Materials Research Innovations

ISSN: 1432-8917 (Print) 1433-075X (Online) Journal homepage: https://www.tandfonline.com/loi/ymri20

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To cite this article: Seyed Hossein Hosseini, Ali Azimi & M. Boujar Dolabi (2019): Study of UV-Visible and near infrared absorption CsXWO3/polypyrrole nanocomposite, Materials Research Innovations, DOI: 10.1080/14328917.2019.1677302

To link to this article: https://doi.org/10.1080/14328917.2019.1677302

Published online: 16 Oct 2019.

MATERIALS RESEARCH INNOVATIONS
https://doi.org/10.1080/14328917.2019.1677302

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Study of UV-Visible and near infrared absorption CsXWO3/polypyrrole nanocomposite

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ABSTRACT

Cs_xWO_3 nanoparticles were successfully synthesised via solvothermal and its nanocomposites with polypyrrole and different thicknesses were prepared via interfacial polymerisation. The UV-Visible and near-infrared (UV-Vis-NIR) of Cs_xWO_3 products were studied. The Cs_xWO_3 and its nanocomposites characterised by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectrometer (EDX) and FTIR. The results indicated that the NIR absorption property of $Cs_{0.3}WO_{2.7}$ is better than $CsWO_3$ and increased by increasing thicknesses.

ARTICLE HISTORY
Received 22 July 2019
Accepted 1 October 2019

KEYWORDS
Near infrared; UV-visible; absorber; nanocomposite; Cs_xWO_3; polypyrrole

Introduction

Hexagonal tungstate oxide nanoparticle (NPs) has received much attention because of its tunnel structure [1]. Guest ions such as Na^+, NH_4^+ reside in the hexagonal window tunnels between layers and can be exchanged by K^+, Cs^+ and others [2]. Alkali-doped tungsten bronze, M_xWO_3 is composed of the arrays of octahedral unit WO_6 interconnected with their corners as is the case with tungsten trioxide ionic and electronic conductivities, which property has prompted studies seeking possible applications to sensors and superconductivity in Na_xWO_3 [3] and Cs_xWO_3 [4]. The effects of processing parameters such as solvent, autoclave rotating and Cs source on the morphology and microstructure of Cs_xWO_3 products were investigated by Liu and co-workers [5]. The results showed that autoclave rotating during the reaction and using ethanol as a solvent are helpful for decreasing the particle size and obtaining pure hexagonal Cs_xWO_3 crystals. The tungsten bronzers (M_xWO_3) with different ions were synthesised and their electrochromic, near-infrared (NIR), nanostructure electrical-optical properties and photothermal oblation cancer trapped were studied, too [6–9]. Recently NIR shielding materials such as $BaTiO_3$ and Cs_xWO_3 have received considerable attention in the development of thermal IR shielding transparent and solar heat shielding for control window of automobiles and architectures [10,11]. The strong absorption in the NIR region, owing to the free electrons was obtained. They also might be efficient as a photosensitiser in NIR photothermal therapy [12]. There has been a strong desire to shield on the window of automobiles, and building [13] in order to reduce the energy consumption. For application as a heat ray shielding materials, excellent shielding ability of NIR rays as well as high visible light transparency is required. The interfacial preparation of polypyrrole (PPy) is a new and powerful method for synthesis of the soluble PPy. This advantage is due to the ease of preparation, nanorod products and its fast reaction [14]. The preceding researches, we reported some of new a nanocomposites based conducting polymers [15–18]. We have synthesised new compounds showed that suitable nanocomposites as multi absorption in both TIR and MW [10,19]. In this work, caesium tungsten bronzes were successfully synthesised by solvothermal conditions. The Cs_xWO_3/PPy nanocomposites are prepared by interfacial polymerisation, too. The research results were studied for both UV-Vis (200–800 nm) and NIR (780–2400 nm) absorptions.

Experimental

Materials

Tungsten hexachloride (WCl_6) and caesium hydroxide ($CsOH.5H_2O$) and benzyl alcohol solution and purchased form Merck. Pyrrole monomer (analytical grade, Merck) distilled twice under reduced pressure and stored blew 0 °*C*. All the other chemical reagents were purchased from Merck without further purification.

Preparation of $CsWO_3$

Caesium tungsten bronzes, Cs_xWO_3 were successfully prepared by solvothermal reaction method. Using tungsten hexachloride (WCl_6) and caesium hydroxide ($CsOH.5H_2O$) as raw materials and benzyl alcohol solution as a reaction solvent. The initial concentration of WCl_6 0.7 g (0.0017 mol) in the 30 mL (0.289 mol) benzyl alcohol solution was adjusted and the nominal atomic ratio of Cs/W was designed. The solvothermal reaction was conducted at 200 °*C* in 14 h for $CsWO_3$ and 240 °*C* in 18 h for $Cs_{0.3}WO_3$ in an electric oven, with the reaction solution in a Teflon-lined autoclave 150 mL of internal volume. The obtained solid precipitates were then centrifuged and washed with water and ethanol a few times and followed by vacuum drying at 60 °*C* overnight.

Preparation of Cs_xWO_3/PPy nanocomposites via interfacial polymerisation

The synthetic procedure involves interfacial polymerisation of pyrrole in a water/chloroform interface with pyrrole

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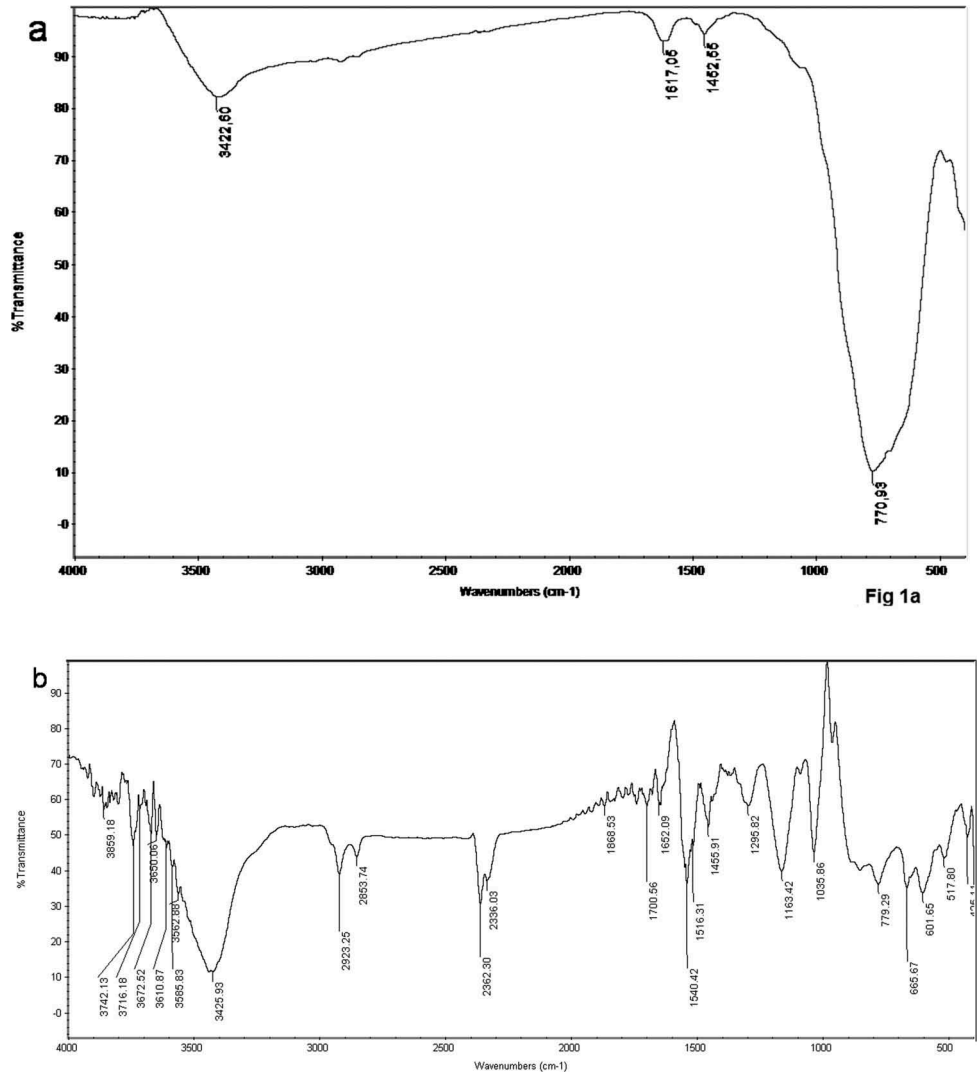

Figure 1. FTIR spectrum of a) Cs_xWO_3 NPs b) Cs_xWO_3/PPy nanocomposite.

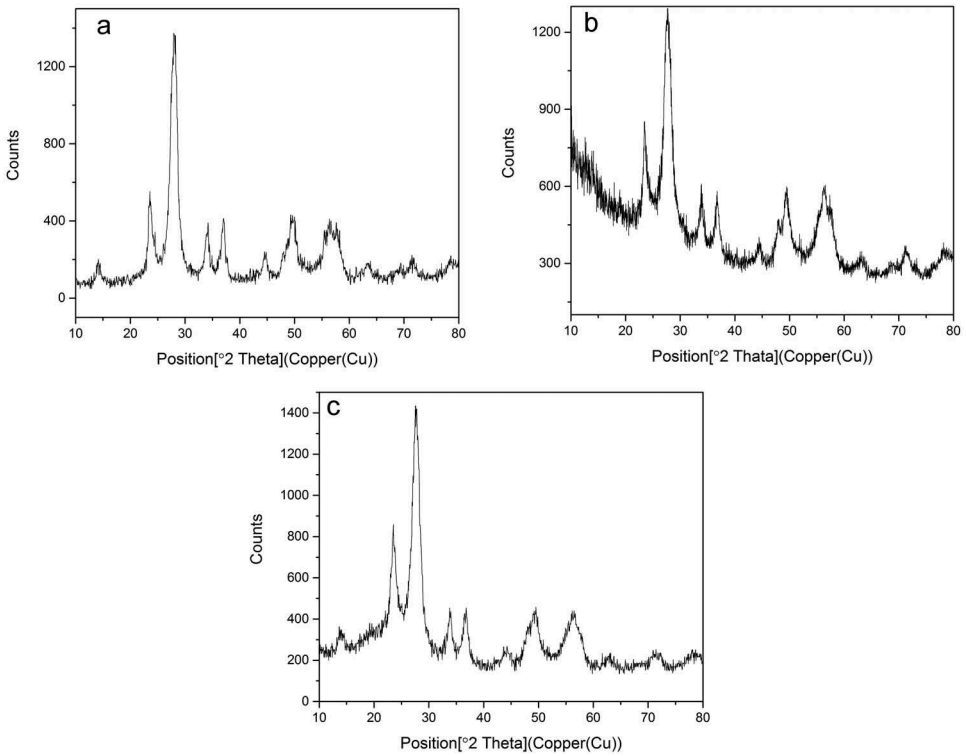

Figure 2. XRD patterns for a) $CsWO_3$, b) $Cs_{0.3}WO_{2.7}$ and c) $Cs_{0.3}WO_{2.7}$/PPy.

monomer and Cs_xWO_3 added in the organic phase, while $Fe(NO_3)_3.9H_2O$ in the aqueous phase. The aqueous solution was slowly added to the organic medium. In a typical preparation, a solution of 6 g (0.045 mol) $Fe(NO_3)_3.9H_2O$ in 35 mL of H_2O were slowly spread to 35 mL CH_3Cl containing 1 mL (0.015 mol) of pyrrole monomer and 0.8 g of Cs_xWO_3. As the reaction proceeded, without stirring a black film appeared in the interface. After 24 h reaction time, the Cs_xWO_3/PPy nanocomposites film was removed and washed carefully by ethanol and acetone and dried at 70 °C.

Characterisation

The ultrasonic experiment was carried out by an ultrasonic disperser (Hielsche, UP4005, Germany). Field emission scanning electron microscopy (FESEM) was performed by TESCAN

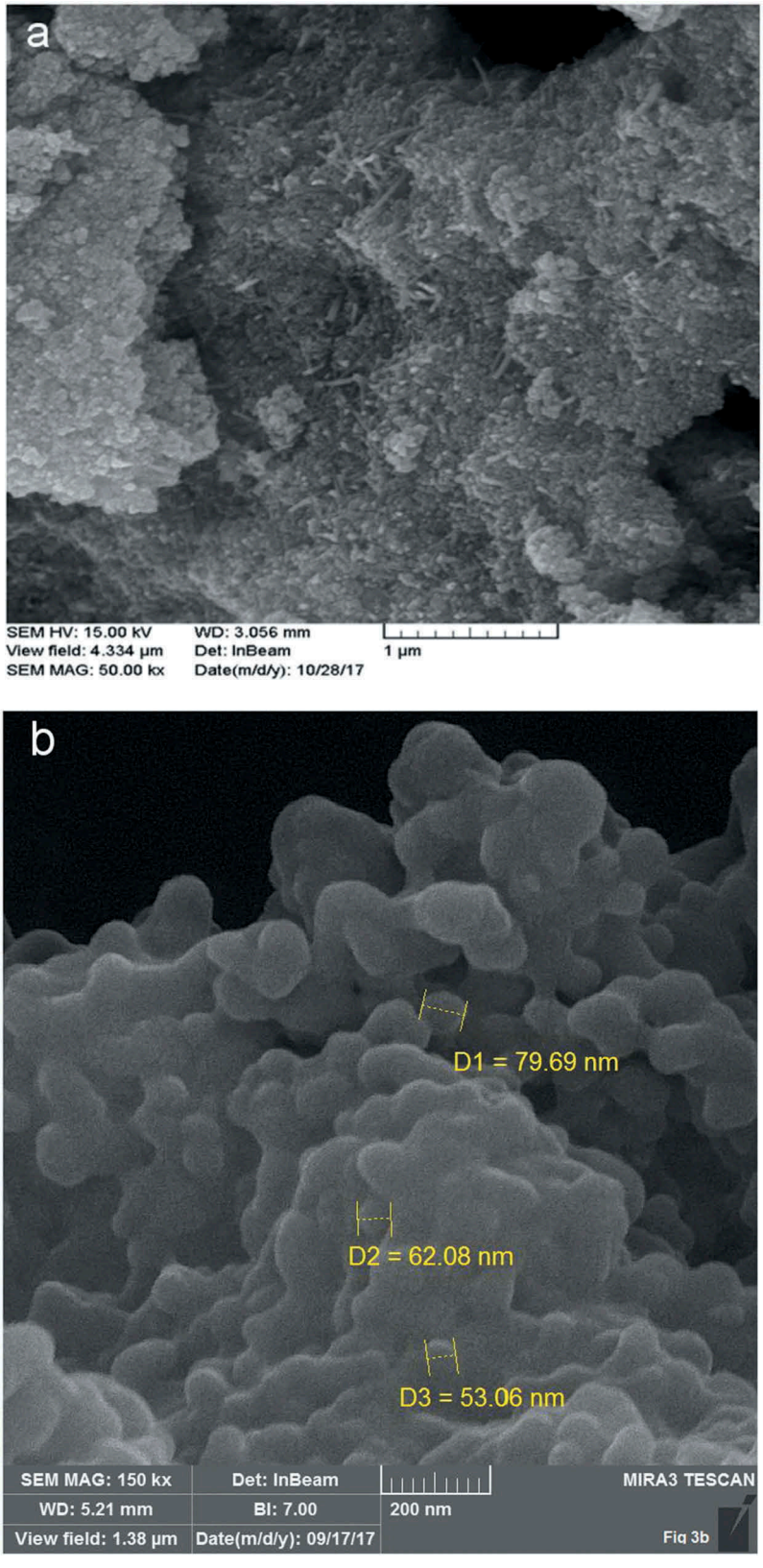

Figure 3. FESEM images of a) $Cs_{0.3}WO_{2.7}$ NPs and b) $Cs_{0.3}WO_{2.7}$/PPy nanocomposite.

MIRA to observe surface morphologies of samples. The XRD patterns of the samples were collected on a Philips-PW 1800 with Cu K_α radiation (λ = 1.54184 Å) in the 2θ = 4–90° with steps of 0.02°, scanning operated at 40 kV and 30 mA (Netherland). The UV-Vis-NIR of the samples was collected on a Analytik jena Specord 250 by ASTM E1331 standard.

Results and discussion

FTIR study

Figure 1(a,b) shows the FTIR spectrum of Cs_xWO_3 NPs and Cs_xWO_3/PPy nanocomposite. As Figure 1(a) illustrates, the band at 770 cm^{-1} is related to special peak for Cs_xWO_3 NPs. As observed in Figure 1(b), the band at 3425 cm^{-1} can be attributed to N-H stretching vibrations of PPy. In addition, the specific peaks around at 1540 and 1455 cm^{-1} are attributed with vibrational modes of quinonic and aromatic type ring for PPy. The peak at 1163 cm^{-1} is related to C-N stretching vibrations for PPy and we can see special peaks for Cs_xWO_3 at 600–700 cm^{-1}.

XRD patterns

Figure 2(a–c) shows XRD patterns for a) $CsWO_3$, b) $Cs_{0.3}WO_{2.7}$ and c) Cs_xWO_3/PPy, respectively. Its standard correspond to JCPDS file no 98-000-4845. All peaks correspond to characteristic peaks of hexagonal type lattice. This result indicated that the increasing temperature changed $CsWO_3$ (green colour) to $Cs_{0.3}WO_{2.7}$ (blue colour). The average crystallite size calculated by debye sherrer formula $\beta = k\lambda/D\cos\theta$, where λ is the wavelength of Cu K_α radiation (0.1541 nm) and the value of K depends on several factors, including the miller index of reflection plan and the shape of the crystal. If the shape is unknown, K is often assigned a value of 0.89, D is average crystallite size, θ is the Bragg's angle, and β is fall width at half maximum of the diffraction peaks. From the obtained peak width of XRD patterns Figure 2(a–c). So, the average crystallite sizes of $CsWO_3$, $Cs_{0.3}WO_3$/PPy calculated as 20.71, 20.68 and 20.41 nm, respectively.

FESEM images

Figure 3(a,b) shows FESEM images of $Cs_{0.3}WO_{2.7}$ NPs and $Cs_{0.3}WO_{2.7}$/PPy nanocomposite. The diameters of NPs and nanocomposite are about 34 and 80 nm, respectively. So, PPy shell is about 45 nm. Figure 3(a) shows NP is clear and uniform and surface of SEM image of nanocomposites is spongy and uniform, too.

EDX pattern

Figure 4 shows the typical EDX of Cs_xWO_3/PPy that Cs_xWO_3 synthesised by solvothermal method for using CsCl as Cs source. The Cs/W atomic ratio of NPs based on EDX analysis is 0.3, which is close to the theoretical maximum of 0.33 [5,20]. In addition, EDX pattern proved existence of NPs in nanocomposite structure for 35% weight ratio.

UV-Vis-NIR spectra analysis

Figure 5 shows the UV-Vis-NIR reflection spectra of $CsWO_3$ and $Cs_{0.3}WO_{2.7}$ plates synthesised by the solvothermal reaction, in different thicknesses. A typical UV-Vis absorption bands (200–800 nm) and NIR-absorption bands (780–2400 nm) of Cs_xWO_3 NPs can be clearly seen for all samples. The former is due to the band gap and the latter is correlated with the free carrier concentration and polarons of the Cs_xWO_3 products [5]. It should be noted that the reflectance peak position of $Cs_{0.3}$

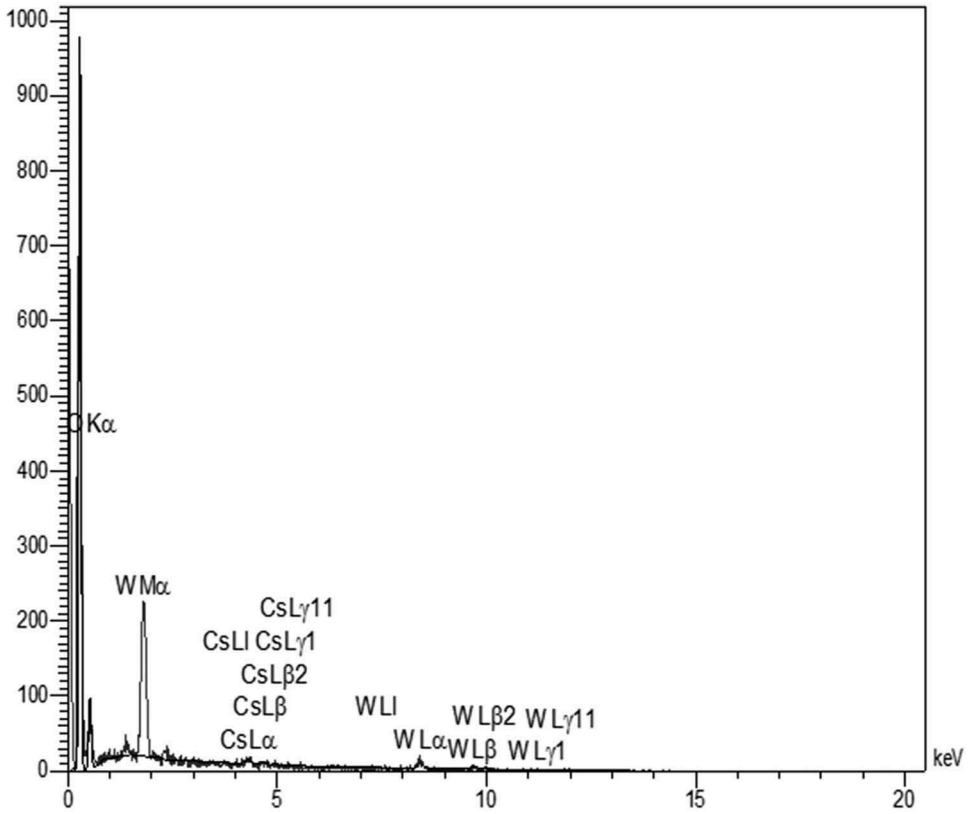

Figure 4. EDX image of Cs_xWO_3/PPy.

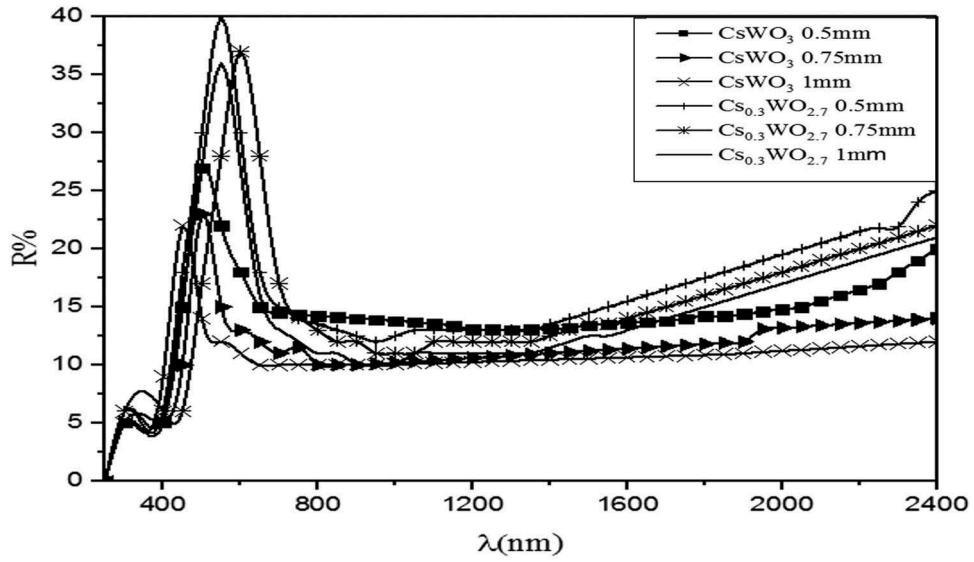

Figure 5. The UV-Vis-NIR reflection spectra of $CsWO_3$ (a) and $Cs_{0.3}WO_{2.7}$ (b) plates synthesised by solvothermal reaction under different thicknesses (0.5 mm, 0.75 mm and 1 mm).

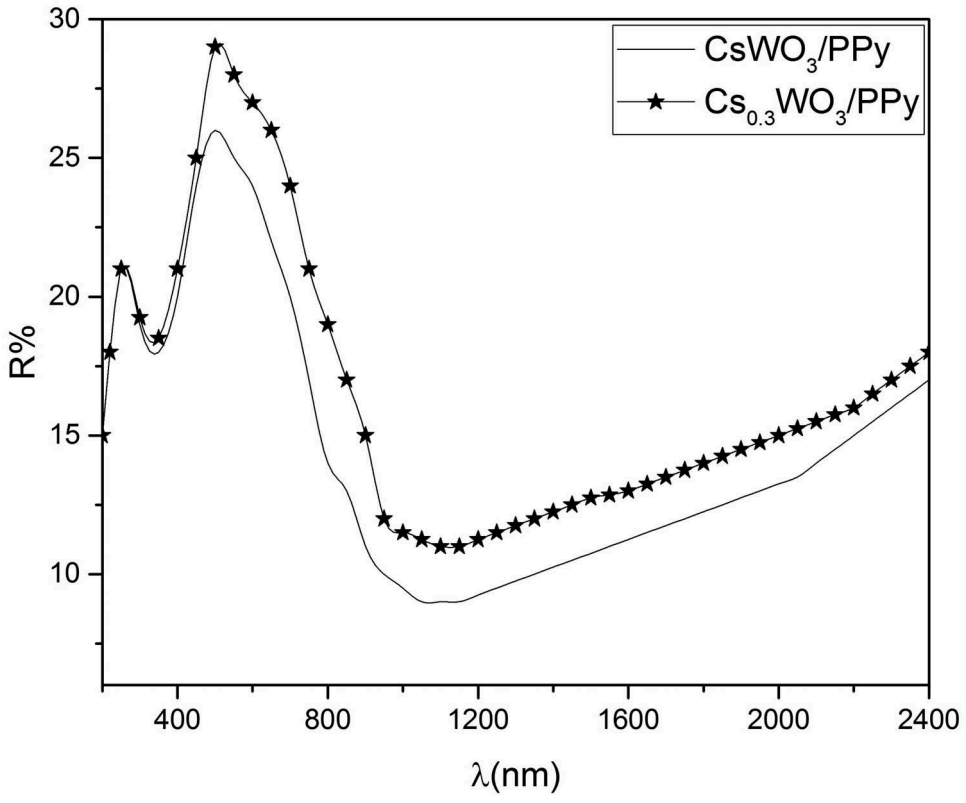

Figure 6. The UV-Vis-NIR reflection spectra of $CsWO_3$/PPy and $Cs_{0.3}WO_{2.7}$/PPy nanocomposites prepared by interfacial polymerisation in 1 mm thickness.

$WO_{2.7}$ in higher thicknesses shifts towards the higher wavelength and low reflectance, which indicates different free carrier concentrations. In addition, $Cs_{0.3}WO_{2.7}$ with more thicknesses exhibit higher NIR absorption (above 1400 nm) than the low thicknesses. Figure 6 shows the reflectance spectra of $Cs_{0.3}WO_{2.7}$/PPy nanocomposite with 1 mm thickness. Due to a lack of free electrons, the fully oxidised compound Cs_xWO_3 showed no NIR shielding and after calcination at autoclave, all the Cs_xWO_3 type products a certain degree of shielding performance of the visible and NIR were increased of PPy. The Cs_xWO_3/PPy showed the best performance as UV-Vis as well as excellent shielding properties in the NIR region.

Conclusions

The caesium tungsten oxides were successfully synthesised by solvothermal and Cs_xWO_3/PPy nanocomposites with about 80 nm diameter were prepared by interfacial polymerisation of pyrrole. FESEM image shows the formation of spongy shape and EDX diagram shows the incorporation of Cs_xWO_3 (x = 0.3) into the polymer. The reflectance spectra result showed that the best UV-Vis-NIR shielding is $Cs_{0.3}WO_{2.7}$/PPy with 1 mm thickness. Such a nanocomposite not only could be used in the transparent solar heat-shielding filters, but also is useful for the development of UV-Vis-NIR shielding materials in medicine and military application.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Seyed Hossein Hosseini is full Professor of Organic Chemistry and Polymer Science at Imam Hossein and Islamic Azad Universities, Tehran-Iran. His was born in 10 September 1965. His main research interests cover conducting polymers (synthesis and applications) focus are design of new materials such as toxic gas sensors, conductive liquid crystalline polymers. The recent research activities include electromagnetic absorbing materials such as microwave, thermal IR, UV and laser, X-neutron-gama-ray, and etc. for industry and military applications. He has received numerous awards and he has more than 100 research publications in International journals. His main industrial researches are about cold cracking on extra heavy oil-bitumen, and industry resins.

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