Synergistic Effect of Nickel on Tungsten Oxide Hydrate ($\text{WO}_3 \cdot \text{H}_2\text{O}$) as Photoanode for Dye-Sensitized Solar cell

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Research Article

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Abstract

Tungsten oxide hydrate (PW) and Nickel tungsten oxide nanocomposites (NW NCs) namely NW1, NW2, NW3 and NW4 were successfully synthesized by the hydrothermal method. The synthesized NCs were characterized using advanced techniques such as X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), UV–visible spectroscopy and Photoluminescence (PL). XRD pattern shows the formation of face-centered cubic phase for PW and monoclinic/orthorhombic phase for NW respectively. The nanoflakes like morphology for NW NCs were confirmed by SEM. The MB dye was successfully degraded using NW4 NCs with a rate constant of 0.02266 /minute. The photovoltaic performance of the photoanodes with various weight percentages (wt%) of Ni was measured by recording the photocurrent-photovoltaic curves. The result indicated that NW4 NCs exhibited the best power conversion efficiency (PCE) with high short circuit current density (J_{sc}) and open-circuit voltage (V_{oc}). This enhanced photocatalytic and photovoltaic performance of NWNCs may be due to the visible light-harvesting, increased dye loading capacity and reduced photoelectron recombination.

1. Introduction

Dye-sensitized solar cells (DSSCs) have attracted tremendous interest due to their easy production, low fabrication cost, environmentally friendly and relatively high efficiency. Simultaneously the development of efficient photocatalyst has become the most emerging field of research which plays important role in energy and the environment. Tungsten oxide ($WO_3 \cdot H_2O$) DSSC is one of the most promising devices to convert solar energy into electrical energy [1, 2]. $WO_3 \cdot H_2O$ with indirect bandgap and semiconductor behavior has been widely studied for their photochromic, electrochromic and sensor property [3]. It absorbs visible light and suppresses photogenerated electron-hole pairs. By using different doped nanoparticles [4], one can enhance the photovoltaic and photocatalytic efficiency [5]. Ni-doped $WO_3 \cdot H_2O$ based DSSCs are responsible for the alteration of energy levels [6]. For the degradation of the organic dyes, the utilization of electron-hole pairs plays a major role. As NiO is a p-type material shows improved optical, electrochemical and stability properties [7]. Constructing n-p heterojunctions has been exposed as an effective method to improve both photocatalytic and photovoltaic properties. Upon the formation of n-p hetero junction, electrons are transferred from n-type metal oxide towards p-type nanoparticles, this results in additional electronic charge transfer between p-type nanoparticles supporting n-type metal oxide, modifying the width of the depletion zone and dramatically increasing the electrical conductance [8, 9, 10]. From previous studies, it was noted that adding Ni or Co as doping elements in WO_3 can increase photocurrent. In this record, p-type NiO and n-type WO_3 semiconductors can form heterostructured composites for fabricating nanodevice with enhanced performance.

To the best of our knowledge, this is the first work to report on this work, synthesis of Nickel tungstate (NW) NCs via a facile hydrothermal method, to tune the band structure and thereby to improve its performance as DSSC for the application of power conversion efficiency (PCE). The effects of Ni doping on the crystal structure, morphology, and chemical bonding of PW are investigated by XRD, FT-IR, FE-SEM,

UV-Visible Spectroscopy and electrical conductivity. The PCE of DSSCs based on NW NCs is optimized by varying the Ni doping to achieve the highest power conversion efficiency (PCE).

2. Experimental

2.1. Materials and Method

Sodium tungstate dihydrate ($\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$), citric acid ($\text{C}_6\text{H}_8\text{O}_7$) and hydrochloric acid (HCl), Nickel chloride (NiCl_2), Triton X-100, acetone ($\text{C}_3\text{H}_6\text{O}$), acetonitrile (CH_3CN), Iodine, SnO_2 : F transparent conductive glass (FTO, 2 cm x 1.5 cm, with sheet resistance 15 U/sq), sodium iodide (NaI), ethanol ($\text{CH}_3\text{CH}_2\text{OH}$) and dye are used as purchased.

The structural characterization was performed by using an X-ray diffractometer with (monochromatic $\text{CuK}\alpha$ radiation, $\lambda = 1.5406 \text{ \AA}$). The surface morphology of the prepared NCs was studied using Field-Effect Scanning Electron Microscope (EVO 18 SEM). The optical properties of synthesized samples were investigated using UV-visible spectrophotometer (JASCO UV-Vis V-770PC) in the wavelength range of 200–900nm. The electrical properties of synthesized samples were analyzed using a Keithley electrometer (6517-B). The photovoltaic properties of fabricated DSSCs were characterized by recording the computer-controlled Keithley electrometer (6517-B) with portable solar simulator PEC-L01 to collect the photocurrent density–voltage (I–V) curves under illumination conditions.

2.2. Synthesis of $\text{WO}_3 \cdot \text{H}_2\text{O}$ and $\text{NiWO}_4/\text{WO}_3$

$\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ and $\text{C}_6\text{H}_8\text{O}_7$ were dissolved in 20mL of deionized water to get a transparent solution of 1M solution. To this solution, 20mL of HCl aqueous solution was added dropwise to maintain the $\text{pH} \approx 1$, which in turn produce a light-yellow solution. Then the prepared reactant was transferred into a Teflon coated autoclave reactor and treated at 150°C for 6 hours. The solution was centrifuged and washed repeatedly with deionized water and ethanol followed by drying them at 60°C in air. Finally, the yellow color PW NPs was obtained. The dried sample was crushed using mortar and pestle and named PW. In another series, NW samples were prepared by mixing NiCl_2 [5, 10, 15, and 20 wt%] with $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ and $\text{C}_6\text{H}_8\text{O}_7$ by following the above procedure and it named NW1, NW2, NW3 and NW4 respectively.

2.3. Fabrication of Dye synthesized solar cell

The prepared PW and NW nanocomposites were individually mixed with ethanol, glacial acetic acid, and Triton X to form PW paste and NW paste. It is coated over a fluorinated (FTO) glass plate by the doctor blade technique with a defined area of 0.5cm^2 and calcined at 500°C for 30 min. The N719 dye (0.3 mM of ethanolic dye solution) was sensitized with PW and NW coated FTO substrates for 12 h. At present, platinum counters are used as commercial electrodes. The iodide electrolyte was prepared by dissolving 0.05 M of I_2 and 0.5 M of LiI in acetonitrile and injected between the photoanode and the counter electrode. Similarly, different NW photoanode were prepared by the same procedure. The DSSC's performance was measured with the help of Keithley electrometer under-stimulated light. The

photovoltaic characterization of the fabricate DSSC was measured under the illumination of a 150W Xenon light source from a solar simulator in combination with standard AM 1.5(85mWcm⁻²) [11].

2.4. Photocatalytic activity

Photocatalytic activities of synthesized NCs on methyl blue (MB) was evaluated by adding 0.002mg of NCs irradiated under visible light at different period. The pseudo-first-order rate constant reaction: $\ln(A_0/A_t) = K_{app} t$ was employed to study the degradation, where A_t is the absorbance of MB at time t, A_0 is the absorbance value at t = 0 and k is the pseudo-first-order rate constant.

3. Result And Discussion

3.1. Structural analysis of PW and NW NCs

Fig. 2 shows the X-ray diffraction pattern of PW and NW NCs. Eight characteristic peaks indexed to (002) (110) (220) (222) (260) (262) (440) (442) planes were observed for PW, with a high crystalline quality, which well-matched with standard JCPDS No: 72-0199 (face-centered cubic). The presence of NiWO₄/WO₃ was in good agreement with the standard JCPDS No: 15-0755 (monoclinic) and 72-0199 (orthorhombic). This result is astonishing since Ni²⁺ (0.69Å) has a layer ionic radius concerning W⁶⁺ (0.60Å) [12,13] and thereby substitution of Ni ions in W ions considerably increases the lattice constant. Using the Scherrer formula the average crystallite size for PW and NW NCs were calculated as 22-17nm. Due to the “mutual protective effect” between NiWO₄/WO₃ the crystallite size decreases and the microstrain (ϵ) increases with the increasing concentration of Ni in WO₃ [14]. The formation of oxygen vacancies in WO₃.H₂O upon Ni doping causes lattice distortion and decreases interplanar spacing. Because of the stoichiometric composition of doping, the peak has shifted towards the higher angle of diffraction for the NW3 and NW4 composite. This effect is mainly due to the difference in ionic radii between PW and NW NCs [15,16]. The decrease in crystallite size was observed with an increase in the W% of Ni. This is because of the reduction in the diffusion rate of NW NCs and also due to the distortion in the PW lattice by foreign impurities [17]. Moreover, the difference in the crystallite size can be attributed to the pH of the solution, solvent content, the solubility of the deposit, adhesion to the substrate, etc [2, 18]. This is well supported by the larger average values of dislocation density (δ) and microstrain (ϵ) which also increases the defect in the NW NCs and it may enhance the conductivity and photocatalytic activity in NW NCs [19,20].

Table 1. Structural parameters calculated for PW and NW NCs

Samples	Crystalline Size (D) [nm]	Micro strain (ϵ) $\times 10^{-3}$ [lines ⁻² m ⁻⁴]	Dislocation density	Stacking fault
			(δ) $\times 10^{-14}$ [lines cm ⁻²]	(SF) $\times 10^{-2}$
PW	38.959	0.0789	1.053	0.277
NW1	20.556	0.196	4.064	0.750
NW2	26.437	0.148	2.114	0.429
NW3	23.632	0.191	4.038	0.543
NW4	17.610	0.218	4.406	0.675

3.2. FTIR spectral analysis of PW and NW NCs

Fig.3 represents FTIR spectra of PW and NW NPs. The band around 3000-3500cm⁻¹ and 1600 - 1650 cm⁻¹ attributed to O-H stretching vibrations [21, 22]. The band observed from 800- 500 cm⁻¹ was due to W–O–O–W stretching vibration. The band at 780 cm⁻¹ corresponds to Ni-O stretching [23]. The FTIR investigation confirms that PW nanoplates have a lower amount of surface OH groups than NW4 NCs. The photocatalytic activity has improved by the charge transfer of photo-generated holes of the hydroxyl group in the surface [24].

3.3 Tauc plot of PW and NW NCs

In Fig.4, the bandgap (E_g) value was calculated using the Tauc plot for the PW and NW NCs from the given relation, From Fig.4 the bandgap values were found in the range of 2.9eV to 2.5eV. The narrowing of the bandgap is due to the influence of electron transition between the valence and conduction bands [25]. Moreover, decreasing the optical band gap of higher concentrations of Ni in NW4 suggested that the presence of Ni strongly influenced the optical parameters of the NW composites. The reduction in E_g which forms an acceptor level led to redshift denotes the localized nature of Ni species in PW lattice contributing to the creation of surface oxygen vacancies [26,27].

3.4. FE-SEM analysis of PW and NW NC

Fig.5 shows FE-SEM images of PW are nanoplate-like structures and NWNCs are nanoflake structures. Due to the increase in the concentration of Ni, plate-like structures are transformed into agglomerated nanoflakes. Compared to plate-like structures, nanoflakes have a large surface area which results in enhanced dye absorption and hence facilitates an enhanced photocurrent [28]. The edges of NW4 nanoflakes are curly and twisted which form a porous structure with each other. This porous structure will help to absorb the dye and enhance photocatalytic activity [29].

3.5 DC electrical conductivity

The dc electrical conductivity (σ_{dc}) and activation energy (E_a) was calculated by using following relation

$$\sigma_{dc} = t/RA \quad (2)$$

$$E_a = \text{slope value} \times (K_B / e) \text{ eV} \quad (3)$$

Where t is the thickness of the interfacial layer, R is the series resistance of the structure and A is the rectifier contact area, K_B is the Boltzmann constant and V is the applied voltage.

The calculated conductivity is found to vary in the range ($2.5 \times 10^{-8} \text{ eV}$) to ($3.3 \times 10^{-7} \text{ eV}$) for different concentrations listed in Table 2. The NW4 NCs showed maximum conductivity, $3.3 \times 10^{-7} \text{ eV}$. Due to the oxygen vacancies in the grown semiconducting nature of the NCs, the conductivity reaches maximum. [30]. As the Ni concentration increases, the average activation energy values decrease suggesting the fact that the Ni atom could have created some impurities in the bandgap of $\text{NiWO}_4/\text{WO}_3$ NCs. Moreover, the increase in the oxygen vacancies is mainly due to the presence of Ni clusters in the WO_3 lattice [31]. Similar behavior of decreasing E_a values with increasing dopant concentration was reported [32,33]. The above results confirm the stability of PW and NW NCs and their employability in optoelectronics devices.

Table.2. The variation of average conductivity and activation energy of PW and NW NCs thin film

Samples	Avg. Conductivity $\sigma(=1/\rho)$ (S/cm)	Avg. Activation energy (eV)
PW	2.49647×10^{-8}	0.16909
NW1	3.47219×10^{-8}	0.12245
NW2	1.34253×10^{-7}	0.09941
NW3	2.03321×10^{-7}	0.09451
NW4	3.33903×10^{-7}	0.08824

3.6 Photoluminescence

The PL spectrum is a vital technique to study the surface processes involving photogenerated electron-hole pairs. This study has been widely used to find out the efficiency of charge carrier trapping, migration, and to understand the pairs of electron-hole in semiconductors. In Fig. 7, the higher intensity of the green emission peak was observed at 529nm and 532 nm. The wider and higher intensities are usually assigned to the deep level of emissions due to oxygen vacancies, and anionic or cationic interstitial defects for NW NCs [34, 35]. Higher intensity of NW4 NCs leads to a lower recombination rate of photo-generated electrons and holes [36]. In which the optical response of the material is largely determined by its underlying electronic properties that are closely related to its chemical, atomic arrangement, and physical dimension for nanometer-sized materials. It's also noted that the PL emission peak slightly

shifts towards a higher wavelength (redshift). This may be due to the increase in Ni^{3+} ions which act as a conducting center and reduces the E_g [27]. From the PL emission spectra, the addition of NW4 NCs indicates an effective carrier extraction and reduced recombination [37]. Thus it will help to improve J_{sc} and the performance of DSSC. Moreover, the grain size of the DSSC could make an important attribution to PL spectra and the device performance.

3.7 Photocatalytic activity

Fig .8 shows the rate of decomposition of methyl blue by PW and NW4 NCs under visible light irradiation. Pseudo-first-order rate constant reaction: $\ln (A_0/A_t) = K_{app} t$ was employed to study the degradation, where A_t is the absorbance of MB at time t , A_0 is the absorbance value at $t=0$ and k is the pseudo-first-order rate constant. NW4 NCs photocatalyst exhibited a high response in the visible light region. In general, the photocatalytic efficiency depends on the consequence of photo-generated hole-electron pairs in the photocatalytic process of semiconductors under light irradiation [38]. The photo-generated electrons in CB of NW4 NCs can quickly retransfer to the surface to participate in the photocatalytic reaction, thus decrease the recombination probability of photo-generated electrons and holes, suggesting that the introduction of Ni ions into WO_3 can induce little more trap-state, which can further reduce the recombination rate [39]. The photo-generated electrons and holes react with the surface substances adsorbed, O_2 , OH , etc to form reactive species such as O_2 , OH^* (hydroxyl radical), which is major oxidative species for the decomposition of organic pollutants [40]. Then the oxidative species degrade the organic pollutant MB into small molecules like CO_2 , H_2O , etc. To prevent the recombination of photo-induced electron-hole pairs, structure, size, and E_g play an important role [27]. The K_{app} value of PW and NW4 NCs is 0.02249 and 0.02266 respectively. Due to the larger E_g , PW shows a less degradation rate constant. This was improved with narrow E_g of NW4 NCs, which supports the better photocatalytic activity. Moreover, the substitution of Ni reduces the recombination rate of the electron-hole pair due to charge compensation between Ni and W. This may be due to the creation of oxygen vacancies that can act as oxidizing species in enhancing photocatalytic activity [26].

3.8 Photovoltaic performance of DSSCs

The light-harvesting activity of a dye molecule is a key parameter that determines the ability of solar energy capture and in turn affects the photocurrent generated by solar cells.

$$FF = (J_{max} \times V_{max}) / (J_{sc} \times V_{oc}) \quad (4)$$

$$\eta(\%) = ((FF \times J_{sc} \times V_{oc}) / (P_{in})) \times 100 \quad (5)$$

Fig.9 shows the J-V characteristic curve of the DSSCs under simulated sunlight illumination. Their corresponding photovoltaic parameters were calculated and given in Table 3. From Table 1 it was obvious that the short circuit current density (J_{sc}), the open-circuit voltages (V_{oc}), Fill factor (FF) and the

photo electrolytic cell efficiency (η), enhanced gradually with an increase in Ni content. The photovoltaic parameters reveal that the cell efficiencies increased from 2.48% to 3.49% for PW to NW4.

Table. 3 Photovoltaic parameters of the synthesized sample.

Samples	J_{sc} (mA/cm ²)	V_{oc}	FF	η (%)
PW	7.4	0.8	0.42	2.48
NW1	7.3	0.8	0.49	2.91
NW2	7.3	0.8	0.51	2.94
NW3	7.5	0.8	0.55	3.30
NW4	7.5	0.8	0.58	3.49

In addition, reduced recombination of NW NCs acts as good electron transfer. The photoanode generates small crystallites on the surface of the NCs, thereby increasing the adsorption of dye molecules and leading to the generation of more photoelectrons and light-harvesting- capacity [41]. The enhanced light absorption of NW4 electrode-based DSSC to the visible region is due to Ni doping, wherein J_{sc} and FF of NW4 electrode get increased [41]. Here improved J_{sc} is due to the higher chemical capacitance of NW4 and higher FF reveals reduced recombination kinetics in NW-based DSSCs [42]. The adsorption of carboxyl groups in the N719 dye molecule leads to increased dye loading capacity and enhanced cell performance [43]. NW4 NCs are responsible for better photovoltaic properties for lower recombination rate, superior light-harvesting capability, surface area prolonged charge transport pathway-based device [15]. The increment in Ni concentration improves the electrochemical performance of PW, which confirms the feasibility of their role in DSSC. The maximum PCE obtained for NW4 DSSC is 3.49% which may be due to its higher incorporation of Ni concentration in PW.

Conclusion

PW and NW NCs were synthesized by the hydrothermal method. Powder XRD studies confirmed the formation of NiWO₄/WO₃ NCs. The particle size decreases while NW NCs. A large amount of hydroxyl group on the particle surface moreover contributes to improving the photocatalytic activity supported by the FTIR study. UV-V is spectra indicate that the bandgap becomes narrow due to Ni substitution in PW. SEM result confirms the nanoflakes morphology of PW. The Conductivity increased and E_g decreased while Ni is doped in PW NCs. PL emission peak act as conducting center and reducing the energy band gap observed. NW NCs intense of the dye absorption in surface area, increase the power conversion efficiency because interactions with photogenerated giving better charge transfer, electron-hole pair inhibits moreover supported by the photovoltaic study.

Declarations

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Figures

Figure 1

The schematic diagram for DSSC

Figure 2

X-ray diffraction patterns of PW and NW NCs

Figure 3

FTIR Spectra of PW and NW NCs

Figure 4

Tauc plot of PW and NW NC $(\alpha h\nu)^{1/2} = A(h\nu - E_g)(1)$

Figure 5

FE-SEM images of PW and NW NCs

Figure 6

The average conductivity and average activation energy plot of PW and NWNCs.

Figure 7

Photoluminescence spectra of PW and NW NCs

Figure 8

Rate of decomposition of MB using visible light PW and NW NCs

Figure 9

I-V characteristic curves of the cells made by synthesized samples.