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Experimental data on synthesis and characterization of WO₃/TiO₂ as catalyst

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ABSTRACT

WO₃/TiO₂ is a composite photocatalyst that is being widely used in heterogeneous photocatalysis because it presents better photocatalytic properties than TiO₂. For example, the probability of recombination of the electron/hole pairs is diminished and a more range of the solar spectrum is used for its excitation. However, this depends of variables such as tungsten oxide concentration, calcination temperature and synthesis method. This work is focused in establish the effect of WO₃ on the morphological and structural characteristics of TiO₂. WO₃/TiO₂ was synthesized by sol-gel method at different calcination temperatures and at different concentrations of tungsten oxide. The surface area, the possible transition between valence band and conduction band, particle size, elemental analysis and crystallography were examined through the BET, DRS, SEM-EDS and XRD analysis.

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1. Data

Doping TiO₂ pretend to improve its photocatalytic performance, since even though it presents a great effectiveness in the degradation of recalcitrant compounds only it achieves its excite state by absorption UV energy, which correspond to 5% of solar spectrum. So more than 50% of visible radiation is being wasted [2,3]. Therefore, it is necessary the coupling of this catalyst with another compound or

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Specifications table

Subject area	Chemical engineering, chemistry
More specific subject area	Synthesis and characterization of materials
Type of data	Figure and table
How data was acquired	Data were obtained by means of characterization techniques such as XRD, SEM-EDS. The specific surface area was determined by BET methodology and the band gap (Eg) values were determined by Kubelka-Munk methodology from DRS analysis.
Data format	Analyzed
Experimental factors	Effect of the calcination temperature (500, 600 and 700 °C) and percentages by weight of WO ₃ (1, 3, 5% w/w) on the characteristics of the photocatalyst such as crystal structure and morphology were evaluated.
Experimental features	Synthesis of WO ₃ /TiO ₂ was carried out through the implementation of the Sol-Gel method with the objective of improving the photocatalytic activity of TiO ₂ having as a response variable the reduction of Eg, which could imply a redshift in terms energy absorption that result beneficial in photocatalytic process under natural sunlight.
Data source location	GAOX, Universidad del Valle, Cali, Colombia.
Data accessibility	The data is found only in this article. A. Arce-Sarria, F. Machuca-Martínez, C. Bustillo-Lecompte, A. Hernández-Ramírez, and J. Colina-Márquez, Degradation and Loss of Antibacterial Activity of Commercial Amoxicillin with TiO ₂ /WO ₃ -Assisted Solar Photocatalysis, <i>Catalysts</i> . 8 (2018) 1–14. https://doi.org/10.3390/catal8060222 [1].

Value of data

- Data obtained allow knowing the calcination temperature effect and the percentage by weight of WO₃ in the crystalline structure of the synthesized photocatalyst.
- It can be observed that the addition of WO₃ allows that anatase phase of TiO₂ be more thermally stable which could contribute to the improvement of photocatalytic activity of WO₃/TiO₂.
- The band gap data for each sample of photocatalyst at different percentage by weight of WO₃ and calcination temperature were obtained, which could serve as references for improving the doping with another oxide.
- Data may be useful for future research.

mixed oxides and characterize the properties of the new materials product of doping. In this case WO₃/TiO₂. Some physicochemical properties of titanium oxide and tungsten oxide are shown in [Table 1](#).

2. Experimental design, materials and methods

Calcination temperature directly affects the crystalline structure of TiO₂. It was found that anatase phase presents a better photocatalytic performance than rutile phase [4], so in this work three calcination temperatures 500, 600 and 700 °C were evaluated. Another parameter for improving the photocatalytic activity of TiO₂ is the doping percentage by weight of WO₃, which favor the shift in the energy absorption toward visible light region. In this case it was varied in 1, 3 and 5% w/w.

2.1. General procedure

The synthesis of WO₃/TiO₂ photocatalyst was carried out by Sol-Gel methodology using Titanium (IV) n-butoxide, 98+ % ACROS (CAS RN 5593-70-4) and p-Tungstate ammonium, 99.99% Aldrich (CAS 11120-25-5) as precursors of the obtained materials [5].

2.2. Characterization

Surface area of the photocatalyst obtained was determined by nitrogen physisorption onto material surface using the Brunauer, Emmett and Teller (BET) theory. The Kubelka-Munk function was used for estimating the Eg based on the reflectance spectroscopy values [1]. The surface area and band gap of the synthesized photocatalyst to different conditions are shown in [Table 2](#) and [Table 3](#), respectively.

Table 1

Physicochemical properties of oxides.


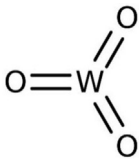
	Titanium Oxide	Tungsten Oxide
Molecular structure		
Molecular formula	TiO ₂	WO ₃
Molecular Weight [g/mol]	79.8	231.8
Band gap Eg [eV]	3.2	2.8
Water solubility	Insoluble	Insoluble

Table 2Surface area of bare TiO₂ and TiO₂ doped with WO₃ (m₂/g).

WO ₃ percentage	Calcination temperature		
	500 °C	600 °C	700 °C
0%	41.8	5.4	0.11
1%	52.3	16.8	0.16
3%	64.6	37.2	10.2
5%	68.7	45.1	18.4

Table 3Band gap results of WO₃/TiO₂ particles.

Sample	Eg (eV)	Wavelength (nm)
TiO ₂ -500 °C	3.18	388.72
TiO ₂ -600 °C	3.15	391.99
TiO ₂ -700 °C	3.11	396.65
1% WO ₃ /TiO ₂ -500 °C	3.04	406.37
1% WO ₃ /TiO ₂ -600 °C	2.98	414.69
1% WO ₃ /TiO ₂ -700 °C	2.96	416.82
3% WO ₃ /TiO ₂ -500 °C	3.02	408.57
3% WO ₃ /TiO ₂ -600 °C	2.97	416.30
3% WO ₃ /TiO ₂ -700 °C	2.95	418.45
5% WO ₃ /TiO ₂ -500 °C	2.99	413.44
5% WO ₃ /TiO ₂ -600 °C	2.95	418.15
5% WO ₃ /TiO ₂ -700 °C	2.93	420.92

In order to know the morphology and composition WO₃/TiO₂ photocatalyst samples, SEM and EDS analyzes were performed. The results are shown in Fig. 1 and Table 4 respectively.

XRD analysis was performed on samples calcined at 600 °C (Fig. 2.) and 700 °C (Fig. 3.) because at these temperatures the crystalline transition is achieved. JCPDS 21–1272 and JCPDS 21–1276 cards were used as patterns for the anatase phase and the rutile phase respectively.

The Fig. 4 shows the relationship between the Anatasa and Rutile phase on different catalysts synthesized.

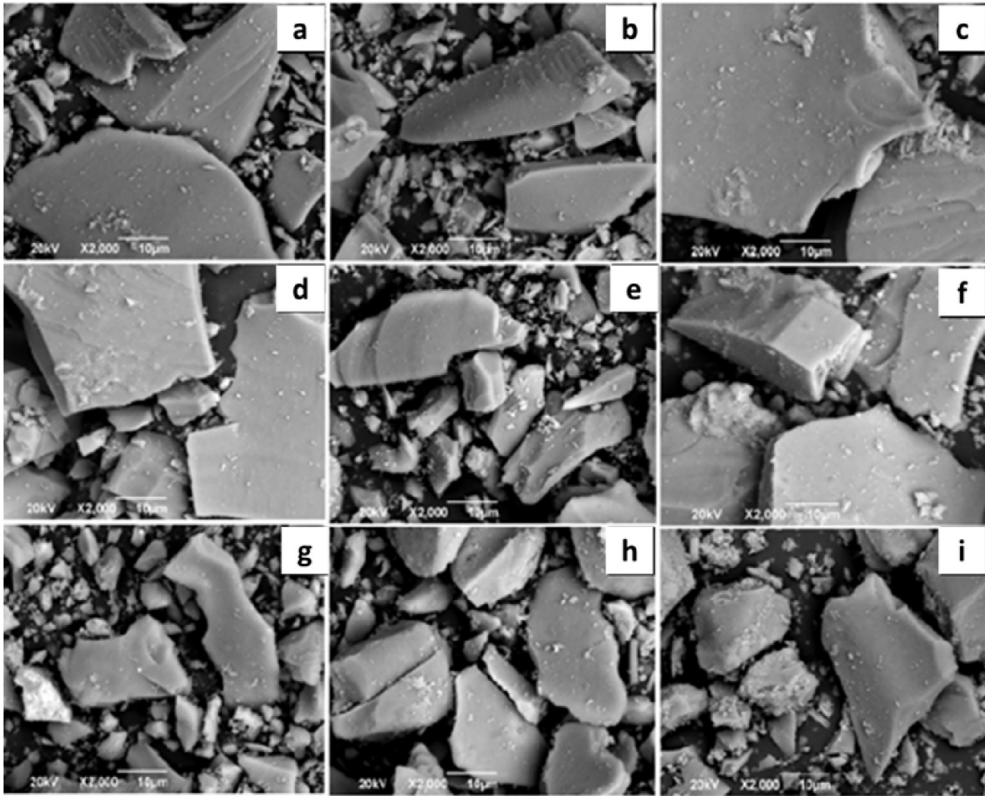


Fig. 1. SEM for WO_3/TiO_2 materials. (a) TiO_2 -600 °C, (b) TiO_2 -700 °C, (c) 1% WO_3/TiO_2 -600 °C, (d) 1% WO_3/TiO_2 -700 °C, (e) 3% WO_3/TiO_2 -600 °C, (f) 3% WO_3/TiO_2 -700 °C, (g) 5% WO_3/TiO_2 -500 °C, (h) 5% WO_3/TiO_2 -600 °C, (i) 5% WO_3/TiO_2 -700 °C.

Table 4

Elemental composition according to EDS analysis, given in percentage of element in the sample.

Sample	O	Ti	W
TiO_2 - 600 °C	43.27	56.73	—
TiO_2 - 700 °C	47.28	52.72	—
1% WO_3/TiO_2 - 600 °C	44.29	54.82	0.89
1% WO_3/TiO_2 - 700 °C	43.07	56.05	0.88
3% WO_3/TiO_2 - 600 °C	43.09	54.27	2.64
3% WO_3/TiO_2 - 700 °C	38.76	58.54	2.71
5% WO_3/TiO_2 - 500 °C	51.08	44.08	4.84
5% WO_3/TiO_2 - 600 °C	41.91	52.95	5.14
5% WO_3/TiO_2 - 700 °C	36.77	58.46	4.78

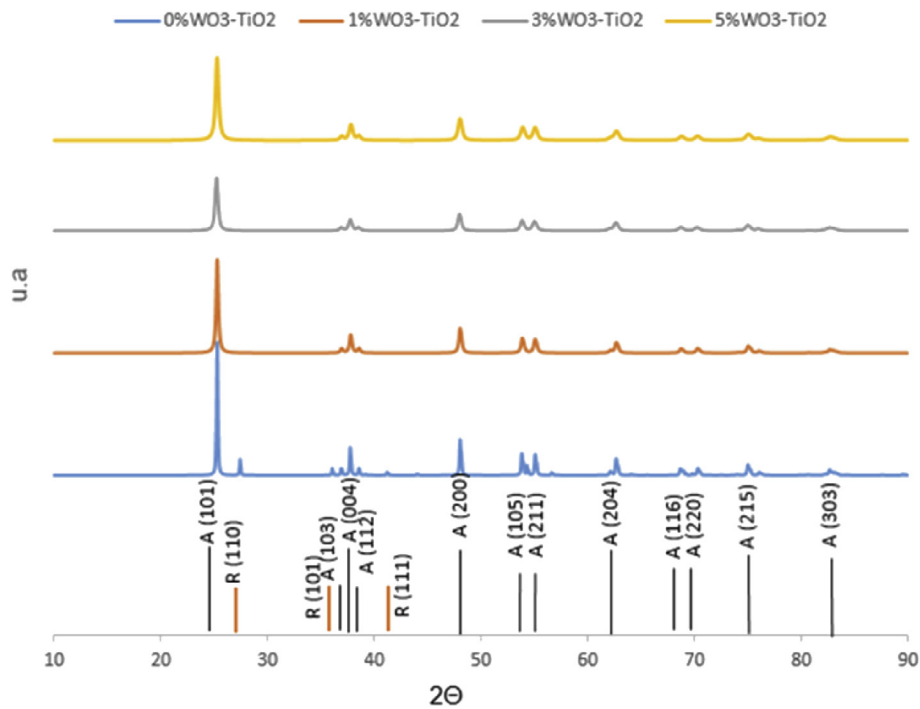


Fig. 2. Diffractograms obtained from samples synthesized from WO_3/TiO_2 calcined at 600°C (Anatase) and R (Rutile).

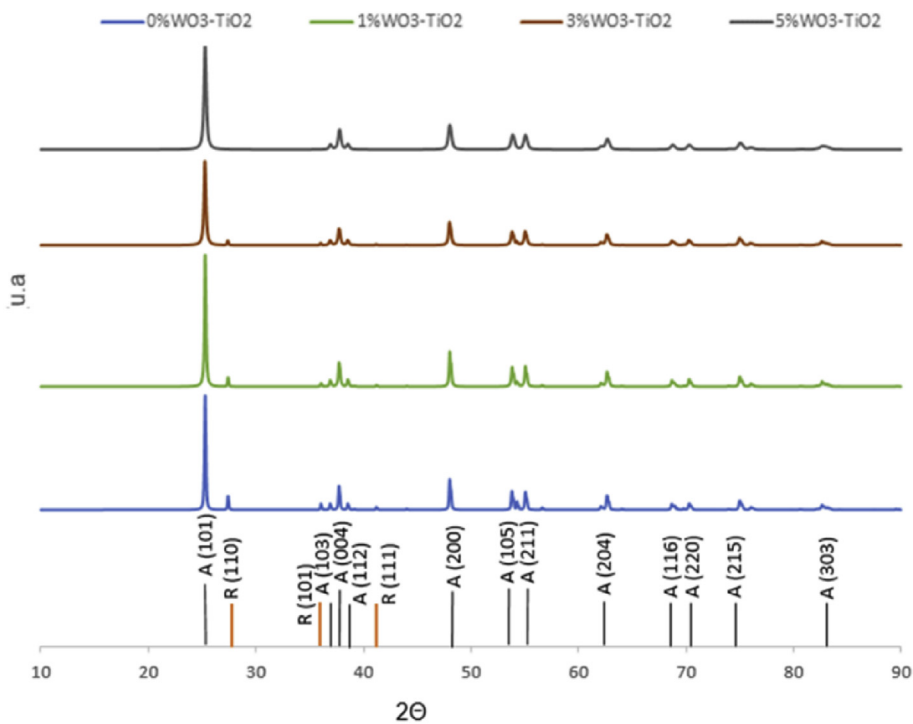
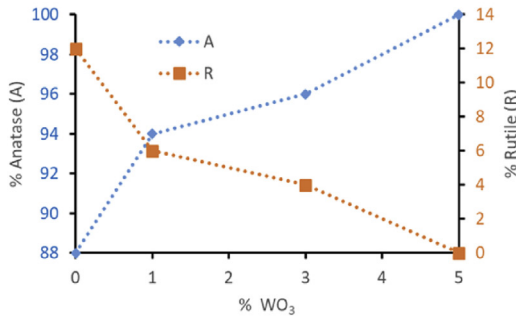


Fig. 3. Diffractograms obtained from samples synthesized from WO_3/TiO_2 calcined at 700°C A (Anatase) and R (Rutile).



Sample	Crystalline phases (%)		Crystal size (nm)	
	A	R	A	R
TiO ₂ -600°C	95.8	4.2	540.1	33.8
TiO ₂ -700°C	88.2	11.8	248.2	46.2
1% WO ₃ /TiO ₂ -600°C	100.0	0.0	494.5	*
1% WO ₃ /TiO ₂ -700°C	93.9	6.1	255.3	27.7
3% WO ₃ /TiO ₂ -600°C	100.0	0.0	258.7	*
3% WO ₃ /TiO ₂ -700°C	96.4	3.6	213.9	18.2
5% WO ₃ /TiO ₂ -600°C	100.0	0.0	255.3	*
5% WO ₃ /TiO ₂ -700°C	100.0	0.0	224.3	*

Fig. 4. Diagram of crystalline phases distribution A (Anatase), R (Rutile) and crystal size.

Acknowledgment

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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