Effect of the Concentration of Dissolvent and Reductor Agent in the Synthesis of Tungsten-based Structures Using Microwave Approach and It Use as Catalysts in Dmf Hydrogen Donor

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Abstract

Tungsten-based structures by microwave approach were synthesized. Four different essays were used to analyze the effect of some synthesis variables; in two of them the effect of the concentration of the solvent was studied using pure ethylene glycol and a mix of ethylene glycol and water, while the other samples the effect of the presence of reductor agent using sodium borohydride was studied. All the samples were prepared using as precursor ammonium tetrathiotungstate and sulfur in excess to incentive the growth of structures enriched in this element, for each synthesis 1 minute of irradiation was used with microwave power level of 1200 watts and frequency of 2.64 GHz. The structural, morphological, and optical characterizations were carried out with Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), infrared and uv-vis spectroscopies. By SEM, all the samples demonstrated differences in the size and morphology derivate of the modification of the experimental variables, the order of magnitude of the synthesized materials ranged from 0.01 μm until 6.5 μm , with morphologies of tetragonal prisms, flakes, irregular agglomerates or flower-like depends on the characteristics of the precursor solution. TEM lets to determine the composition of each sample, corroborated also by IR and Diffuse Reflectance spectroscopies. The four samples were used as catalysts in the study of dimethylformamide (DMF) hydrogen donor.

Full Text

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Tables

Table 1

Loss Tangent ($\tan \delta$) of different solvents at 2.45 GHz and 20°C [20]

High		Medium		Low	
Solvent	$\tan \delta$	Solvent	$\tan \delta$	Solvent	$\tan \delta$
Ethylene glycol	1.17	2-Butanol	0.45	Chloroform	0.091
Ethanol	0.94	2-Methoxyethanol	0.41	Ethyl acetate	0.059
2-Propanol	0.8	1-Hexanol	0.34	Acetone	0.054
1-Propanol	0.76	Dichlorobenzene	0.28	Dichloromethane	0.042
Formic acid	0.72	Acetic acid	0.17	Toluene	0.04
Benzyl alcohol	0.67	DMF	0.16	2-Ethoxyethanol	0.039
Methanol	0.66	Dichloroethane	0.13	Hexane	0.02
Nitrobenzene	0.59	Water	0.12	o-Xylene	0.018
1-Butanol	0.57	Chlorobenzene	0.1		
Isobutanol	0.52				

Table 2

Interplanar spacing (theoretical and experimental)

Sample	Measured d (Å)	PCPDF Card	Theoretical d (Å)	hkl	Crystalline system
Water - ethylene glycol with NaBH ₄	3.918	89-1287	3.88	001	Tetragonal
	3.684		3.695	200	
	3.436		3.4353	101	
	3.317		3.3049	210	
	3.116		3.1151	111	
	2.701		2.6757	201	
	2.638		2.6127	220	
	2.505		2.5159	211	
	2.341		2.3369	310	
	2.168		2.1672	221	
	2.089		2.0796	301	
	2.000		2.0018	311	
	1.798		1.7923	410	
	1.724		1.7178	202	
	1.665		1.6680	401	
	1.650		1.6524	420	
	1.629		1.6271	411	
	1.557		1.5575	222	
	1.517		1.5203	421	
	1.439		1.4493	510	
	1.243		1.2380	441	
	1.119		1.1188	521	
	1.079		1.0937	323	
Water - ethylene glycol without NaBH ₄	3.842	89-1287	3.88	001	Tetragonal
	3.711		3.695	200	
	3.484		3.4353	101	
	3.297		3.3049	210	
	3.126		3.1151	111	

	2.703		2.6757	201	
	2.602		2.6127	220	
	2.475		2.5159	211	
	2.355		2.3369	310	
	2.177		2.1672	221	
	1.982		2.0018	311	
	1.839		1.8475	400	
	1.810		1.8187	112	
	1.657		1.6524	420	
	1.583		1.5890	331	
	1.5246		1.5203	421	
	1.477		1.480	430	
	1.439		1.4493	510	
Ethylene glycol without NaBH_4	3.875	20-1324	3.85	001	Orthorhombic
	3.728		3.75	020	
	3.597		3.69	200	
	3.468		3.43	011	
	3.338		3.35	120	
	2.700		2.686	021	
	2.635		2.633	220	
	2.161		2.173	221	
	1.900		1.923	002	
	1.836		1.846	400	
	1.798		1.806	112	
	1.65		1.657	420	
Ethylene glycol with NaBH_4	6.165	84-1398	6.1615	002	Hexagonal
	3.093		3.0807	004	
	2.641		2.6660	101	

Table 3

Values of band gap reported of WO₃, WO₂ and WS₂

Rename	Crystalline structure	Reported Band Gap (eV)
α -WO ₃	Tetragonal	2.9 - 3.21 eV [38-40]
β -WO ₃	Orthorhombic	2.52 eV [41]
γ -WO ₃	Monoclinic	1.98 eV y 2.58 eV, 2.4 eV [39]
δ -WO ₃	Triclinic	2.43 eV y 2.60 eV [37]
ϵ -WO ₃	Monoclinic	2.55 eV - 2.85 eV [37]
WO ₂	Hexagonal	1.863 eV [42]
WS ₂	Hexagonal	1.3 - 1.94 eV [43], [44] 1.51 - 2.03 eV [45]

Figures

Figure 1

SEM micrographs of the samples: a) water-ethylene glycol without NaBH₄, b) ethylene glycol without NaBH₄, c) water-ethylene glycol with NaBH₄ and d) ethylene glycol with NaBH₄

Figure 2

TEM micrographs of the samples: a) water-ethylene glycol without NaBH₄, b) water-ethylene glycol with NaBH₄, c) ethylene glycol without NaBH₄ and d) ethylene glycol with NaBH₄

Figure 3

General IR spectrum of the samples, with functional groups identification.

Figure 4

IR spectral of the different samples: a) ethylene glycol - water without NaBH₄, b) ethylene glycol - water with NaBH₄, c) ethylene glycol without NaBH₄, d) ethylene glycol with NaBH₄. Before irradiation - black lines and after irradiation - red lines.

Figure 5

Band Gap of the samples using the Kubelka Munk function: a) ethylene glycol - water without NaBH₄, b) ethylene glycol - water with NaBH₄, c) ethylene glycol without NaBH₄, d) ethylene glycol with NaBH₄

Figure 6

DMF structure

Figure 7

IR spectrums of DMF