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Original Research Article

Synthesis WO_3 nanoparticle via the electrochemical method and study its super-hydrophobicity properties

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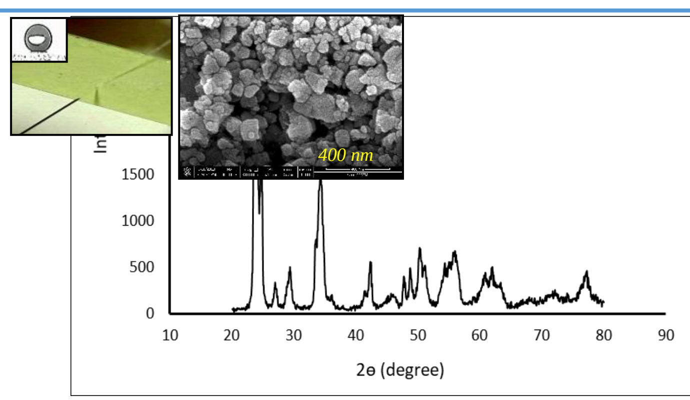
ABSTRACT

This work introduces a simple method to prepare WO_3 nanoparticles via tungsten rod electrooxidation in the presence of NaCl solution as an electrolyte. In this process, WO_3 nanoparticles with sizes between 10nm and 20 nm were prepared and characterized using X-ray diffraction (XRD), scanning electron microscope (SEM), and energy dispersive X-ray spectroscopy (EDX) techniques. WO_3 nanoparticles are used to modify polysiloxane surface. The WO_3 coated polysiloxane surface showed a very high-water contact angle of 158°.

Graphical Abstract



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Introduction

Superhydrophobic surfaces have attracted considerable attention in recent years [1]. Coating surfaces with various metal oxide nanoparticles is a practical method for creating superhydrophobic surfaces. A smooth and homogeneous surface is created with these nanoparticles [2]. In this way, different superhydrophobic surfaces are produced by changing the properties of the particles. Tungsten oxide (WO_3) is one of the nanoparticles metal oxide, which revealed super-hydrophobicity properties [3]. The tungsten oxide nanoparticles have been prepared with a variety of technologies including such as sol-gel [4], hydrothermal [5], thermal decomposition [6], acid precipitation [7], electrophoresis deposition (EDP) [8], chemical vapor deposition (CVD) [9], and physical vapor deposition (PVD) [10]. WO_3 is utilized for various applications such as gas sensing [11], photo-catalyst [12], electrochromic windows [13], and electrochemical sensors [14].

Super-hydrophobicity properties of WO_3 and has been reported in the literature. Sun and et. al in 2018 fabricated WO_3 nanoparticles on wood surfaces with a hydrothermal method. Modified WO_3-coated wood surfaces have 152° water contact angle (WCA) [15]. Fan and *et al*. [16] in 2017 synthesized amorphous 100-300 nm WO_3 nanoparticles on a Q235 steel plate with chemical deposition and obtained 161° WCA. Yu and *et al*. [3] in 2018 introduces a super-hydrophobic WO_3@TiO_2 Nano flake prepared via electrodeposition on stainless steel with 162° WCA for corrosion resistance purposes.

In this study, an electrochemical technique was employed to produce WO_3 from tungsten road. Also investigated the structure of the WO_3 nanoparticles, coating effect of polysiloxane with WO_3 nanoparticles, and the super-hydrophobicity properties of a coated surface.

Experimental

Materils and methods

Preparation of WO_3 nanoparticles

WO_3 nanoparticles were prepared via electrolysis in MEGATC MP-1603 electrochemical device under stable current. W rod (purchased from TWECO Company, grade 2) as an anode, stainless plate as a cathode, 0.5 gr NaCl as electrolyte (0.08 M), and 1 A current condition at room temperature. The obtained product was collected with centrifuge, washed with distilled water, and dried at room temperature. Characterization of the size, morphology, and composition performed by using scanning electron microscopy (SEM) (CamScan MV2300), energy dispersive X-ray analysis (EDX). Phase composition reported in the 2θ range of 10-80° characterized by powder X-ray diffractometer (PANalytical/Philips PW1730, U. K.).

Preparation silicone covered surfaces

0.1 g commercial SR (SelSil 100% silicone polymer, purchased from SEL DIS Turke) were solved in 5 mL *n*-hexane (Mojalali 95%) and sonicate for 15 min then poured on a glass slide (rained with distillate water and cleaned by ethanol) when more half of solvent evaporated nanoparticles were sprinkled on a slide and dried at room temperature. For removing unattached nanoparticles slide was brushed with a very soft brush, washed, and then dried at 110 °C for 2 h.

Wettability characterization

To measure the wettability characteristics of the samples sessile drop method was used.

Deionized water droplets were put on the surface with a microsyringe, and the projection of droplets was captured by using a CCD camera. SCA20 data software used for image analysis of the droplet contact angle (CA). CA of water data reported in this paper measured is the average value of at least 12 different points on the surface.

Results and Discussion

WO_3 nanoparticles were synthesis by an electro-oxidation of W in an aqueous solution of 0.5% W/V NaCl. The yellow precipitate was obtained after electro-oxidation and identified by X-ray diffraction. The X-ray diffraction pattern of WO_3 nanoparticles is demonstrated in Figure 1. Diffractogram peaks indexed as corresponding ones to tungsten trioxide (JCPDS no. 431035) [17]. The Peaks at the value 2θ: 23.53, 24.07, 24.69, 34.28, and 50.18 deg corresponding to 002, 020, 200, 202, and 114 planes, respectively. The XRD reveals that the WO_3 nanoparticles are crystalline in monoclinic form.

Energy-dispersive X-ray analysis (EDX) of nanoparticles are summarized in Table 1 and Figure 2. EDX results confirmed XRD results, as Table 1 illustrates the ratio of tungsten to oxygen is one to three. A small amount of NaCl appeared in EDX which related to the electrolyte.

Surface morphology and the size of nanoparticles was analyzed using the SEM analysis, as seen in Figure 3. Size distribution histograms for the particles are shown in Figure 4, and particles vary from 9 nm to 100 nm and mostly around 9-14 nm.

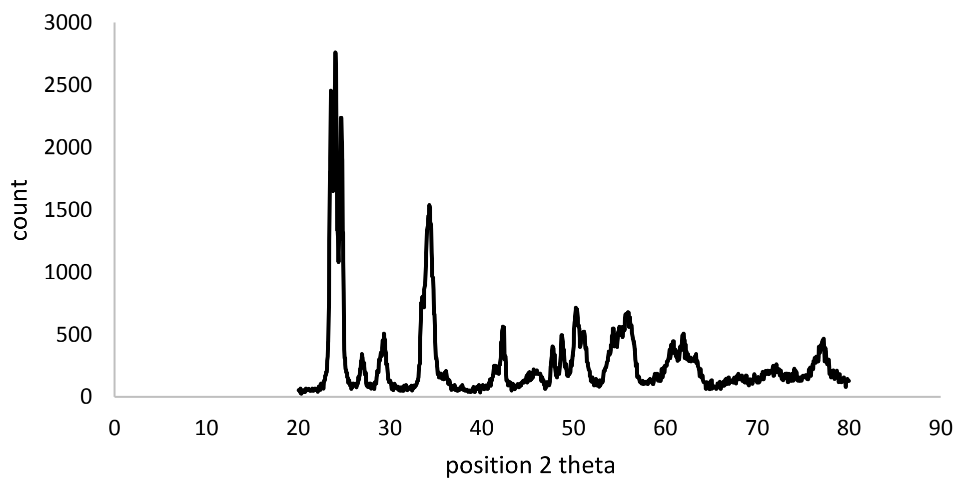


Figure 1. XRD diffractogram of WO_3 nanoparticles

Table 1. EDX results of WO_3 nanoparticles

El	AN	Series	unn. [wt.%]	norm. C [wt.%]	Atom. C [at%]	Error (1 Sigma) [wt.%]
W	74	L-series	55.42	78.04	23.88	2.30
O	8	K-series	15.07	21.23	74.64	4.49
Na	11	K-series	0.26	0.37	0.91	0.09
Cl	17	K-series	0.26	0.36	0.57	0.08
Total: 71.02 100.00 100.00						

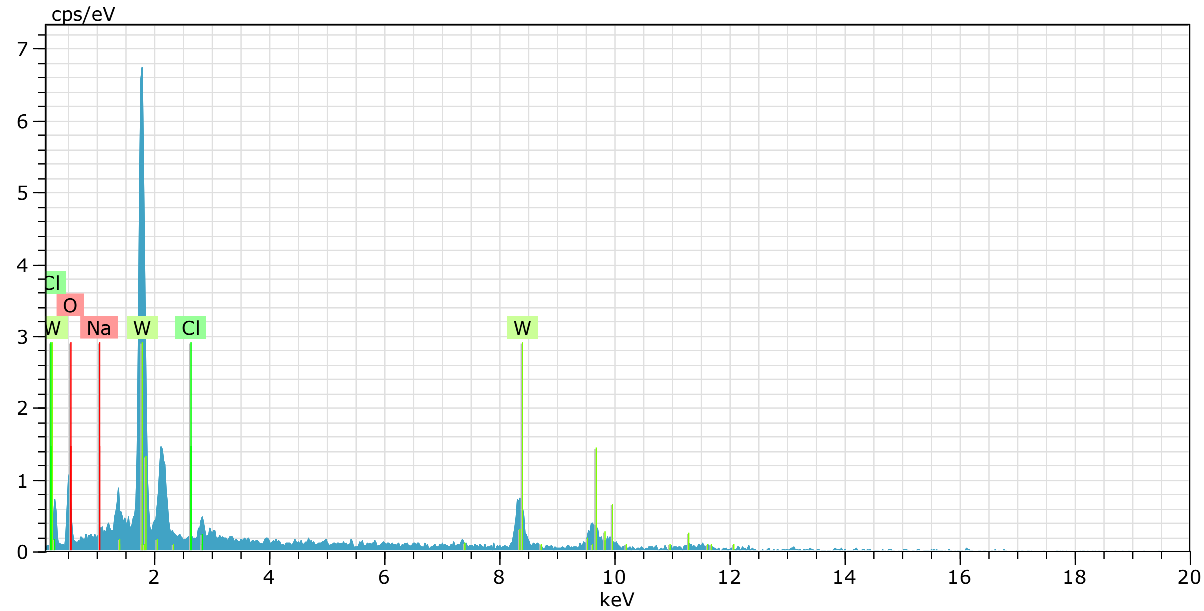


Figure 2. EDX results of WO_3 nanoparticles

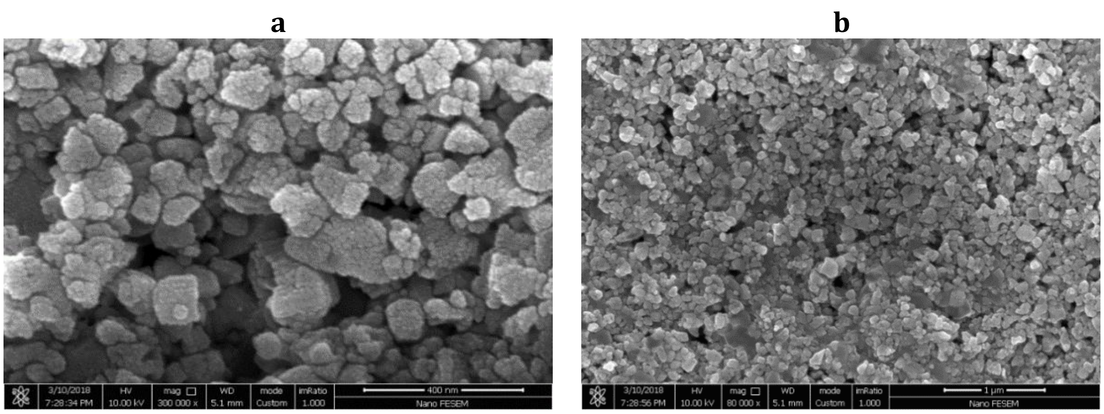


Figure 3. SEM of WO_3 nanoparticles a) 400 nm, b) 1 μm

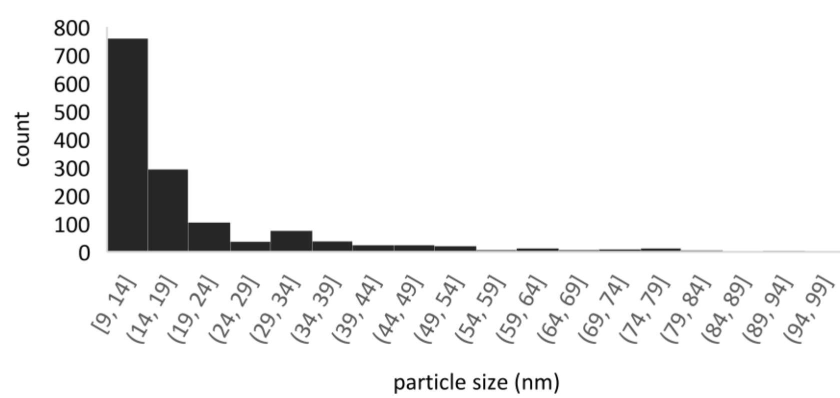


Figure 4. Size distribution histograms for of WO_3 nanoparticles

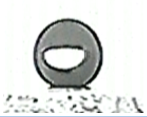
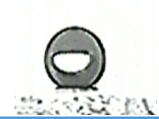
Synthesized WO_3 nanoparticles used for coating polysiloxane surface. The result of the wettability of WO_3 coated on polysiloxane with water, ethylene glycol, and glycerol is summarized in Table 2. Superhydrophobicity stability of the surface studied for 20 min, the result summarized in Table 3. As the results show Contact angle of the sample from the first time until 20 min doesn't show any significant difference. The surface shows a waterjet impact Figure 5.

Figure 5. water jet impact of WO_3 nanoparticles coated polysilicon surface

Table 2. The results of Contact Angle with H_2O, Glycerol, and Ethylene glycol solvents

solvent entry	H_2O	Glycerol	Ethylene glycol
1	161.2	152.2	159.4
2	158.6	157.8	159
3	156.2	156	157.3
4	155.3	153	155.9
5	158.6	156.2	155.7
6	160.2	154.5	154.9
7	157.4	152.2	154.1
8	157.9	157.8	153.7
9	157.5	154.1	153.7
10	158.9	151.8	153.3
11	158.3	156.7	153
12	157.9	152	149.7
Average	158.2	154.5	154.9

Table 3. Stability of Contact Angle with H_2O in 20 min

Time (min)	0	1min	5 min	10 min	15 min	20 min
CA	158	158	158	158	158	158
picture						

Conclusions

In this research study, we introduced a simple, clean, fast, eco-friendly, and economical electrolysis method for the synthesis of WO_3 nanoparticles. WO_3 nanoparticles are prepared in a spherical shape with a particle size of 9-14 nm and characterized by XRD and SEM spectroscopic methods. WO_3 nanoparticles have been used for coating polysiloxane and create a superhydrophobic surface. The wettability of the surface was indicated 158º.

Disclosure Statement

No potential conflict of interest was reported by the authors

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