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Preparation advances of Activated-Carbon/ZnO composite using ground coffee

Avances en la preparación del compósito Carbón Activado/ZnO a partir de café molido usado

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Abstract

In present work, synthesis and characterization of ZnO/ activated carbon-based composite in proposed. Spent Coffee Grounds is used as carbon source. We pretend to take advantage of photocatalytic activity of ZnO and adsorption capacity of Activated Carbon. This composite can be applied in remotion/mineralization of organic dyes in waste water. Conditions for synthesis of composite's precursor were stablished. For electrosynthesis, the current density was 260 mA/cm^2 for 15 min under vigorous magnetic stirring at room temperature, followed by a calcination at 450 °C. Later, all materials were characterized using Raman Spectroscopy, Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS) and Fourier-Trasform Infrared Spectroscopy (FTIR), in order to determine crystalline phase present, morphology, elemental composition and functional groups, respectively.

Composite, SCG, CA/ZnO

Resumen

En el presente trabajo, se propone la síntesis y caracterización un compósito de Óxido de Zinc (ZnO) y carbón activado (CA) que se obtiene a partir del café molido usado (CMU), para aprovechar la capacidad fotocatalítica del ZnO y la capacidad de adsorción del CA, este compósito podrá aplicarse en la remoción/mineralización de colorantes en aguas residuales. La metodología que se siguió para la preparación del compósito fue establecer las condiciones de síntesis por medio de las síntesis de los precursores del compósito; las condiciones que se establecieron fueron realizar una electrosíntesis a 260 mA/cm^2 durante 15 min con agitación magnética vigorosa a temperatura ambiente y calcinación a 450 °C; posteriormente se realizó la caracterización por medio de Espectroscopía Raman, Microscopia Electrónica de Barrido (MEB), Espectroscopia de Dispersión de Energía de Rayos X (EDS) y Espectroscopia de Infrarrojo (FTIR) para determinar las fases cristalinas presentes, la morfología, la composición elemental y los grupos funcionales, respectivamente.

Compósito, CMU, CA/ZnO

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Introdution

Nowadays, metallic oxide-based semiconductor photocatalyst such as SiO_2, Al_2O_3, CuO, FeO, V_2O_5, TiO_2, ZrO_2, ZnO has become in an interesting option due to its potential contribution to environmental remediation. Among them, TiO_2 and ZnO has been studied and used due to its photocatalytic properties (Palanive, N.R., & Selvakumar, 2019) (Saravanakkumar, y otros, 2019) (Macwan, Dave, & Chaturvedi, 2011).

Heterogenous photocatalysis is a method used for waste-water treatment, it employs low-cost and efficient materials for pollutants remotion.

Particularly, ZnO is one of those materials that can be used for heterogeneous photocatalysis because it is inexpensive and easy to obtain (Lee, Lai, Ngai, & Juan, 2016) (Janotti & Walle, 2009) (Abdessemed, Rasalingam, Abdessemed, Djebbar, & Koodali).

ZnO belongs to the group II-VI semiconductors compound and, like many of them, crystallizes in cubic zinc blende and hexagonal wurzite, where the last one is are the most stable form in standard conditions of pressure and temperature.

ZnO is a type-n semiconductor material that possess a wide bandgap of 3.3 eV and an exciton binding energy of 60 meV. Bangap value for ZnO permits activation using UV light for photodegradation of organic pollutants (Abed, Bouzidi, Elhouichet, Gelloz, & Ferid, 2015) (Daneshvar, Salari, & Khataee, 2004).

Activated carbonous material or Activated Carbon (AC) is a product that possess a reticular crystalline structure similar to that of graphite, a great surface area, an abundant quantity of pores by volume unit. For these reasons, activated carbon is a material well stablished, versatile and widely used as adsorbent (Abdessemed, Rasalingam, Abdessemed, Djebbar, & Koodali) (Guo & Rockstraw, Activated Carbons prepared from rice hull by one-step phosphoric acid activation, 2007).

It is important to highlight that commercial AC are still expensive, especially in developing countries, due to the need to process raw material, by chemical or physical means. For the above reasons, interest in obtaining AC from renewable sources and in a more economical way has grown recently. For many years, researchers have demonstrated that is possible to produce Activated Carbons using industrial and agricultural by-products such as waste paper, used tires, rice hull, coconut hull, apricot and date pits. Using ACs for pollutants adsorption in waste water are well documented (Abdessemed, Rasalingam, Abdessemed, Djebbar, & Koodali) (Belhachemi, Rios, Addoun, & Silvestre-Albero, 2009) (Okada, Yamamoto, Kameshima, & Yasumori, 2003) (Tan, Ahmad, & Hameed, 2008) (Baccar, Bouzid, Fekib, & Montiel, 2009).

Coffee is a drink which has been enjoyed by millions of people around the world and many studies has demonstrated all its benefits. However, this drink has a high waste production: Spent Coffee Ground (SCG) (Cruz-Lopes, Domingos, Ferreira, & Esteves, 2017).

Spent Coffee Grounds contains a great quantity of organic compound (fatty acids, lignin, cellulose, hemicellulose and another polysaccharides). Due to its chemical richness and its biodegradability, SCG can be used in biodiesel production, sugar sources, composting material, ceramics additive, heavy ions and dyes adsorbent, and additionally as an activated carbon source (Campos-Vega, Loarca-Piña, Vergara-Castañeda, & Oomah, 2015) (Arulrajah, Kua, Suksiripattanapong, Horpibulsuk, & Shen, 2017) (Colantoni, y otros, 2021) (Oliveira, Silva, Pereira, Filho, & Carvalho, 2013) (Kante, Nieto-Delgado, Rangel-Mendez, & Bandosz, 2012).

As mentioned above, it is possible to give an additional utility to SCG, instead of deposit it in Dumps, so SCG can produce methane and to contribute to greenhouse effect (Arulrajah, Kua, Suksiripattanapong, Horpibulsuk, & Shen, 2017).

In present paper, we propose to combine photocatalytic activity of ZnO and adsorption capacity of AC obtained from SCG, to prepare a composite that can be used for mineralization/remotion of dyes in waste waters.

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Methodology

AC preparation

For obtaining AC, SCG was recollected and dried using a natural convection electric oven for 48 h to evaporate all water. Once SCG is dried, it is ready to be stored. 1g of SCG is taken and thermally analyzed in a hot cell coupled to Raman equipment. The temperature range studied was from 50 to 600 °C using steps of 50 °C for a period of 2 h at each step. Temperature ramps of 50 °C/min were used. With this procedure, 450 °C was the determinate temperature for obtaining AC.

ZnO synthesis

Preparation of ZnO was done using two metallic sheets of Zn with an effective area of 3 cm^2. Previous to the synthesis, Zn sheets were polished and rinsed with anhydrous ethanol. After, they were ultrasonically cleaned using anhydrous ethanol for three cycles. Between each cycle, Zn sheets were rinse with anhydrous ethanol.

Electrosynthesis was carried out in galvanic mode using a current density of 260 mA/cm^2 for 15 min with vigorous magnetic stirring at room temperature. Electrolytic solution contained 0.5 g of sodium chloride dissolved in 50 mL on distilled water. Following this procedure, a white colloidal suspension of Zn $(OH)_2$ is obtained and it is the precursor for ZnO.

Composite synthesis

For composite preparation, an electrolytical cell is installed following procedure described in section 2.2. Only a change is done, 5 g of SCG is added to electrolytical solution previous to electrosynthesis. The electrolyte is magnetically stirred for 5 min to homogenize the solution, and then current is supplied to obtain precursor of ZnO. Colloidal suspension containing SCG/Zn $(OH)_2$ is centrifuged 4 times at 14000 rpm during 5 min and it is rinsed between each cycle with distilled water to remove NaCl remains.

The obtained precipitates are dried at 80 °C in an electric oven. The final product is an amorphous light brown which is thermally treated at 450 °C for 1 h using an electric furnace.

Material characterization

Raman spectroscopy

The structure of powders was studied with Raman spectroscopy. Raman spectra were recorded using a Horiba Jobin Yvon model XploraPlus μ-Raman system. A solid-state diode (λ= 532 nm) is used to induce scattering, with a maximum power of 2.5 mW at sample's surface. A 1200 l/mm was employed; 100 spectra were averaged with an exposure time of 3 s each.

Scanning Electron Microscopy

Morphology and elemental composition of AC/ZnO composite was done using a Jeol IT-100 microscope coupled to a Bruker X-ray microprobe. SEM is operated at high vacuum conditions with an accelerating voltage of 20 kV; secondary electron signal was used to acquire images.

Fourier-Tranformed Infra-Red spectroscopy (FTIR)

In order to determine functional groups present in individual components and in composite, an Perkin-Elmer FT model 2000 equipment in ATR mode, ranging from 4000 a 530 cm^{-1}.

Results

Raman spectroscopy

In figure 1, Raman spectrum corresponding to SCG/$Zn(OH)_2$ composite is shown. It can be seen there is not signals in all spectral window; this is an indicative of an amorphous material.

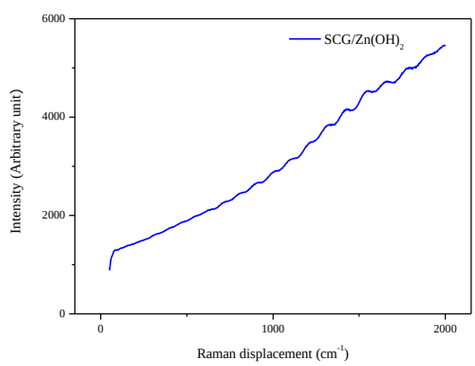

Figure 1 Raman spectrum for as-synthesized composite (SCG/Zn $(OH)_2$)
Source: own elaboration

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After the material is thermally treated (figure 2), Raman signals corresponding to ZnO and AC appears.

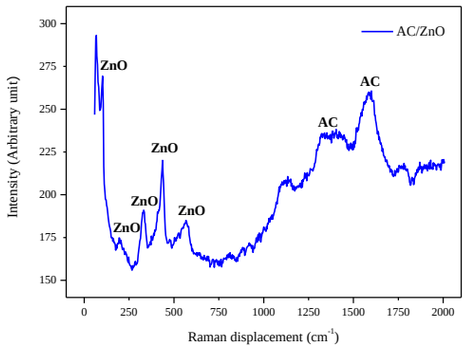

Figure 2 Raman spectrum for composite after thermal treatment (AC/ZnO)
Source: own elaboration

Signal for ZnO are located at 99 (E_{2l}), 203 (E_{2l}), 332 ($3E_{2h}$-E_{2l}), 388 (A_1(TO)), 410 (E_1(TO)), 437 (E_{2h}), ~550 (A_1(LO)) and 588 cm^{-1} (E_1(TO)), corresponding to hexagonal structure wurzite type, This phase has been reported in applications such as photocatalysis and solar cells (Periyat & Ullattil, 2015) (Decremps, Pellicer-Porres, Saitta, Chervin, & Polian, 2002) (Venkatesha, Nayaka, Viswanatha, Vidyasagar, & Chethana, 2012) (Mauro, Fragalà, Privitera, & Impellizzeri, 2017) (Bairamov, Heinrich, Irmer, & Toporov, 1983).

Raman spectrum for all carbon-based materials, that includes AC, shows a diverse behavior in the range of 800-2000 cm, in which G and D bands (or peaks) are located around 1500-1630 cm^{-1} and 1350 cm^{-1} respectively, for visible light excitement. If UV irradiation is used, a T band will appear near at 1060 cm^{-1} (Ferrari & Robertson, Raman Spectroscopy of Amorphous, Nanostructured, Diamond-like and Nanodiamond, 2004) (Ferrari & Robertson, Interpretation of Raman spectra of disordered and amorphous carbon, 2000) (Ferrari A. C., 2002).

In case of our composite after thermal treatment it is possible to observe D band at ~1380 cm^{-1} and G band at ~1590 cm^{-1}, these signals confirm presence of a carbonous material in AC/ZnO composite.

Scanning Electron Microscopy

Several images were taken before (fig. 3a) and after (fig. 3b). In both images, it is possible to observe ZnO particles are adhered to AC's surface.

Through figures 4a to 4b, it can be appreciated with more detail ZnO particles on AC's surface, which in both cases, shows particle agglomerates of submicrometric size.

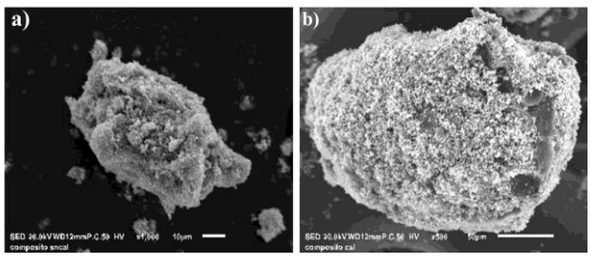

Figure 3 SEM images for a composite a) without thermal treatment (SCG/Zn $(OH)_2$) and b) with thermal treatment (AC/ZnO)
Source: own elaboration

Table 1 reports results obtained by EDS analysis, elemental composition was measured before and after thermal treatment.

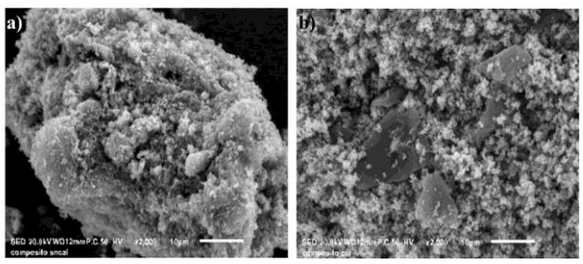

Figure 4 SEM images for a composite at 2000X a) without thermal treatment (SCG/Zn $(OH)_2$) and b) with thermal treatment (AC/ZnO)
Source: own elaboration

In as-synthesized composite, is evident the content of several chemical elements (C, O, Mg, Cl, K, Ca and Zn) belonging to SCG, except for Zn which comes from electrosynthesis. Previous to thermal treatment, the composite is centrifuged to 14000 rpm and rinse with distilled water 5 times, in order to eliminate precursor remains. For this reason, the elemental analysis of composite after treatment, only shows elements of interest (C, O and Zn).

An elemental mapping was performed in order to know elements distribution in our composite. Figure 5 illustrates the elemental mapping for C (yellow) and Zn (purple), it can be seen that Zn is all over the surface of AC particles.

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Element	% at.	
	$SCG/Zn(OH)_2$	AC/ZnO
C	87.93	28.02
O	4.95	33.29
Mg	1.16	----
Cl	0.69	----
K	0.22	----
Ca	0.57	----
Zn	4.48	38.64
Sum	100	100

Table 1 Results for elemental analysis for samples before ($SCG/Zn(OH)_2$) and after (AC/ZnO) thermal treatment
Source: Own elaboration

Figure 5 SEM mapping for C (yellow) an Zn (purple)
Source: Own elaboration

FTIR spectroscopy

Figure 6 shows IR spectra for individual precursors corresponding to composite, Spent Coffee Ground (SCG), Activated Carbon (AC), zinc oxide (ZnO), composite precursor without calcination ($SCG/Zn(OH)_2$) and calcinated composite (AC/ZnO). Figure 6a corresponds to SCG, it shows signal for functional groups of C-H at 2929, 2869, 874 and 815 cm^{-1}; C=O at 1747 cm^{-1}; C=C at 1658 and 1448 cm^{-1} and C-O at 1039 cm^{-1}; all these signals are attributed to SCG (Orozco-Castro, 2019).

For AC (fig. 6b), it can be seen band attributable to aromatic groups such as C=O at 1560 cm^{-1}, C-O at 1039 cm^{-1} and C-H at 874 and 815 cm^{-1}. ZnO precursor is shown in figure 6c, spectrum displays a band for C=O at 1560 cm^{-1} due to CO_2 and it is similar to that obtained by WU et al, 2010 (Dapeng Wu, 2010). for zinc oxide.

In figure 6d, it can be observed spectrum for composite before calcination (precursor) and it shows a binding vibration at 3413 cm^{-1}, this signal is associated with presence of $Zn(OH)_2$ (Dapeng Wu, 2010), which is a ZnO precursor.

Others functional groups can be also observed for C-H at 2929, 2869, 874 and 815 cm^{-1}; C=O at 1747 cm^{-1} C=C at 1658 and 1448 cm^{-1}; and C-O at 1039 cm^{-1}, all these bands are attributable to SCG (Orozco-Castro, 2019).

After calcination, ZnO is obtained supported on AC (fig. 6e), spectrum displays a small signal corresponding to O-H binding and two small signals attributable to aliphatic and aromatic groups of C-H binding, which were also observed in precursor. Due to IR spectroscopy was performed in ATR mode, it is impossible to observe signal located at 440 cm^{-1} corresponding to ZnO (Oo, 2007), some authors use signal located at 530 cm^{-1} to identify it (Ruiz-Peralta, 2012).

IR spectrum for calcinated composite is very similar to that obtained using zinc nitride and hexamine to produce ZnO supported on SCG synthesized by find by Ruiz et al. (Ruiz-Peralta, 2012).

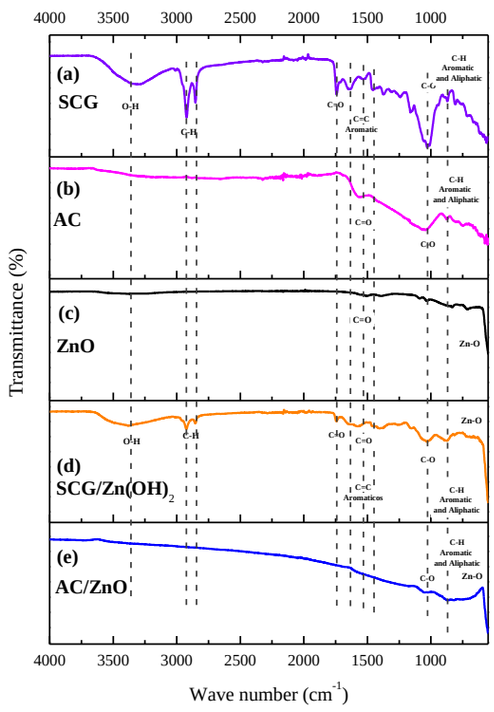

Figure 6 FTIR spectra for (a) SCG, (b) AC, (c) ZnO, (e) $SCG/Zn(OH)_2$ and (d) composite AC/ZnO
Source: Own elaboration

Aknowledgement

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Conclusions

It was possible to synthesized an AC/ZnO composite using electrosynthesis technique followed by a thermal treatment. Raman spectroscopy allowed to determine hexagonal wurzite phase as the only present phase of ZnO.

Using SEM, it was possible to determine composite's morphological characteristics and to observe that ZnO is covering all Activated Carbon's surface. Additionally, EDS confirms that some undesired elements are removed during rinsing and centrifuging process.

Finally, IR spectroscopy from all precursors, uncalcinated and calcinated composite allows to determine characteristic bands for each individual component of composite. This is material was synthesized in order to be used in remotion/mineralization of dyes in waste water.

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