

International Interdisciplinary Congress on Renewable Energies, Industrial Maintenance, Mechatronics and Informatics Booklets



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

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Editorial label ECORFAN: 607-8695 BCIERMMI Control Number: 2021-01 BCIERMMI Classification (2021): 271021-0001

Pages: | | RNA: 03-2010-032610115700-14

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Introduction

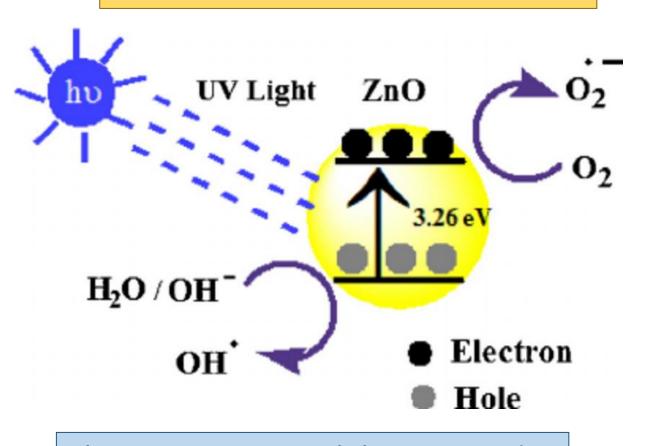
Oxide-based semiconductor photocatalyst

 SiO_2 , Al_2O_3 , CuO, FeO, V_2O_5 , TiO_2 , ZrO_2 , ZnO

Bangap value for ZnO permits activation using UV light for photodegradation of organic pollutants

ZnO (hexagonal wurzite)

Bandgap of 3.3 eV Exciton binding energy of 60 meV.



The **ZnO** is a material that can used in heterogeneous photocatalysis

Introduction

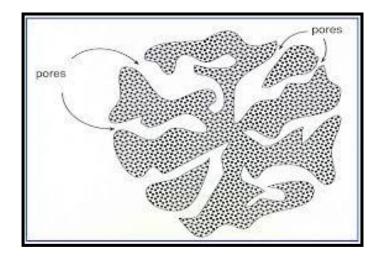
Activated carbonous material or Activated Carbon (AC)

- Crystalline structure similar to that of graphite
- A great surface area
- An abundant quantity of pores by volume unit.

Objective:

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.





Methodology

AC preparation ----

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 analized in a hot cell coupled to Raman equipment.

Temperature ramps of 50 °C/min were used. With this procedure, 450 °C was the determinate temperature for obtaining AC.



The temperature range studied was from 50 to 600 °C using steps of 50 °C for a period of 2 h at each step.

Methodology

ZnO synthesis

It was done using two metallic sheets of Zn with an effective area of 3 cm².

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/cm² for 15 min with vigorous magnetic stirring at room temperature.

Electrolytic solution contained 0.5 g of sodium chloride dissolved in 50 mL od distilled water.

Following this procedure, a white colloidal suspension of $Zn(OH)_2$ is obtained and it is the precursor for ZnO.

Methodology

Composite synthesis



Electrolytical cell is installed following procedure described in ZnO synthesis. 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.

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.



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.



Results

Raman Spectroscopy

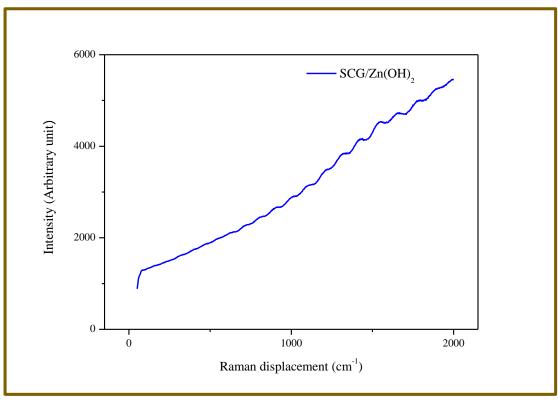


Figure 1. Raman spectrum for as-synthesized composite (SCG/Zn(OH)₂). Source: own elaboration.

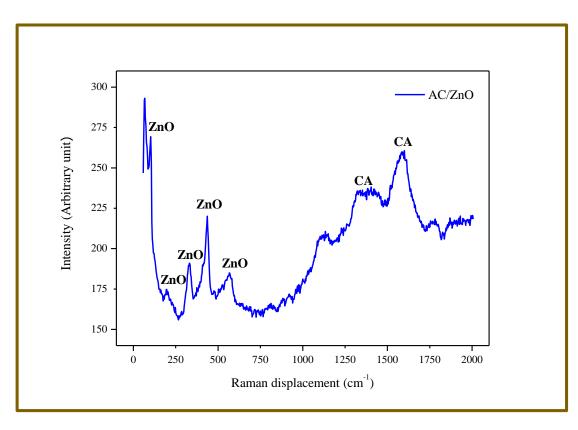


Figure 2. Raman spectrum for composite after thermal treatment (AC/ZnO). Source: own elaboration.

Results

Scanning Electron Microscopy

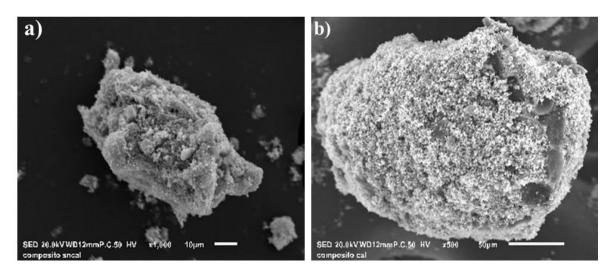


Figura 3. SEM images for a composite a) without thermal treatment $(SCG/Zn(OH)_2)$ and b) with thermal treatment (AC/ZnO) Source: own elaboration.

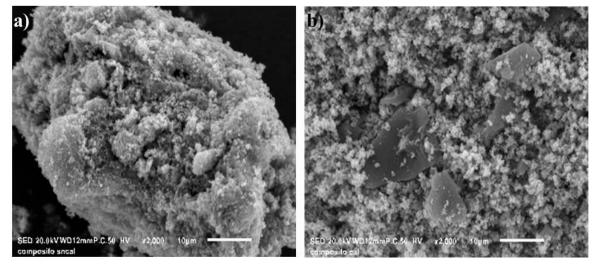


Figura 4. SEM images for a composite at 2000X a) without thermal treatment ($SCG/Zn(OH)_2$) and b) with thermal treatment (AC/ZnO). Source: own elaboration.

Results

Scanning Electron Microscopy

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

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

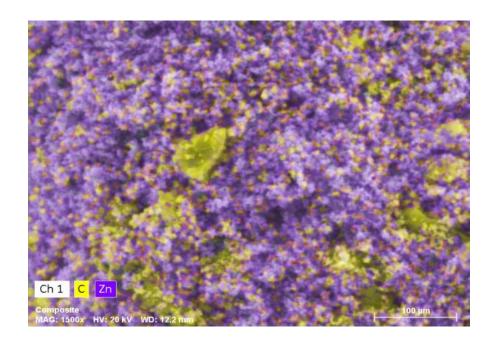


Figure 5. SEM mapping for C(yellow) an Zn (purple). Source: own elaboration.

Results FT-Infrared spectroscopy

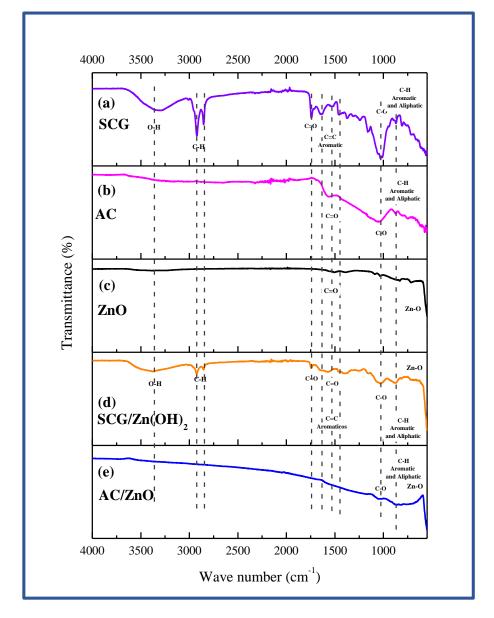


Figura 6. FTIR spectra for (a) SCG, (b) AC, (c) ZnO, (e) SCG/Zn(OH) $_2$ and (d) composite AC/ZnO. Source: own elaboration.

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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