stability of Ag_3PO_4 show that its distribution on molecular sieve Santa Barbara Amorphous-15 (SBA-15) contributes to high photocatalytic activity, besides its Ag/Ag3PO4 stability (MA et al. 2014; CHAI et al. 2015; COSTA et al. 2018); ever since the support favors the separation of the electron pair/hole (e-/h+) and provides a better distribution of the active sites, because silver phosphate nanoparticles (Ag_3PO_4 NPs) have a strong tendency to agglomerate (ZHAO 1998; AMORNPITOKSUK et al. 2013; LEBOGANG et al. 2019; YUANA et al. 2020).

Therefore, this paper aims to optimize the photocatalytic properties of Ag_3PO_4 distributed on the external surface of the mesoporous molecular sieve SBA-15, applied in the degradation of the RhB dye under white light radiation from emitting (WLEDs).

2 I MATERIALS AND METHODS

2.1 Materials and Reagents

Silver nitrate (AgNO3, Aldrich, purity ≥ 99%); sodium hydrogen phosphate (Na_2HPO_4, Aldrich, 99% purity); tetraethyl orthosilicate (TEOS, Aldrich, purity ≥ 98%); triblock copolymer pluronic ($EO_{20}PO_{70}EO_{20}$; EO: ethylene oxide, PO: propylene oxide,) (P123, Aldrich); hydrochloric acid (HCl, Vetec, purity ≥ 37%); ethyl alcohol (DinâmicaÆ, purity ≥ 95%); deionized water; and Rhodamine B (C28H31ClN2O3) (Vetec, purity ≥ 95%) were used in the study.

2.2 Synthesis of Ag_3PO_4 Nanoparticles

The silver phosphate nanoparticles were synthesized by the simple precipitation method, using 1.0 mL of 0.15 mol L^{-1} Na_2HPO_4 aqueous solution and 3.0 mL 0.15 mol L^{-1} $AgNO_3$ aqueous solution. The two volumes were diluted together in 500 mL of deionized water (H_2O-DI) and this reaction mixture was left to rest by 72 h in the dark with the temperature varying gradually from 4 °C to about 20 °C. Then, excess supernatant was removed and a yellow solid obtained was placed in a porcelain capsule and dried at 65 °C for 6 h. The dry powder was collected and then stored in a dark container.

2.3 Synthesis of SBA-15

The SBA-15 was prepared using the hydrothermal method proposed by ZHAO (1998), in which TEOS, P-123, HCl and H_2O-DI were mixed at the molar ratio 1.000: 0.015: 2.750: 166.0. The quantities of TEOS, P-123, HCl and deionized water required to prepare 100 g of gel were determined based on this molar ratio. Initially, P-123 was dissolved in a solution of H_2O-DI and HCl under magnetic agitation for 2 h at 40 °C. Then, TEOS was added to the obtained system and the stirring was continued for more 24 h at 40 °C.

Thereafter, the sample was transferred to a Teflon™ flask, which was placed in a stainless-steel autoclave and which was heated at 100 °C and held for 48 h. After this period, the sample was cooled and the product obtained was vacuum filtered, washed five times with ethyl alcohol and dried in an oven at 100 °C for 24 h. The next step was to remove the surfactant, and for that, the sample was calcined at 550 °C for 4 h under air flow at 100.0 mL min^{-1} to unblock the SBA-15 pores.

2.4 Impregnation of Ag_3PO_4 crystals on SBA-15

x-Ag_3PO_4/SBA-15 (x = 5, 10, 20% of Ag_3PO_4) composites with different loading ratios were prepared by wet impregnation using the post-synthesis method. Initially, both Ag_3PO_4 and SBA-15 materials were weighed to prepare 0.200 g of the supported catalyst (Ag_3PO_4/SBA-15) taking into account the above-mentioned proportions. In these cases, the SBA-15 was spilled into 20.00 mL of deionized water and sonicated for 15 min.

The Ag_3PO_4 was then added and the resulting suspension was maintained for 20 min on the ultrasonic bath. After that, the system was stirred magnetically at 100 °C until total water evaporation. Then, the heat treatment of the samples was carried out in a muffle furnace with a heating rate of 10 °C/min at 200 °C for 6 h.

2.5 Characterization of Catalytic Solids

The Raman spectra were obtained using a Bruker model SENTERRA spectrometer equipped with an Olympus BX50 microscope with an automatic resolution of 3 cm^{-1} in the 200-1200 cm^{-1} spectral region, a wavelength of 532 nm, and output power of 5 mW. Fourier Transform Infrared (FTIR) spectra were obtained in the 400-4000 cm^{-1} range, applying the dilution method to potassium bromide (KBr) pellets and using Shimadzu IR Prestige-21 spectrometer with a resolution of 4 cm^{-1}. The optical analysis was performed using a model UV-2600 (Shimadzu, Japan) spectrophotometer in the wavelength range of 200 to 800 nm programmed to diffuse reflectance spectra. E_{BG} values were estimated by applying the Kubelka-Munk functions (LIU et al. 2011).

2.6 Photocatalytic Tests

The photocatalytic performance of Ag_3PO_4, Ag_3PO_4/SBA-15 (5%), Ag_3PO_4/SBA-15 (10%), and Ag_3PO_4/SBA-15 (20%) solids was evaluated individually for the photodegradation of 10^{-5} mol L^{-1} RhB dye aqueous solution. 25.0 mg of the photocatalyst was dispersed in 25.0 mL of the dye solution by ultrasonic bath and kept under to magnetic stirring at 32 °C in a glass cell positioned at a distance of 6 cm from the irradiation source.

The photocatalytic system is constituted by a visible light emitting diodes (WLEDs) reactor with a luminous flux (Φ v) of 1.3 x 10^3 lm and relative power density of 100 mW m^{-2}, similar to that used in the research of CRUZ-FILHO et al. (2019). Before irradiation, the suspension was kept for 30 min in the dark under magnetic stirring to establish the adsorption/desorption equilibrium of the RhB dye solution on the surface of the catalyst. To accompany the degradation of the pollutant, 2.0 mL aliquots were collected and centrifuged at 6000 rpm for 10 min. Then, the supernatant was analyzed by UV-vis spectroscopy

(Shimadzu, UV-2600 model). The degradation rate of the RhB dye was calculated using the following equation 1:

$$Degradation\,rate(\%) = |C_0| - \frac{|C|}{|C_O| \times 100\%} \qquad (1)$$

where C_0 corresponds to the initial concentration (i.e. initial absorbance of the band for the chromophore groups of the RhB dye, in λ_{max}: 554 nm) and C corresponds to the concentration measured in the time interval during irradiation.

3 I RESULTS AND DISCUSSION

3.1 RAMAN SPECTROSCOPY AND FTIR

Raman spectroscopy was realized in order to characterize the structure of Ag_3PO_4 and Ag_3PO_4/SBA-15 composites (5, 10, and 20%), through their characteristic vibrational modes, as well as evaluate the degree of crystallinity of these samples according to the intensity of their bands. The Raman spectra of the samples are presented in Fig. 1. It is possible to verify 10 active vibrational modes characteristic of the phosphate group $[PO_4]$. It is worth emphasizing that the Ag_3PO_4 crystals of the $P\overline{43}n$ space group have 18 active vibrational modes according to the equation 2:

$$\Gamma = 2A_1 + 4E + 12T_2 \qquad (2)$$

However, some of these modes are difficult to identify due to their low intensities and even overlapping bands (BOTELHO et al. 2015).

The intense and wide bands located at 100, 115, 182, and 238 cm^{-1} are attributed to the rotation and translation external modes of the $[PO_4]$ units, and the broad bands at 408, 415 and 553 cm^{-1} refer to the symmetric and asymmetric bending of the $[PO_4]$ cluster.

A discrete band was also found in the region of 715 and 724 cm^{-1} corresponding to the symmetrical stretching of the bonds (P-O-P). In the region of 817 cm^{-1} is located a band attributed to the symmetrical stretching of the terminal oxygen of the $[PO_4]$ clusters. Finally, the bands located at 910, 984, 1039, and 1068 cm^{-1} are related to the asymmetric stretching of the $[PO_4]$ cluster (BOTELHO et al. 2015; CHAI et al. 2014; PRESTON and ADAMS 1979).

On the other hand, infrared spectroscopy was used to evaluate the chemical composition of the samples and their degree of purity. The infrared spectra of the samples are shown in Fig. 2. The bands in the 444, 787, and 1070 cm^{-1} regions are related to the asymmetric stretching of the bonds (Si-O-Si), in the amorphous phase spectra (MA et al. 2014).

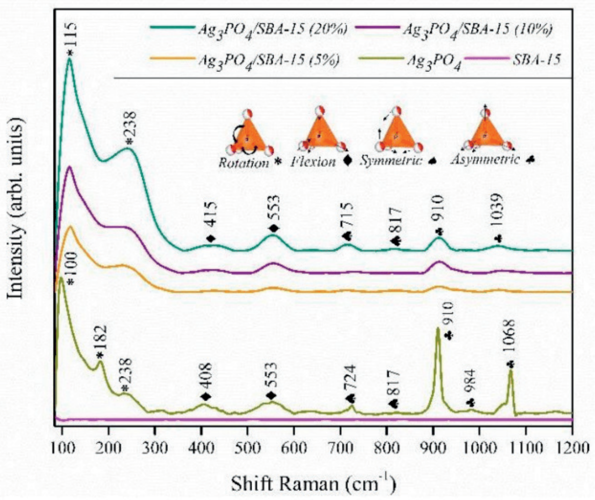

Figure 1: Raman Spectra of Ag_3PO_4/SBA-15 (5%), Ag_3PO_4/SBA-15 (10%), Ag_3PO_4/SBA-15 (20%), and pure Ag_3PO_4 and SBA-15 samples.

The spectra related to Ag_3PO_4 crystals presented vibrational modes characteristic of the material, the bands near 540, 846, and 1365 cm^{-1} can be attributed the vibrations related to the angular flexion of the bonds (O=P-O), symmetrical stretching vibrations of the bonds (P-O-P) and double stretching bond (P=O), respectively (Ma et al. 2015; Wang et al. 2017). A band was also observed at 1010 cm^{-1} and may be correlated to the asymmetric stretching of the $[PO_4]$ cluster (LIANG et al. 2012).

In addition, the spectra of the Ag_3PO_4/SBA-15 (5%, 10%, and 20%) composites show characteristic bands of both the amorphous phase of SBA-15 and the crystalline phase of Ag_3PO_4. However, it is suggested that the band near at 1010 cm^{-1} may have been overlapped by the band at 1070 cm^{-1} of the SBA-15, suggesting an interaction between the two materials (COSTA et al. 2018).

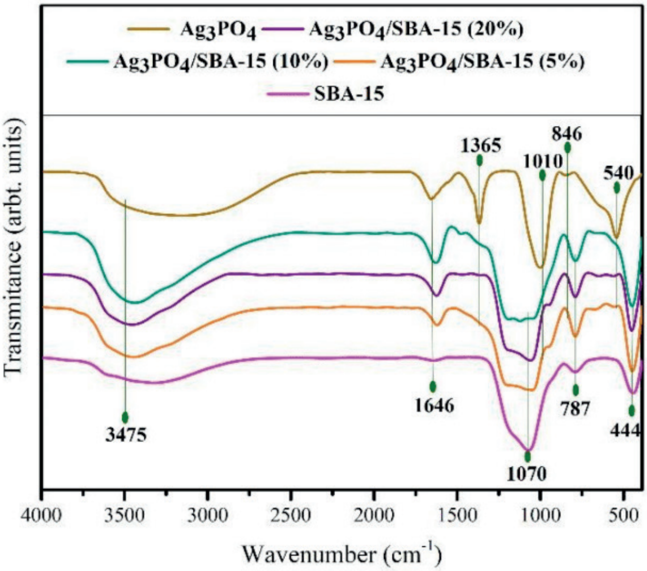

Figure 2: FTIR spectra of the Ag_3PO_4, SBA-15 and Ag_3PO_4/SBA-15 (5%, 10% and 20%) samples.

Finally, it can be confirmed that the Raman and infrared spectra are shown in Fig. 1 and 2 are attributed to the following materials: molecular sieve SBA-15, Ag_3PO_4/SBA-15 (5, 10, and 20%) and pure crystals of Ag_3PO_4, since all the bands detected are related to the vibration modes of these materials. In addition, no secondary band attributed to any other type of material was observed, suggesting that the samples have a high degree of purity.

3.2 UV–VIS Diffuse Reflectance Spectroscopy

The origin of the electronic and photocatalytic properties of Ag_3PO_4 and Ag_3PO_4/SBA-15 composites were investigated using the technique of diffuse reflectance spectroscopy in the UV-vis region. The results obtained indicate their all crystals absorb light in a wavelength of less than 540 nm, allowing the absorption of energy in the visible region (YI et al. 2010; BI et al. 2011; CHEN et al. 2015).

The indirect E_{BG} values were calculated using the Kubelka-Munk function (BESANÇON et al. 2016; INDRA et al. 2014), as shown in Fig. 3 (A-D). The E_{BG} value for pure Ag_3PO_4 was close to 2.40 eV, while the Ag_3PO_4/SBA-15 (5%, 10%, and 20%) composites presented reduced values, being 2.33, 2.34, and 2.39 eV for the samples, respectively.

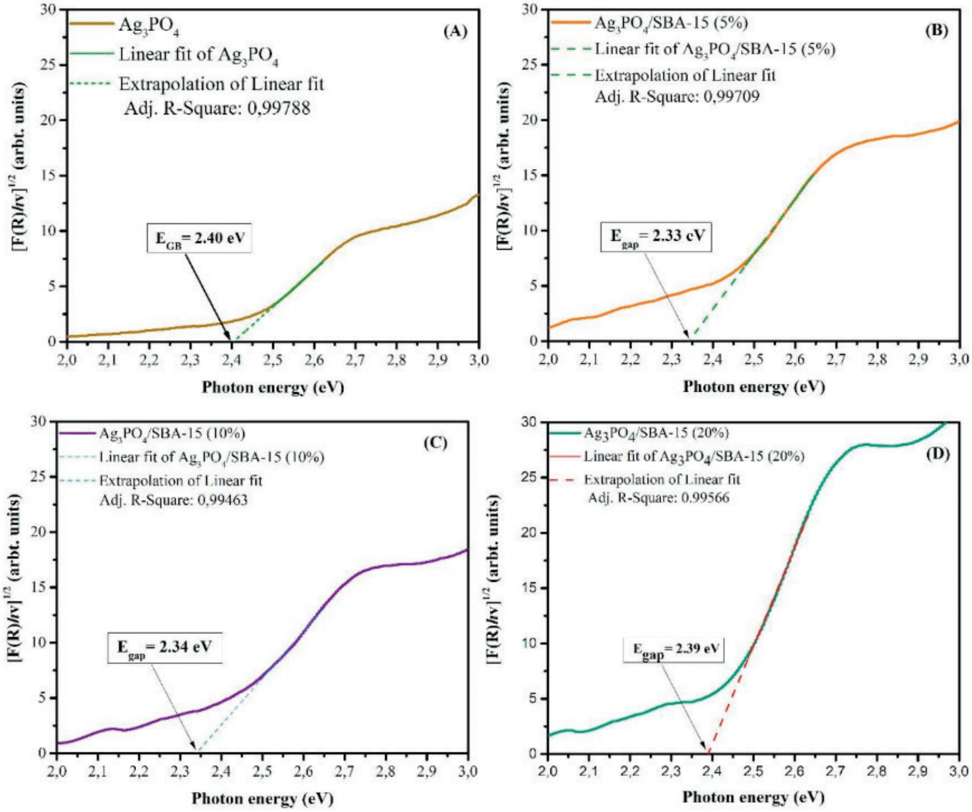

Figure 3: Indirect band-gap energy of Ag_3PO_4 (A) and Ag_3PO_4/SBA-15 (5%, 10%, and 20%) samples (B-D).

It should be noted, however, that the reduced absorption intensity was proportional to the decrease in the content of Ag_3PO_4 used for impregnation, which may suggest that light absorbing ability in the visible region may be related to the Ag_3PO_4 crystals anchored on SBA-15 support (COSTA et al. 2018).

3.3 Photocatalytic Activity

The photocatalytic properties of the pure Ag_3PO_4 and Ag_3PO_4/SBA-15 (5%, 10%, and 20%) composites were evaluated for the degradation of RhB ($C_0 = 10^{-5}$ mol L^{-1}) pollutant under artificial irradiation based on WLEDs light.

These light sources have specific wavelengths of the visible spectral region, corroborating with the energy absorption of the material according to the E_{BG} values estimated; thus, enabling photoactivation of these semiconductors on photocatalysis processes. Also is reported in the literature that RhB absorbs energy in wavelength near 553 nm, besides having high chemical stability, suffering no significant degradation when exposed only to irradiation (CHEN 2007; MA et al. 2014).

Despite its resistance to irradiation, the results of the tests using the photocatalysts were very promising in the discoloration of the pollutant, as shown in Fig. 4. These tests are divided into two steps (dark and irradiated), in the first of them, the pollutant solution, containing the catalyst, is subject only to magnetic agitation in the absence of light for 30 min to establish the adsorption/desorption equilibrium between the materials (MA et al. 2015; GE 2014).

The sample of pure Ag_3PO_4 shows low adsorption rates in relation to the samples impregnated on the SBA-15 in absence of light, as shown in Fig. 4, since the support has a porous structure with a large surface area, an ideal characteristic for this effect to happen (SOUSA et al. 2018).

It is also possible to note that the adsorption values were decreasing with the increase of Ag_3PO_4 content, this may be related to the blockage of pores (an effect caused by the impregnation mechanism used in the synthesis and which is already widely reported in the literature). In view that heterogeneous photocatalytic degradation generally occurs after the adsorbed of the pollutant on the surface of the catalyst, that first step is actually a pre-step for the consequent photocatalytic reaction (MATTHEWS 1988).

The second step is in the decomposition of the pollutant, which occurs after the solution is irradiated, during the period of 80 min test. This step is also shown in Fig. 4 for both the first photocatalytic test and three recycling tests for each material. The results for first use presented degradation rates of 97.63% for Ag_3PO_4, as well as 83.81, 89.79, and 96.98% for Ag_3PO_4/SBA-15 samples in the proportions of 5, 10, and 20% respectively.

It is important to emphasize that after the first catalytic tests all materials were recovered simply. Initially, the solid located at the bottom of the Eppendorf microtubes used in the centrifugation process was collected, and finally, they were oven dried at 50 °C for 3 hours.

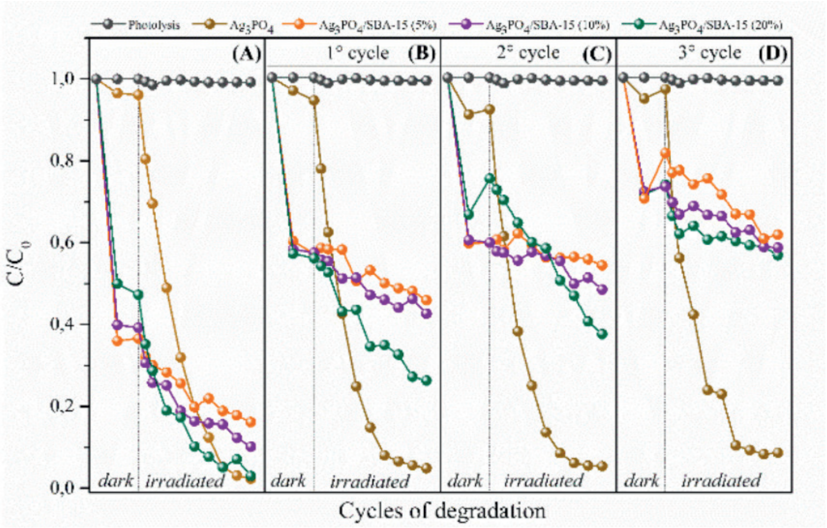

Figure 4: Photocatalytic degradation of the rhodamine B solution using pure Ag_3PO_4 and Ag_3PO_4/SBA-15 (5%, 10%, and 20%) composites under light irradiation of WLEDs (A) and three cycles of reuse (B-D).

Although Fig. 4 expresses the degradation graphs of all samples, Fig. 5 depicts the results of each test more clearly and percentage (%). The degradation rates of the photocatalytic activities of the materials in the 1°, 2° and 3° catalytic cycle of reuse were respectively: (98.87, 98.34 and 94.92%) for pure Ag_3PO_4 sample, (63.79, 47.54 and 39.76%) for the Ag_3PO_4/SBA-15 (5%) sample, (59.72, 53.62 and 43.02%) for the Ag_3PO_4/SBA-15 (10%) sample and (76.68, 64.92 and 45,00%) for the Ag_3PO_4/SBA-15 (20%) sample under the same irradiation time.

This excellent photocatalytic performance of the pure Ag_3PO_4 sample is shown in Fig. 5 as well as their reuse, may have been occasioned by the source of light originating from the WLEDs (since some studies report the instability of this material whenever exposed to polychromatic light sources or solar simulation), the great dispersion of active sites and the positive nature of the dye that causes greater interaction with the photocatalyst (SHARMA et al. 2015; MENEZES et al. 2016; CRUZ-FILHO et al. 2019; HUANGA et al. 2015).

However, the performance of the samples impregnated in SBA-15 differed from what was proposed for this study, since the molecular sieve has the function of maintaining the stable activity of the Ag_3PO_4/SBA-15 during its reuse and the graph of Fig. 4 exhibits a very large decay of activity in the last photocatalytic cycles.

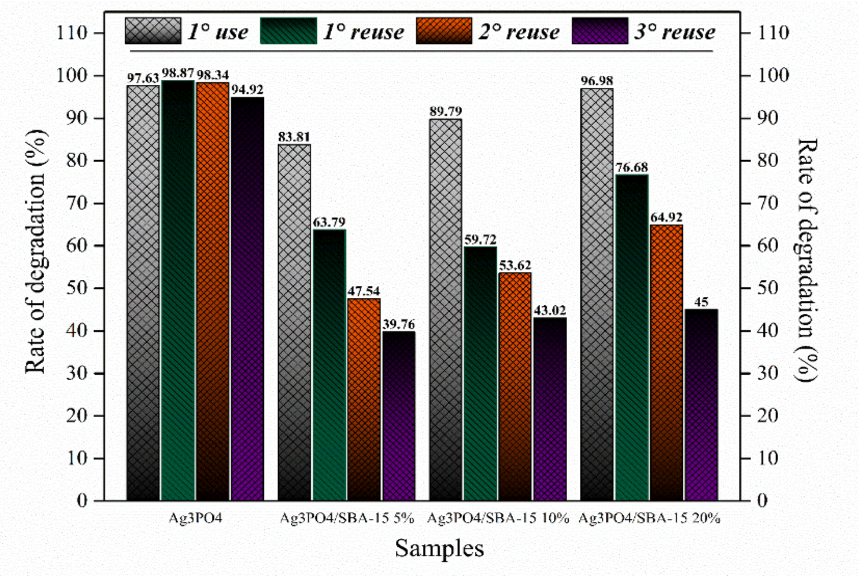

Figure 5: Photocatalytic performance of Ag_3PO_4 and Ag_3PO_4/SBA-15 (5%, 10%, and 20%) samples in degradation of dye Rhodamine B and three cycles of reuse.

The decrease in percentages of degradation is considered one of the main problems found for heterogeneous photocatalysis, since it is caused by the deactivation of the catalyst. Many mechanisms seek to explain the deactivation of these materials, but for this work it is suggested that Ag_3PO_4/SBA-15 is under the influence of chemical inactivation by photocatalyst poisoning and leaching due to the unstable nature of Ag_3PO_4 and the synthesis pathway of Ag_3PO_4/SBA-15, although no analysis has been carried out to confirm these suspicions (WANG et al. 2012; CHEN et al. 2015; ARGYLE and BARTHOLOMEW 2015; ZHANG and WANG 2017).

However, a relevant aspect of this material is the fact that the samples of Ag_3PO_4/SBA-15 are constituted only by a small amount of Ag_3PO_4 and even with these decreases in degradation rates, the impregnated materials proved to be efficient in the degradation of the RhB dye, since the values obtained for the last cycles are almost than 50% of the values obtained in the first cycles. From the same point of view, it should be emphasized that the SBA-15-impregnated materials have only 5, 10 and 20% by weight of Ag_3PO_4 in its composition, whereas the pure sample has 100% mass of Ag_3PO_4, that is, there is an economy in the use of Ag_3PO_4.

4 I CONCLUSION

This work consisted of the synthesis, characterization and application of Ag_3PO_4 crystals and Ag_3PO_4/SBA-15 nanocomposites (5, 10 and 20%) for the photodegradation of the aqueous solution of RhB. Structural investigation was realized by Raman and infrared spectroscopy techniques revealed that pure Ag_3PO_4 crystals have very marked active modes that are indexed to a very crystalline Ag_3PO_4 () structure. Ag_3PO_4/SBA-15 composites (%) also presented these characteristic active modes, however, vibration modes of the amorphous phase of the SBA-15 molecular sieve were also observed in infrared spectra. UV-vis spectroscopy showed that the sample of pure Ag_3PO_4 absorbed light of greater wavelength in the visible region than Ag_3PO_4/SBA-15 (X%) samples, allowing E_{BG} values of 2.40 eV for pure Ag_3PO_4 and 2.33, 2.34 and 2.39 eV for Ag_3PO_4/SBA-15 samples (5%, 10%, and 20%), respectively. The photocatalytic tests developed with samples of pure Ag_3PO_4 and Ag_3PO_4/SBA-15 (X%) under the influence of light of WELDs, showed high efficiency in pollutant decomposition the RhB. The rates of degradation were from 97.63, 83.81, 89.79 and 96.98% for the pure Ag_3PO_4, Ag_3PO_4/SBA-15 in 5, 10 and 20%, respectively, thus allowing the reuse of these catalysts.

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