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Title: Advances in the Preparation of hydroxyapatite/ZnO composites from eggshells

Authors: CONTRERAS-DE LA CRUZ, M.A., GARCÍA-GONZÁLEZ, N. ENRÍQUEZ-PÉREZ, Ma. Ángeles and CASTREJÓN SÁNCHEZ, V.H

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ECORFAN-México, S.C.
143 – 50 Itzopan Street
La Florida, Ecatepec Municipality
Mexico State, 55120 Zipcode
Phone: +52 1 55 6159 2296
Skype: ecorfan-mexico.s.c.
E-mail: contacto@ecorfan.org
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Methodology

Reactants

Hydrogen peroxide (90%, Sigma Aldrich), phosphoric acid (85%, Sigma Aldrich), distilled water, anhydrous ethanol, Zinc Acetate and oxalic acid.

Hydroxyapatite Synthesis (HAp)

For Hap synthesis, methodology proposed by Enríquez et al. (Enríquez-Pérez, Ma. Angeles, Castrejón-Sánchez, Víctor Hugo, Rosales-Davalos, Jaime Y Díaz-Camacho Francisco Javier A., 2020), was followed. HAp was prepared using eggshell impregnated with H_3PO_4 and subsequently calcined for 2 h at 800 °C.

Se hizo la recolección de cascaron de huevo (900 g), se lavó con agua corriente y después con peróxido de hidrogeno al 70%, después se secó a 80 ºC por 48 h. El material seco se trituro con un mortero de agata y se pasó por un tamiz #80, posteriormente se impregnó el material con ácido fosfórico 1 M por 5 h, después se hacen lavados con agua destilada hasta un pH neutro. El material se calcina a 800ºC, los tiempos de calcinación se establecieron en 2, 4 y 6 h. El cerámico obtenido se muele en un mortero de agata y se pasó por un tamiz #80.

Sol-gel synthesis of Zinc Oxide

Initially, two solutions were prepared, both solutions contain ethanol; solution A, was heated at 60 °C and solution B; both of them with slow stirring. Once temperature was reached, zinc acetate and oxalic acid was poured into solution A and solution B, respectively. Both solutions were magnetically stirred until they were completely homogeneous. Later, solution A is poured into solution B under continuous magnetic stirring. The

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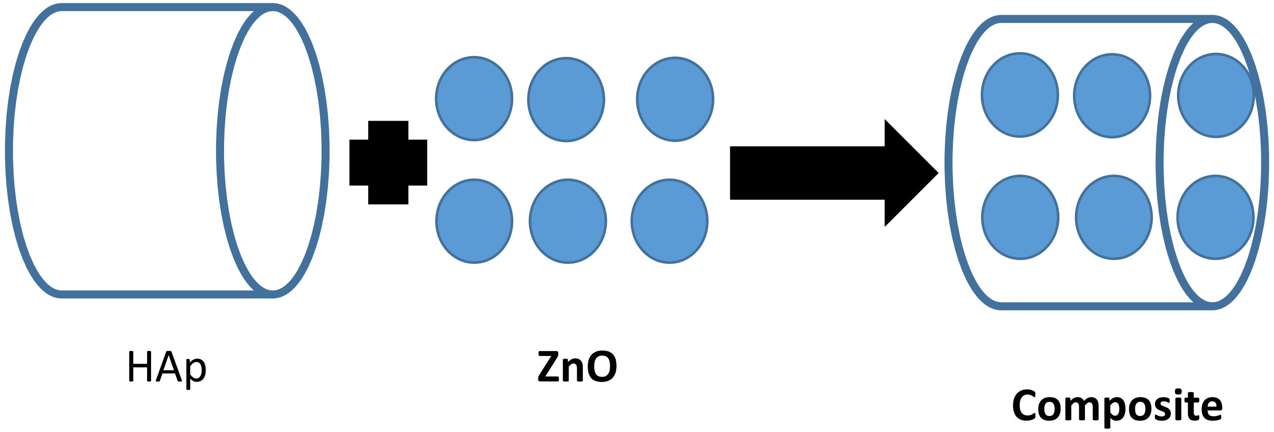
Introduction

In present work, a composite material synthesis is reported using HAp and ZnO

They were obtained reusing eggshells and by sol-gel method, respectively. Later, both components are coupled.

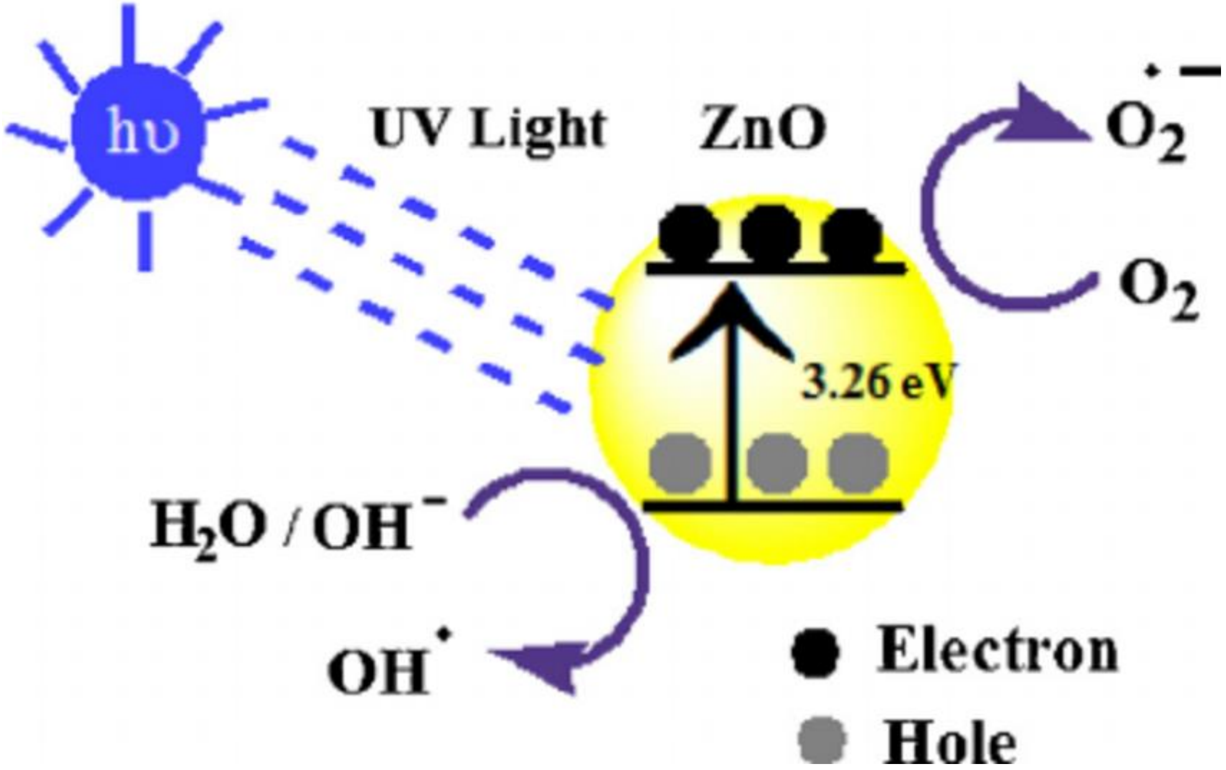
Composite material is tended to be used in organic dyes degradation.



Introduction

ZnO (hexagonal wurzite)
Bandgap of 3.3 eV
Exciton binding energy of 60 meV.

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**

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

Introduction

Hydroxyapatite (HAp) is made up by calcium, phosphor and hydrogen atoms, according to next formula: $Ca_{10}(PO_4)_6(OH)_2$.

HAp belongs to a larger group of compounds known as apatites.

General formula

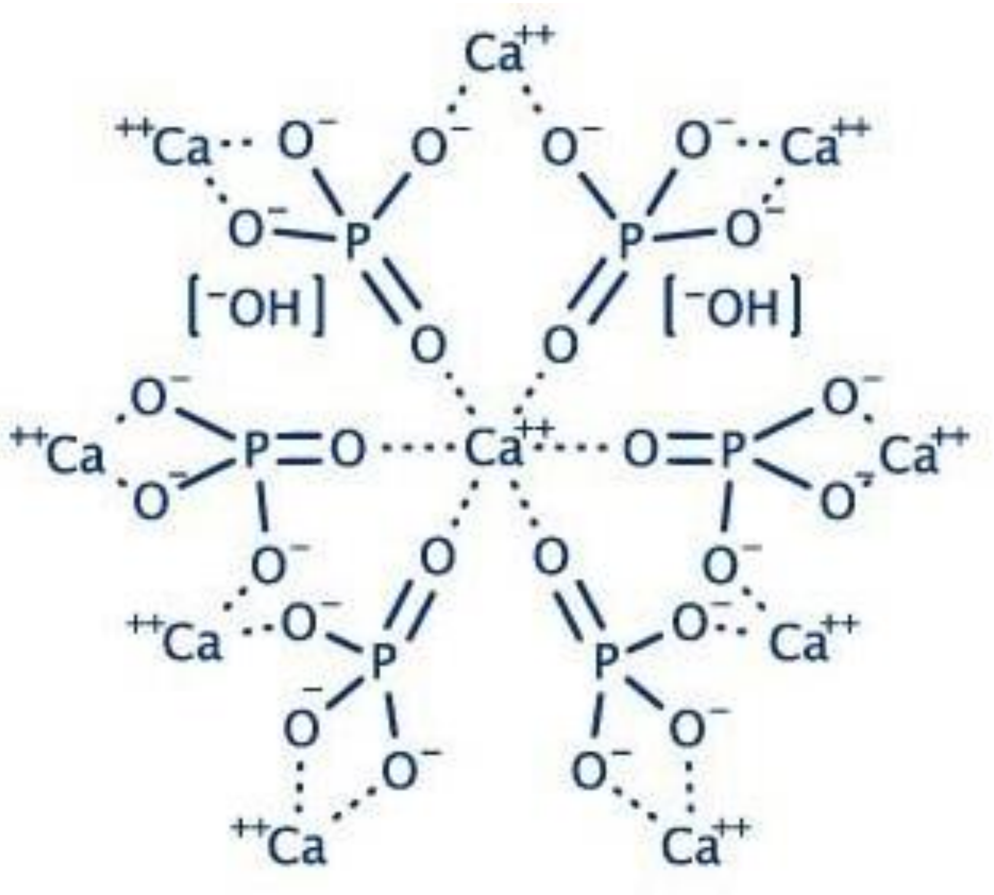
$X_3\ Y_2\ (TO_4)\ Z$

Where:

X o Y= Ca. Sr, Ba, Re, Pb, U, Mn and sometimes Na.

T = P, As, V, Si, S y C (as CO_3).

Z = F, Cl, OH^- y O



Methodology

Reactants

Hydrogen peroxide (90%, Sigma Aldrich), phosphoric acid (85%, Sigma Aldrich), distilled water, anhydrous ethanol, Zinc Acetate and oxalic acid.

Hydroxyapatite Synthesis (HAp)

The eggshell is wash with water and peroxide, after it is dry at 80 °C

The eggshell is impregnated with H_3PO_4 and subsequently calcined for 2 h at 800 °C.

Sol-gel synthesis of Zinc Oxide

Solution A: Ethanol (heat at 60 °C) and admix with zinc acetate (slow stir).

Solution B: Ethanol (heat at 60 °C) and admix oxalic acid (slow stir).

Blend Solution A + Solution B under continuous magnetic stirring.

The solution is aged for 24 h. Later, it is calcined at 650 °C for 30 min.

Methodology

Hydroxyapatite (HAp) / Zinc oxide (ZnO) composite by sol-gel method.

Composite was prepared mixing HAp powders with Zinc under magnetic stirring for 2 h and it was annealed at 650 °C for 30 min.

Results

Raman Spectroscopy

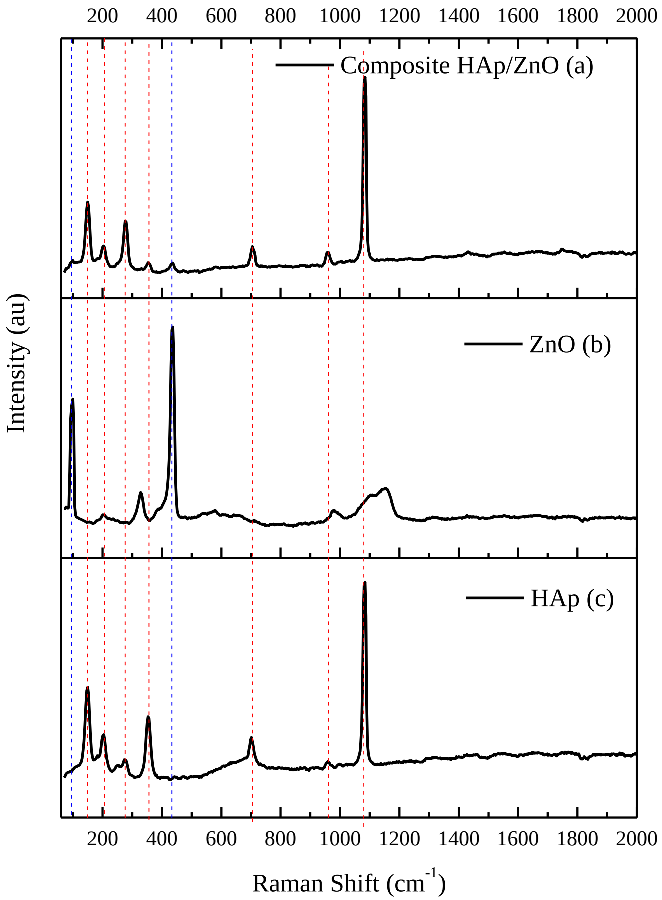


Figure 1 Raman spectra of the composite (a), ZnO (b) and Hydroxyapatite (c) synthesized. *Source: own elaboration*

X-Ray Diffraction (XRD)

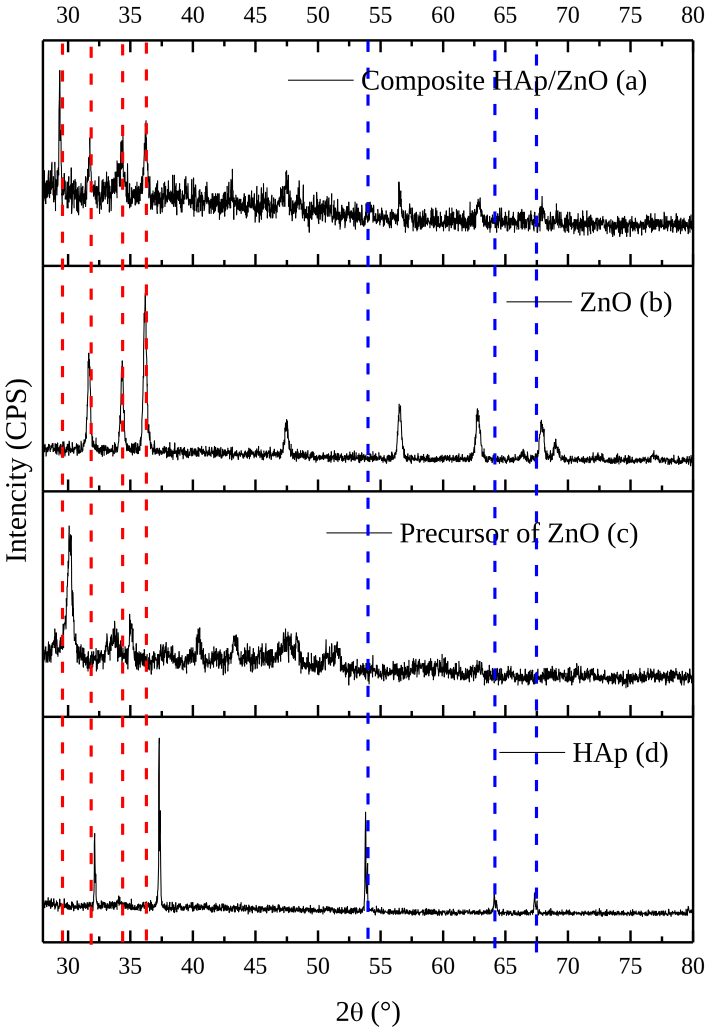


Figure 2. Diffractograms of the composite (a), ZnO (b) ZnO Precursor (c), and synthesized Hydroxyapatite (d). *Source: own elaboration*

Results

Scanning Electron Microscopy

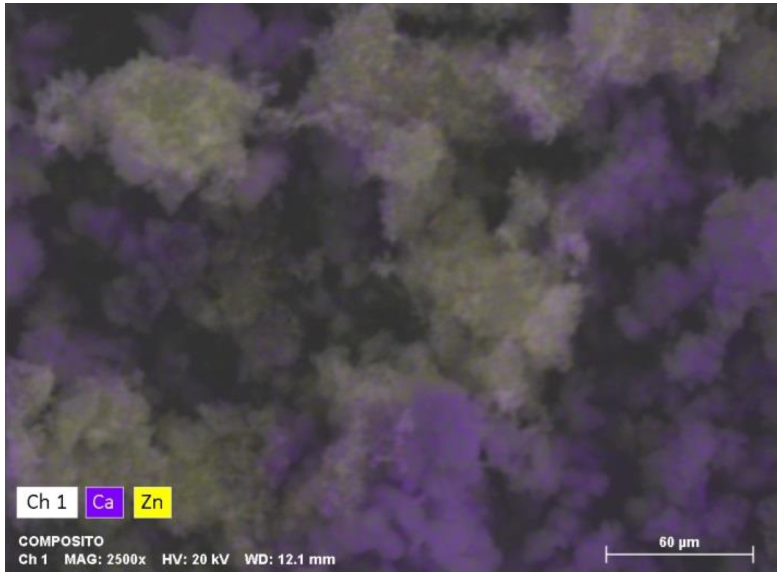


Figure 3. Mapping for Zn (yellow) and a (purple) Source: Own elaboration

Atoms	ZnO (% At.)	HAp (% At.)	Compósite (% At.)
C	-	22.59	34.28
O	57.26	53.13	45.47
Zn	42.74	-	7.63
P	-	0.12	0.32
Ca	-	24.16	12.3
Ca/P		201	38.44
C/P		188	107
Ca/Zn		-	1.61

Table 1. Elemental analysis results for ZnO, HAp and Composite. *Source: own elaboration*

Results

Scanning Electron Microscopy

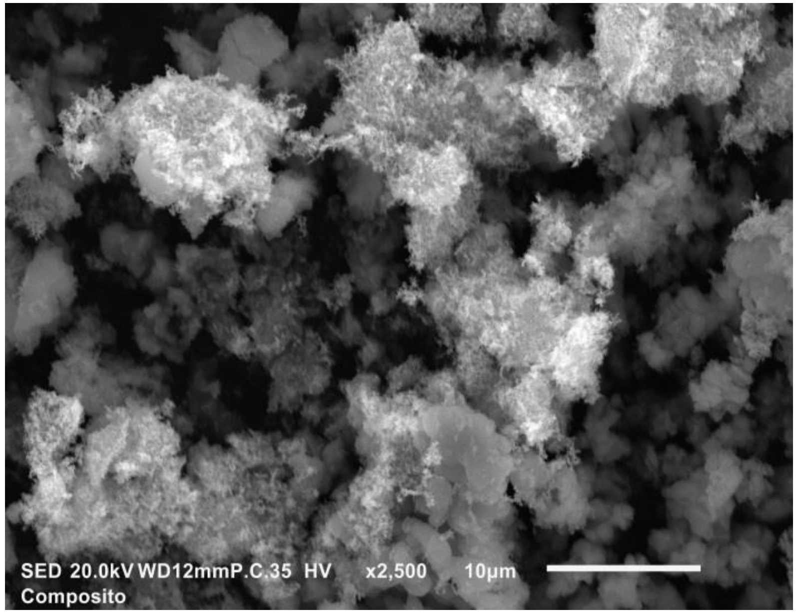


Figure 4. SEM image of composite. *Source: own elaboration*

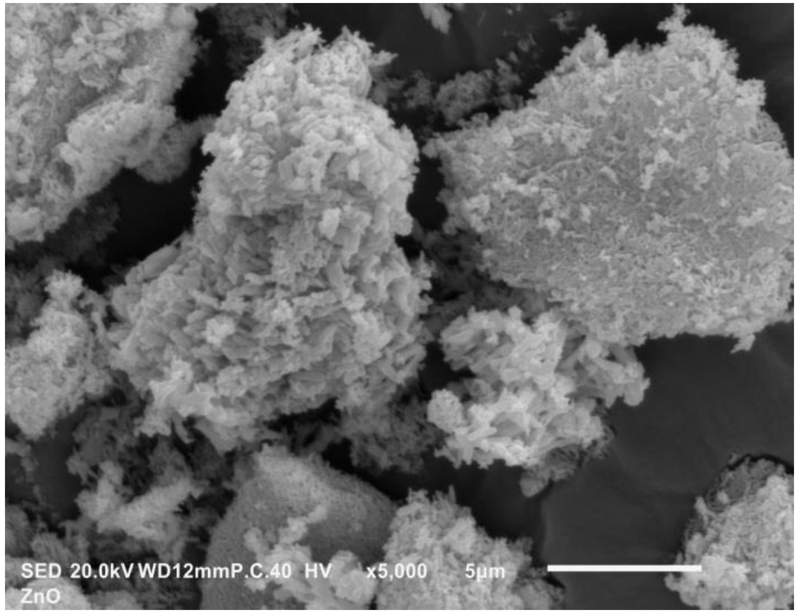


Figure 5. SEM Image of ZnO. *Source: own elaboration*

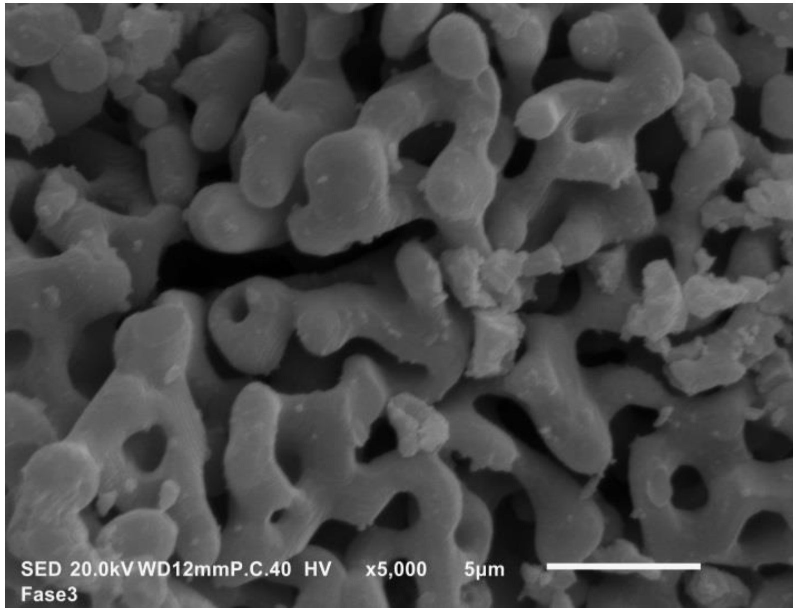


Figure 6. SEM Image of Hydroxyapatite. *Source: own elaboration*

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