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Study physical properties of composite ZnO- Sb_2O_3 using liquid Impregnation Method

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ABSTRACT

Physical mixing method was employing to prepare composite ZnO – Sb_2O_3. In this method 1 gm. of ZnO mixed with different weight of Sb_2O_3 (0.25, 0.50, 0.75, 1gm) and calcing in furnace (700ºC) for three hours. The physical properties have been studied using liquid Impregnation Method, such as the effect of apparent porosity, the effect of Pore Volume, the effect of Bulk Density. The effect of Temperature on the physical properties such as Apparent Porosity, Pore Volume and Bulk Density was studied. The coupled characterized were investigated by using FTIR, XRD, and UV-Visible techniques.

KEY WORDS: Apparent Porosity, Calcination Temperature, Pore Volume, Bulk Density, liquid Impregnation Method.

1. INTRODUCTION

Stoyanova (2013), showed that firstly synthesized TiO_2/ZnO powders then studied the photoactivity of semiconductor on E. coli bacteria (ATCC 25922). Algubury (2015), indicated how can Prepare the composite of ZnO-Co_2O_3, were prepared by thermal wet mixing method, photo activity of this coupled using n-unidecane, explain that the study of Photcatalytic degradation of Rohdamine b using zinc oxide. Bandara (2002), observed that prepared ZnO/SnO_2 nanocrystalline particles then compared with individual ZnO , TiO_2. The result indicates the couple is more active in photocatalytic degradation of dyes, Algubury (2015), successed in Photocatalytic Decolorization of Brilliant Cresyl Blue using Zinc Oxide. Amelia (2010), synthesized the mixture of ZnO and Sb_2O_3 at different ratio. The characters of obtained sample such as density and microstructure was analyzed using SEM, and X-Ray diffraction.

The homogeneous co-precipitation method was proposed to synthesize nanoscale binary mixed oxide ZnO – SnO_2 in the presence of ethyl acetate. The products were characterized by powder X-ray diffraction (XRD) and transmission electron microscopy (TEM). Their photocatalytic activity was investigated for the liquid-phase photocatalytic degradation of methyl orange (MO) (Zhang, 2013).

Algubury (2016), Study the effect of coupled titanium dioxide and cobalt oxide on photo catalytic degradation of malachite green. Photocatalytic Cracking of P-nitro aniline using coupled ZnO – Sb_2O_3.

2. EXPERIMENTAL PROCEDURE

Chemicals: Zinc oxide (ZnO): purity (98%) , supplied by Fluka AG; Antimony tri Oxide (Sb_2O_3).

Study the physical properties of coupled ZnO-Sb_2O_3: In this thesis some of physical properties of prepared coupled ZnO-Sb_2O_3 determined such as Apparent Porosity, Total of pore Volume and Density of catalysts ZnO-Sb_2O_3 using liquid Impregnation Method (Chen, 2013; Mohammed, 2015; Yogendra, 2011). This method involves drying beads then weighing (w_1). After that, immersed the beads in boiling water for five minutes then cooled gradually using cloth for removal of the increase of water then weighing (w_2). Put the beads in the water using Buckle hanging from the balance then weigh (w_3).

Apparent Porosity: The percentage ratio between the volume fluids that can enter into particles of semiconductor (pores space) and the total volume of semiconductor is, as shown in the equation:

$$P_A = \frac{W_2 - W_1}{W_2 - W_3} X100 \qquad (1)$$

W_1: weight of dry piece, W_2: weight of piece soaked, W_3: weight of the piece soaked and immersed.

Pore Volume: The total pore volume can be calculated according to the equation below:

$$V_P = \frac{W_2 - W_1}{W_1 X d_w} \qquad (2)$$

d_w : density of water

Bulk Density: Density is the mass of unit volume, calculation of bulk density by using the equation

$$D_c = \frac{W_1 d_w}{W_2 - W_3} \qquad (3)$$

3. RESULTS AND DISCUSSION

XRD Spectrum: In this technique (XRD) diffraction, we can study the effect of mixing of two semiconductors ZnO-Sb_2O_3 at temperature (700ºC). From the Fig.1below: The zinc oxide spectrum (1 - a), antimony tri oxide spectrum (1 – b) and coupled ZnO-Sb_2O_3 spectrum (1- c), with specific two theta 2Ɵ and intensity.

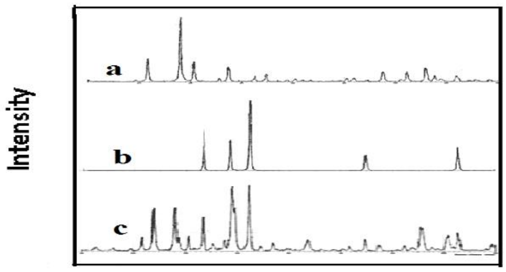

Fig.1. XRD diffraction spectrum of a) Zinc oxide, b) Antimony tri oxide, c) Coupled ZnO - Sb_2O_3

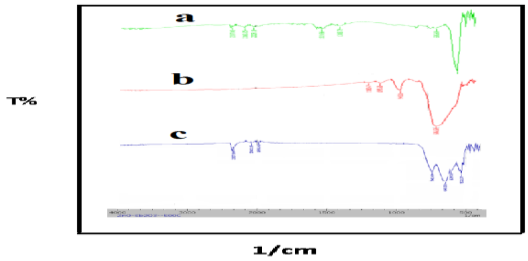

Fig.2. FTIR spectrum of a) Zinc oxide, b) Antimony tri oxide, c) Coupled ZnO - Sb_2O_3

The result appear, in Fig.1, from the graph above its easy to calculate the diffraction angle (2θ), inter planer spacing (d), and relative intensity for the naked ZnO by using X-Ray technique. From Fig.1a above the values of (2Ɵ) for the naked ZnO in spectrum are (36.2911, 34.4626, 31.805) respectively. From the Fig.1b above diffraction angle (2θ), inter planer spacing (d), and relative intensity for the naked Antimony trioxide can be calculated using X-Ray technique, the values of (2Ɵ) for the naked Antimony Trioxide are (29.0753, 25.8860, 30.3901) respectively. The value of (d) equals (3.06873, 3.43912, 2.93889) respectively. At last the value of relative intensity of Sb_2O_3 equal (100, 34, 32) respectively. From the Figure above diffraction angle (2θ), inter planer spacing (d) , and relative intensity for the coupled ZnO-Sb_2O_3 (100:100 %), calculated by using X-Ray technique. From Fig. 1c, the values of (2θ) for the coupled ZnO-Sb_2O_3 (100:100%) are (34.6441, 36.2914, 27.0492) respectively. The value of (d) equals (2.58714, 2.47339, 3.29381) respectively. The value of relative intensity of coupled ZnO-Sb_2O_3 (100:100 %) equals (100, 87, 61) respectively.

FTIR Spectrum: The figure 2a, represent the spectrum of FTIR for naked zinc oxide different peaks shows (2337.80, 2160.35, 2029.1, 1519.96, 1388.79, 686.68, 265 cm^{-1}), while the spectrum of FTIR 2b shows different peaks representing naked Antimony Trioxide (1180.47, 1095.60, 949.01, 686.69 cm^{-1}). For mixed semiconductors (ZnO-Sb_2O_3) Fig.2c, the spectrum of FTIR shown different peaks represent Coupled ZnO-Sb_2O_3 at 700^0C (2337.80, 2003.19, 1990.60, 740.69, 640.10, 609.53, 532.37) cm^{-1}. From the Fig.2c, can see the shift in peaks and reduce its intensity, this mean the mixed between two semiconductors occurs.

Study the Physical properties of coupled ZnO-Sb_2O_3: Physical properties include apparent porosity, Pore volume and Bulk density were investigated for naked ZnO, Sb_2O_3, and the coupled ZnO-Sb_2O_3 by using liquid Impregnation Method.

Apparent porosity: In this project apparent porosity was studied using liquid Impregnation Method, using different masses of Sb_2O_3 (0.25, 0.5, 0.75, 1 gm), mixed with (1gm) of naked ZnO (1 gm). The Table1 and Fig. 3 contained all results obtained .

Table.1.The porosity of naked ZnO, naked Sb_2O_3 and coupled ZnO-Sb_2O_3 at different percentage weight

Mixture of ZnO-Sb_2O_3 (gm/gm)	% of Sb_2O_3	% of Porosity
1.00 : 0.00	0.0	15.67%
0.00 : 1.00	25	13.37%
1.00 :0.25	50	17.94%
1.00 :0.50	75	23.59%
1.00 :0.75	100	30.90%
1.00 :1.00	100	40.66%

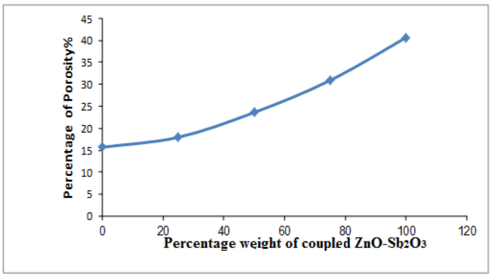

Fig.3. The relation between percentage of Apparent porosity and percentage of coupled ZnO-Sb_2O_3.

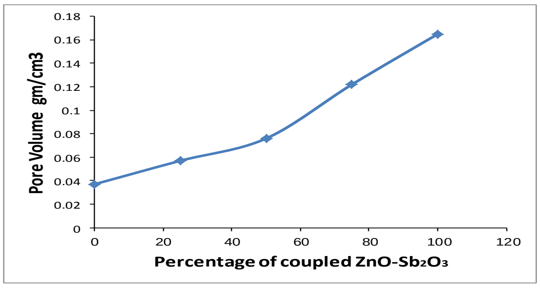

Fig.4.The relation between percentage of pore Volume and percentage weight of coupled ZnO-Sb_2O_3.

As shown in Fig. 3 and Table 1, the values of apparent porosity for ZnO (15.67%), after adding different amount of naked Sb_2O_3 are (0.25, 0.5, 0.75 and 1.0 gm). The porosity increases due to removing partials of water, carbon dioxide and volatile materials, leaving holes in the coupled semiconductors. On the other hand, the bonded between zinc oxide and antimony tri oxide creates new holes.

Pore Volume: Studying pore volume for the naked ZnO, naked Sb_2O_3 and coupled ZnO-Sb_2O_3 again using liquid Impregnation Method at different weights of Sb_2O_3 are (0.25, 0.5, 0.75, 1 gm), with fixed value of naked ZnO (1 gm). The result appears in Table 2 and Fig. 4.

Table.2.The Pore Volume of naked ZnO, naked Sb_2O_3 and coupled ZnO-Sb_2O_3 at different percentages

No.	Mixture of ZnO-Sb_2O_3 (gm/gm)	% of Sb_2O_3	Pore Volume gm/cm^3
1	1.0 : 0.00	0.0	0.037
2	0.0 : 1.00	25	0.038
3	1.0 : 0.25	50	0.057
4	1.0 : 0.50	75	0.076
5	1.0 : 0.75	100	0.122
6	1.0 : 1.00	100	0.165

Table.2 and Figure.4 show that the pore volume of naked ZnO is (0.037cm^3/gm). This value increase when different amounts are added to the naked ZnO until reaching the maximum value at (0.165). This result occurs due to the stacking and rapprochement between the particles. It will be the largest symmetric in the same type of particles such as particles of ZnO but will be less symmetric when mixing ZnO particles with Sb_2O_3 particles. This leads to increasing the pore volume of different particles.

Apparent Density: In this work the apparent density are studied by using liquid Impregnation Method, for naked ZnO, naked Sb_2O_3 and coupled ZnO-Sb_2O_3 when different weight of Sb_2O_3 (0.25, 0.5, 0.75, 1 gm) mixed with the ZnO semiconductor as shows in Table.3 and Figure.5.

Table.3.The Apparent Density of naked ZnO, naked Sb_2O_3 and coupled ZnO-Sb_2O_3 at different percentage

Mixture of ZnO-Sb_2O_3 (gm/gm)	% of Sb_2O_3	Apparent Density gm/cm^3
1.0 :0.00	0.0	4.194
1.00: 0.25	25	3.096
1.0 :0.50	50	3.073
1.0 :0.75	75	2.523
1.0 :1.00	100	2.454

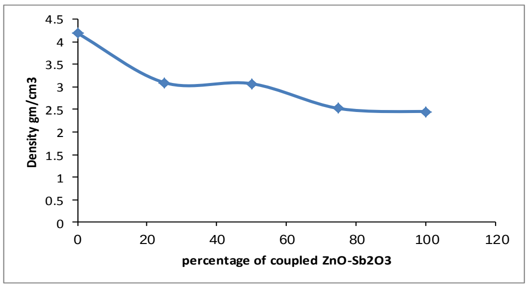

Fig.5.The relation between Apparent Density and percent weight of coupled ZnO-Sb_2O_3.

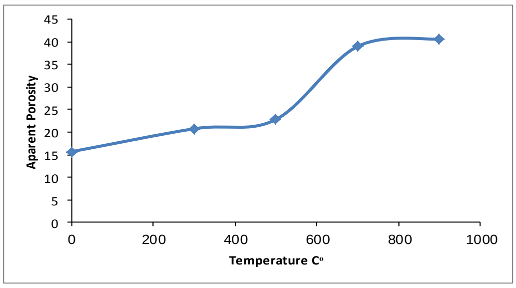

Fig.6.The relation between Apparent Porosity and different temperatures at constant percentage weight of coupled ZnO-Sb_2O_3 (1:1)

The Table.3 and Figure.5 include the measurement of apparent density for naked ZnO (4.194 gm /cm^3). This value of apparent density decreases after adding different amounts the naked Sb_2O_3 until reach to minimum value at (2.454gm/ cm). This behavior happened because of increasing the porosity of coupled ZnO-Sb_2O_3 when increasing the amount of Sb_2O_3 ,this lead to decrease of weight of Tablet of coupled ZnO-Sb_2O_3 and as a result of that apparent density decreases according to the following equation: [density = weight (gm) / volume (cm^3)]. That mean apparent density of oxide (ZnO, Sb_2O_3) is greater than coupled ZnO-Sb_2O_3.

The effect of Temperature on the physical properties of coupled ZnO-Sb_2O_3 (1:1): The effects of different temperature (300, 500, 700, and 900^{0}C) on the physical properties (Apparent porosity, Pore volume and Apparent density) of coupled ZnO-Sb_2O_3 (1:1) were studied at constant time (4 hours), using liquid Impregnation method.

Apparent Porosity: The Apparent Porosity of coupled ZnO-Sb_2O_3 (1:1) was studied at different temperatures (300, 500, 700 and 900^{0}C). The time of calcination process for coupled ZnO-Sb_2O_3 was (4 hours). The results are clear and they are indicated in the Table.4 and Fig.6.

Table.4.The Apparent Porosity of coupled ZnO-Sb_2O_3 (1:1) at different temperatures

Catalyst	Apparent Porosity %			
Coupled ZnO-Sb_2O_3 (1:1)	300	500	700	900
	20.75%	22.80%	39.01%	40.66%

Pore volume: The pore volume of coupled ZnO-Sb_2O_3 (1:1) was studied at different temperatures (300, 500, 700 and 900^{0}C). The time of calcination process for coupled ZnO-Sb_2O_3 (4 hours). The results appear in the Table.5 and Figure.7.

Table.5.The Pore Volume of coupled ZnO-Sb_2O_3 (1:1) at different temperatures.

Coupled ZnO-Sb_2O_3	**Pore Volume**			
	300	500	700	900
	0.067	0.082	0.152	0.165

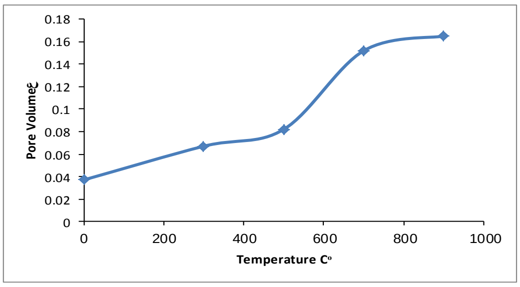

Fig.7. The relation between Pore Volume and different temperatures at constant percentage weight of coupled ZnO-Sb_2O_3 (1:1).

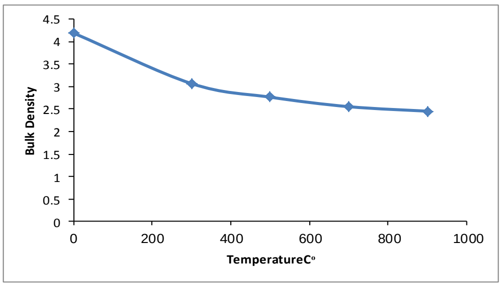

Fig.8. The relation between Bulk Density and different temperatures at constant percentage weight of coupled ZnO-Sb_2O_3 (1:1).

Bulk Density: The Bulk Density was studied at constant percentage of coupled ZnO-Sb_2O_3 (1:1) using different temperatures (300, 500, 700 and 900^{0}C), the duration of calcination (2 hours), the results are noted in the Table.6 and Fig.8.

Table.6.The Bulk Density of coupled ZnO-Sb_2O_3 (1:1) at different temperatures

catalyst	**Bulk Density**			
Coupled ZnO-Sb_2O_3 (1:1)	300	500	700	900
	3.069	2.766	2.554	2.454

From Figure.8 and Table.6 it is shown that the calcination increases the porosity and the maximum value found at 900^{0}C because the water and carbon dioxide and other volatile materials removal when the temperature increase leaving holes in the semiconductors.

4. CONCLUSION

a) The coupled ZnO-Sb2O3 was prepared by using 1 gm of ZnO and 1 gm of antimony tri oxide then calcined at 700^{0}C.

b) The physical properties of coupled ZnO-Sb_2O_3 were studied using liquid Impregnation method.

c) The porosity increases due to removing partials of water, carbon dioxide and volatile materials, leaving holes in the coupled semiconductors. On the other hand, the bonded between zinc oxide and antimony tri oxide creates new holes.

d) This value increase when different amounts are added to the naked ZnO until reaching the maximum value at (0.165).

e) This value of apparent density decreases after adding different amounts the naked Sb_2O_3 until reach to minimum value at (2.454gm/ cm).

f) The calcinations increase the porosity and the maximum value found at 900^{0}C.

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