Research Square

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Fabrication of Nanofibers from a novel chalcone material by Electro Spinning Generator

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Abstract

High-quality fibers were fabricated from 1-(4-methylsulfonyl phenyl)-3-(4-N, N dimethyl (amino phenyl)-2-propen-1-one (MSPPP) oxide on well-cleaned glass substrates by a simple electrospinning generator. The surface morphology, crystalline structure, composition, thermal stability, and optical properties of the prepared fibers with annealed temperatures 400, 500, and 600°C were studied using Field emission scanning electron microscopy (FESEM), High-resolution transmission electron microscopy (HRTEM), X-ray diffraction, energy dispersive X-ray analysis (XRD), Thermogravimetric analysis (TGA), Photoluminescence, and Ultraviolet–visible spectroscopy characterization techniques. The nanoscale diameter of fibers from10 to 300 nm, and covers the entire length of the collector, and is continuous almost straight, and defect-free. The chemical composition of fibers confirms the1-(4-methylsulfonylphenyl)-3-(4-N, N dimethyl (amino phenyl)-2-propen-1-one oxide crystallization starts around 220°C and the complete removal of Polyvinyl acetate at 500°C. The absorption edge of the1-(4-methylsulfonyl phenyl)-3-(4-N, N dimethyl (amino phenyl)-2-propen-1-one nanofibers is nearly 3.5 eV. The nanofibers of the1-(4-methylsulfonyl phenyl)-3-(4-N, N dimethyl (amino phenyl)-2-propen-1-one oxide can be used as an excellent UV emitter, and touch screens Transparent Conducting Oxide, and they will be excellent material for nanodevices fabrication.

1. Introduction

In recent years, the development and usage of touch screens in all electronic devices is the default option and are more user-friendly in nature. The screen should be transparent to light and conduct electric currents. Many sensors, solar cells, etc., are using Transparent Conducting Oxide (TCO) [1–2]. In this way, the1-(4-methylsulfonyl phenyl)-3-(4-N, N dimethyl (amino phenyl)-2-propen-1-one (MSPPP) oxide is one of the excellent conducting oxides, due to this high degree of transparency in the visible region, low operating temperature, high thermal stability, and robust physical and chemical interaction with adsorbed species. Most thin film researchers find numerous problems in the fabrication of metal oxide on glass plates using non-vacuum techniques. The Physical Vapor Deposition (PVD) method includes many stages such as evaporation, transportation, and condensation of materials in the vacuum environment [3]. Many features such as self-assembly, defect-free, nanometer size diameter, and well interconnecting of fibers, can be achieved when the non-vacuum technique, especially the electro-spin coating technique, was used [4–5]. Thus, the research on the fabrication of MSPPP oxide by electrospinning technique and characterization is the targeted task for us and introduces a new breakthrough in transparent conducting oxide among semiconductor researchers.

2. Materials and methods

2.1. Materials

Experimental details

The synthesis of a novel compound 1-(4-methylsulfonyl phenyl)-3-(4-N, N dimethyl (amino phenyl)-2-propen-1-one (MSPPP) $C_{18}H_{19}NO_3S$ (Fig. 1) was reported [6]. Single crystals of MSPPP were grown from acetone by slow evaporation technique at room temperature. A saturated solution of MSPPP was obtained by dissolving the synthesized material in acetone with continuous stirring at room temperature. This saturated solution was filtered using Whatman filter paper and the solution was transferred to a beaker, and the solvent was allowed to evaporate slowly. After 6 days, transparent thin plates like crystals were successfully harvested. The image of the MSPPP crystal was recorded on Euromex optical microscope equipped with an optical polarizer and CMEX digital camera (Fig. 2).

Figure 2 Optical microscope image of MSPPP crystal

The 3 g purity tested (M_w 85,000-124,000, 99% Sigma Aldrich) Polyvinyl acetate (PVA), and 6 g MSPPP were dissolved with 40 ml triple distilled water in two separate beakers, and then the solutions were mixed in three different ratios 1:1, 1:2 and 1:3 in three separate beakers using magnetic stirrer for three hours at room temperature. The mixtures were stored for one day aging period at room temperature to attain high viscous state for this sol-gel technique [7]. The prepared precursor solution was filled with no air bubbles in the syringe. Well cleaned glass plates were placed in the collector setup which is 15 cm from the tip of the needle. The precursor solution loaded syringe was fitted in the syringe holder setup and aligned to be in the same horizontal axis with the shaft of the stepper motor. The variable electro-static high voltage of (0–30 kV) was applied across the syringe needle and metal collector terminal. The high voltage applied between the needle and metal holder was used to extract high surface tension across the needle tip and collector. Due to the forward movement of piston and applied high voltage, the Taylor cone was formed (without decomposition of PVA and MSPPP) and made as nanofibers. The composite nanofibers of PVA and MSPPP were spawned on the cleaned glass substrate (mounted on collector holder). The nanofibers deposited in glass substrates were collected labeled and preserved in desiccators then and there. The pure MSPPP Oxide nanofibers were obtained by calcining the deposited glass substrates at 400, 500 and 600°C in a muffle furnace for 1 h to remove the organic polymer components in the samples and to crystallize MSPPP oxide. The cost-effective microcontroller aided electro-spin coating unit is shown in Fig. 3.

3. Results and Discussion

3.1 Structural Properties

Figure 2 shows the powder XRD patterns of MSPPP oxide nanofibers. The peaks at 2θ values 26.59°, 33.94°, 38.01°, 44.24°, 51.85°, 54.79°, 57.82°, 62.06°, 64.64°, 66.11°, 71.48°, and 78.92° correspond to the lattice planes of (1 1 0), (1 0 1), (2 0 0), (1 1 1), (2 1 1), (2 2 0), (0 0 2), (3 1 0), (1 1 2), (2 0 2), (3 2 1) respectively. All the peaks in the XRD pattern of MSPPP oxide are shown in Fig. 4. The lattice parameters ‘a’ and ‘c’ of the MSPPP oxide nanofibers are determined from the peaks of the XRD patterns. The calculated values of ‘a’ and ‘c’ of MSPPP oxide nanofibers are 4.8133 Å (a), and 3.2900 Å (c). The values of the different crystal parameters are listed in Table 1.

Table 1
Crystal size, lattice parameter, strain, and dislocation density of MSPPP oxide thin films prepared

Annealed Temperature [^{0}C]	Miller indices			Crystal size [nm]	Lattice constant [D]		Strain [$\times10^{-7}$ lines^{-2} m^4]	Dislocation Density[d] [$\times10^{-7}$ lines^{-2} m^2]
	h	k	l		a [Å]	c[Å]		
400	1	1	0	13.65			2.300	4.600
	1	0	1	11.59	4.75	3.16	4.300	6.000
	2	0	0	13.44			7.700	4.300
500	1	0	1	14.78			2.100	2.100
	1	0	0	12.86	4.81	3.20	3.200	5.000
	0	0	2	15.21			4.200	2.300
600	1	0	1	21.72			2.000	5.300
	1	0	0	14.08	4.88	3.21	2.400	7.400
	0	0	2	20.72			2.500	5.300

Crystal size, lattice parameter, strain, and dislocation density of MSPPP oxide thin films prepared at 400, 500 and 600°C [8]:

$$\mathbf{D} = \frac{0.9\varvec{\lambda}}{\varvec{\beta}\cos\varvec{\theta}} \quad (1)$$

The strain (ε) was calculated from the slope of β cos θ versus sin θ plot by using the relation [9]:

$$\varvec{\beta} = \frac{\varvec{\lambda}}{\mathbf{D}\cos\varvec{\theta}} + \varvec{\epsilon}\tan\varvec{\theta} \quad (2)$$

The dislocation density (δ) was determined from the relation.

$$\varvec{\delta} = \frac{1}{\mathbf{D}^2} \quad (3)$$

3.2 SEM, TEM and EDX Analysis

The size and external morphology of the prepared nanofibers have been examined by FESEM and HRTEM. Figure 5a, b denotes FESEM images of PVA and MSPPP composite nanofibers and (c, d) illustrates MSPPP oxide nanofibers.

Figure 6 shows TEM images of nanofibers of PVA and MSPPP prepared at 400°C. It is clearly shown that the MSPPP oxide with PVA nanofibers are distinct with slight variation of diameter (± 5 nm) from Fig. 5a, b, upon annealing the MSPPP oxide nanofibers are formed with uniform size and unique characteristics.

The chemical composition of the fabricated nanofibers was explored by EDAX analysis. Figure 7 portrays the classic EDAX spectra of the nanofibers. The EDAX investigation of nanofibers displays the S and O elements only. The molecular formula of the fiber is identified as MSPPP oxide (Table 2).

Table 2
UV-Vis Optical properties of MSPPP oxide nanofibers

Annealed temperature [°C]	Band gap [Eg eV]
400	3.51
500	3.49
600	3.46

3.3 Thermal Analysis

Thermal analysis of a material gives useful information regarding the thermal stability of that material [9]. Thermal gravimetric analysis (TGA) and Differential Scanning Calorimeter (DSC) are most important as far as fabrication technology is concerned as they provide thermal stability of the material for fabrication where a considerable amount of heat is generated during the cutting process. Thermal stability of the nanofibers has been studied by TGA from 27 to 800°C. The complete thermal behavior of the nanofibers with time and temperatures is shown in Fig. 8.

It indicates different main weight losses taking place in the thermogravimetric (TG) characteristics curve. Due to the loss of the residual water molecules in the precursor composite fibers, 7% of weight loss occurred in the range 30–175°C. The first endothermic peak around 115°C appeared in the Differential Scanning Calorimeter (DSC) curve. The range between 200 to 400°C, 27% of weight loss appeared due to loss of volatile components. The breaking of carbon–carbon (–C–C–) bonds of the main structure of PVA. leads the other two weight losses around 23% in the range of 400–600°C. From the DSC curve, we identified the exothermic peaks around 175, 190, 431 and 525°C due to evaporation. The decomposition of the PVA becomes constant beyond 500°C. Thermal analysis results indicate that there is no weight loss that occurs after 500°C.

3.4 Photoluminescence (PL) Analysis

The data presented are PL spectra of all the fibers which were taken by using a spectrometer and they are shown in Fig. 9a–d. It is observed that the strong UV-Visible emission band for the corresponding UV excitation of 290 nm. The Violet PL emission at 590 nm and the corresponding average energy is 3.5 eV which is lower when compared to pure MSPPP oxide (3.6 eV). It is attributed to the direct electronic transition between donor levels to the valence band. MSPPP oxide nano systems, the oxygen vacancy, is

one of the active luminescent centers, thus greatly influencing the PL emission. The occurrence of the PL band is associated with luminescent centers and dangling in the MSPPP oxide nanofibers.

3.5 Optical Studies

The optical properties of all fibers were studied by using a spectrometer from 190 to 2500 nm. The absorbance spectra of all fibers are shown in Fig. 10a–d. Range from 200 to 800 nm. The spectra results show that the absorption edge of MSPPP oxide nanofibers varied from 3.46 to 3.51 eV. The absorption edge is maximum for the fibers annealed at 400°C and minimum at 600°C. These results clearly show that band gap energy is closely associated with annealing temperatures at 290 nm excitation, MSPPP oxide nanoparticles exhibit emission at 600 nm.

4. Conclusion

In summary, high-quality MSPPP oxide nanofibers were fabricated by using an indigenously prepared simple electrospinning unit. The fibers are continuous, almost straight, defect-free, and cover the entire length of the glass substrate. The diameter of the MSPPP oxide fiber is in the order of a few tens of nanometers with a large draw ratio. The crystal size increases while the surface area decreases with an increase in annealing temperature. The optical band gap energy of MSPPP oxide is about 3.5 eV. Instead of a high technological vacuum deposition method, this novel technique of preparing very high-quality MSPPP oxide nanofiber thin films will be a breakthrough in preparing many window layers, photo electrodes, counter electrodes, touch screen sensors, UV emitting films, and many micro and nano electronic devices.

Declarations

Authors' contribution

The present work was conducted in collaboration with all the authors. The authors, Mohana Attia and Mariam Saeed, are responsible for the conceptualization of the present idea. The development of the theory, the performance of the calculations, and the validation of the analytical methods were the responsibility of Mohana Attia. Mariam Saeed was responsible for the review of the final draft.

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Figures

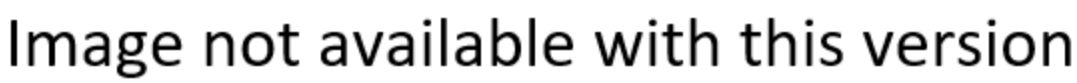


Figure 1

Molecular Structures of (MSPPP)



Figure 2

Optical microscope image of MSPPP crystal

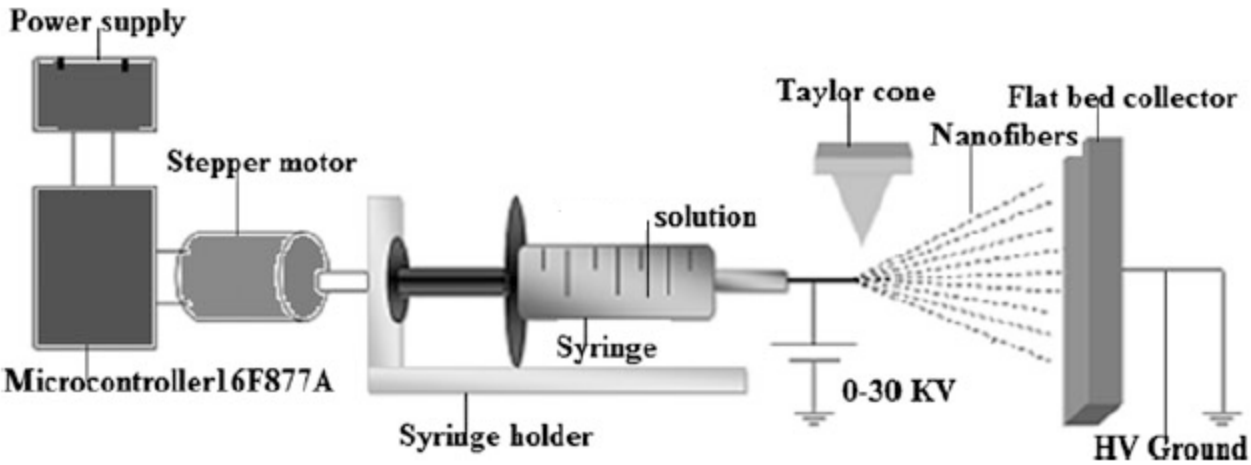


Figure 3

Electro-spin coating set up

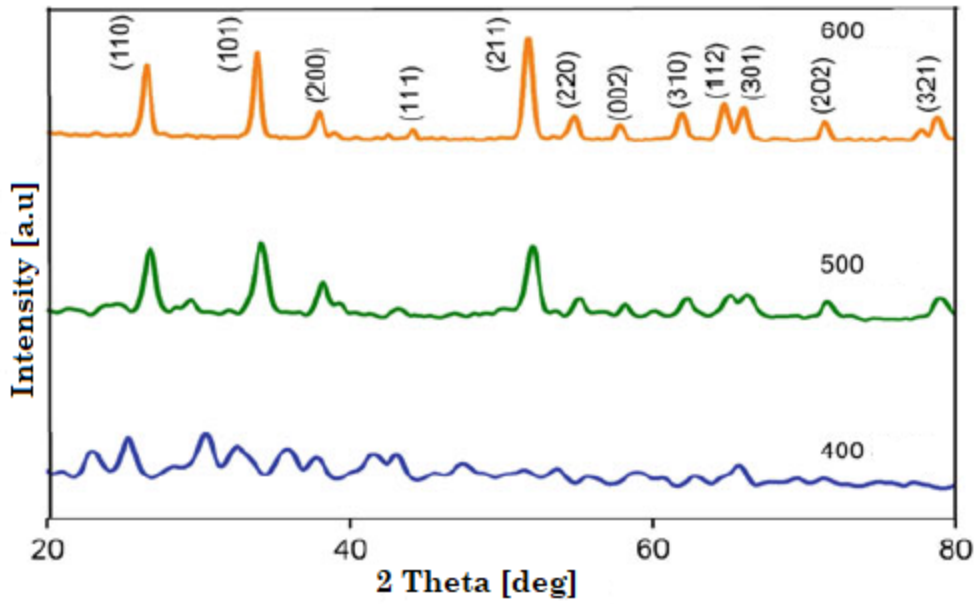


Figure 4

XRD patterns of the MSPPP oxide nanofibers with different calcination temperatures (400, 500, and 600 °C)

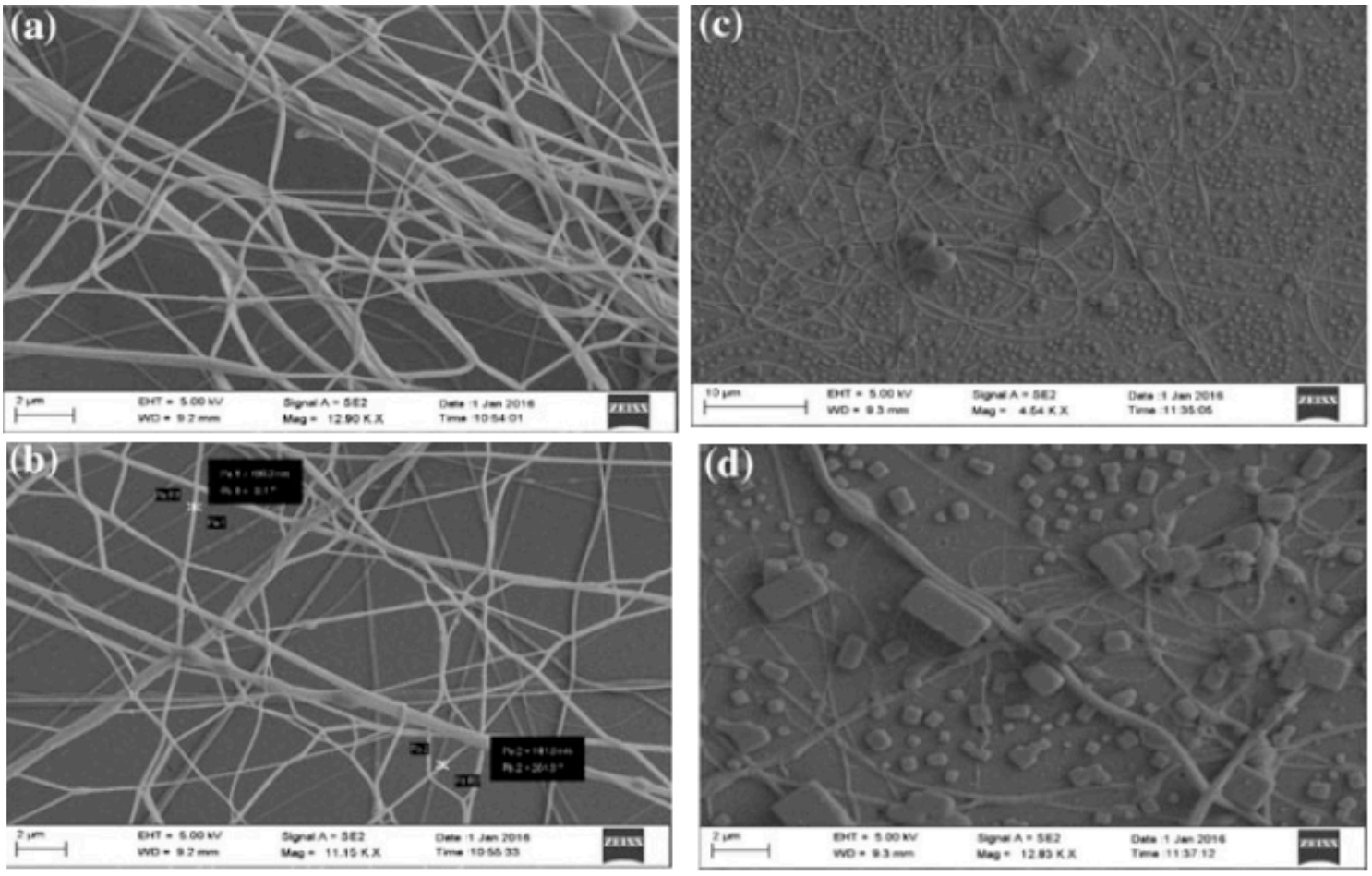


Figure 5

FESEM images of (a and b) PVA and MSPPP nanofibers, (c and d) MSPPP oxide nanofibers deposited on glass substrates

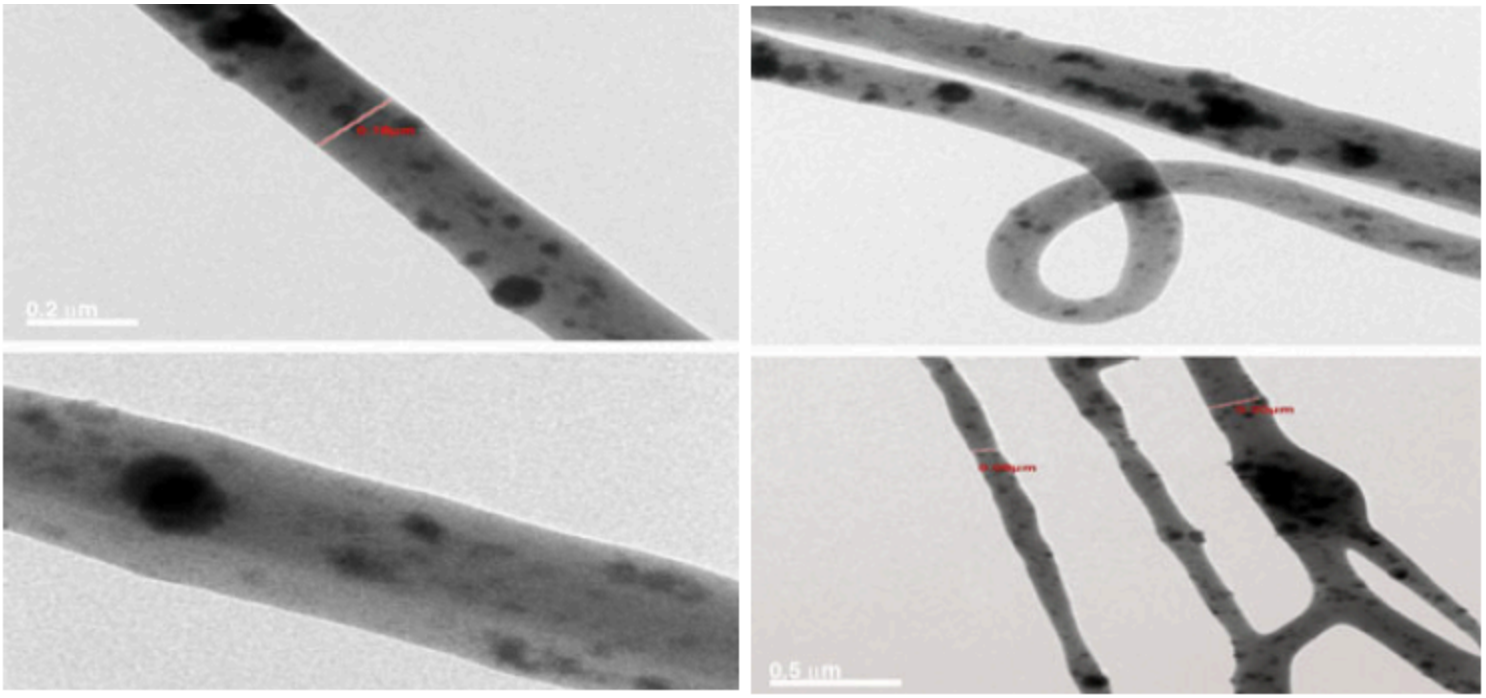


Figure 6

TEM images of PVA embedded with MSPPP oxide particles

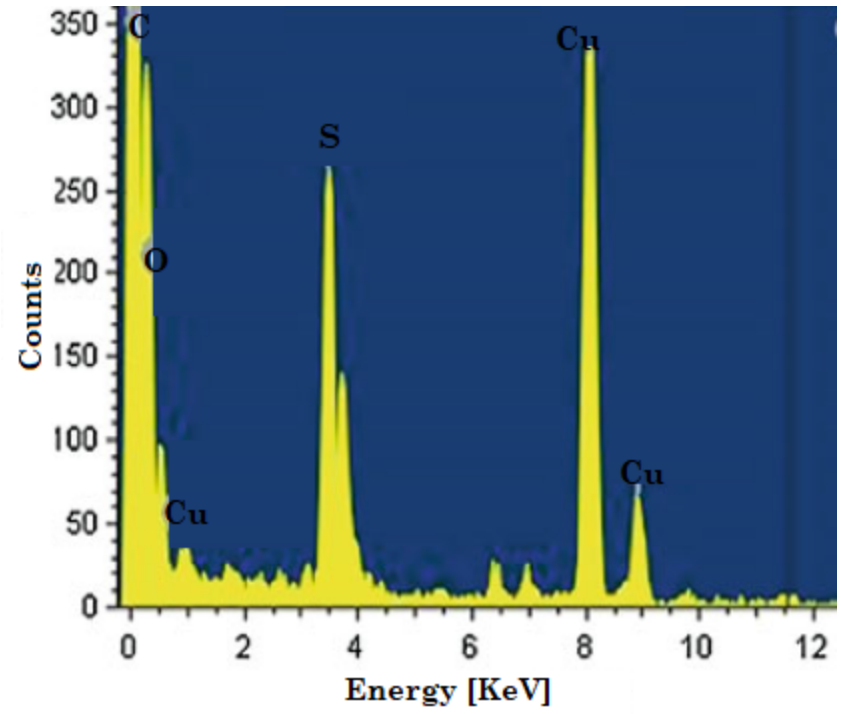


Figure 7

EDAX Spectrum of MSPPP oxide nanofibers

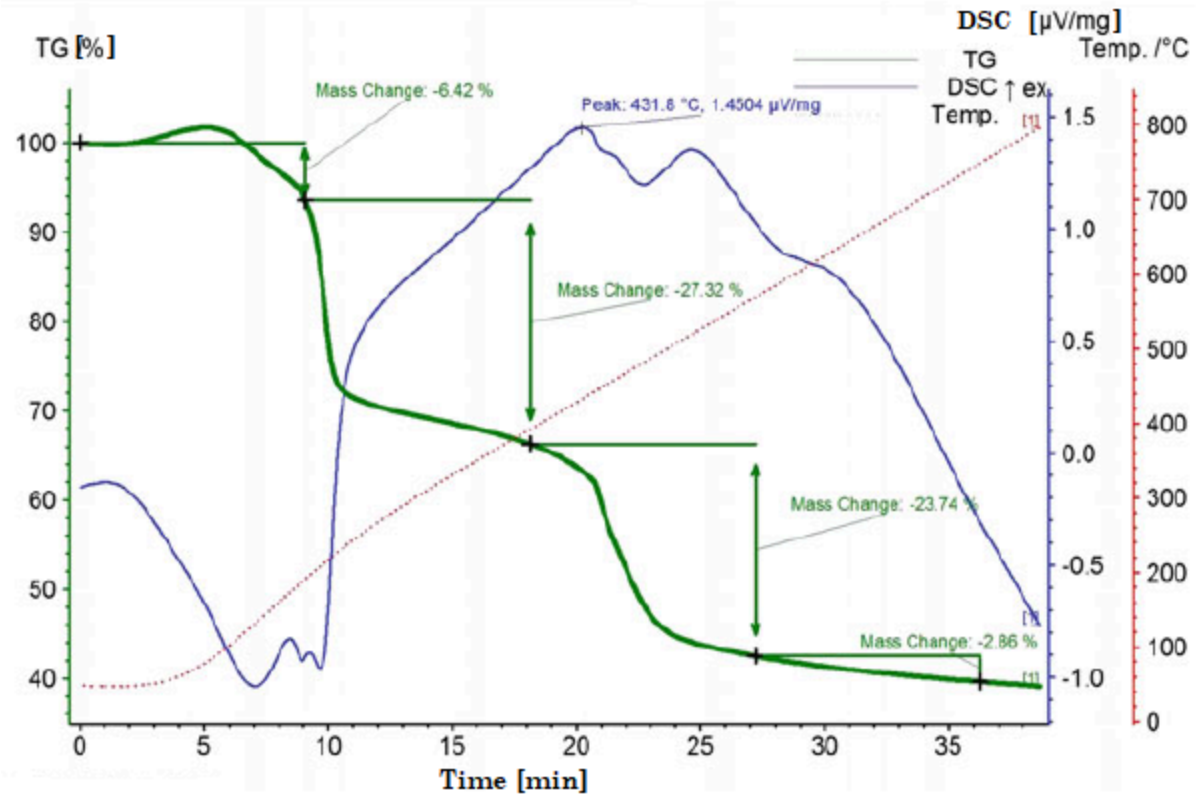


Figure 8

TGA-DSC thermal decomposition of electro spun PVA/MSPPP oxide composite nanofibers with mass change graph

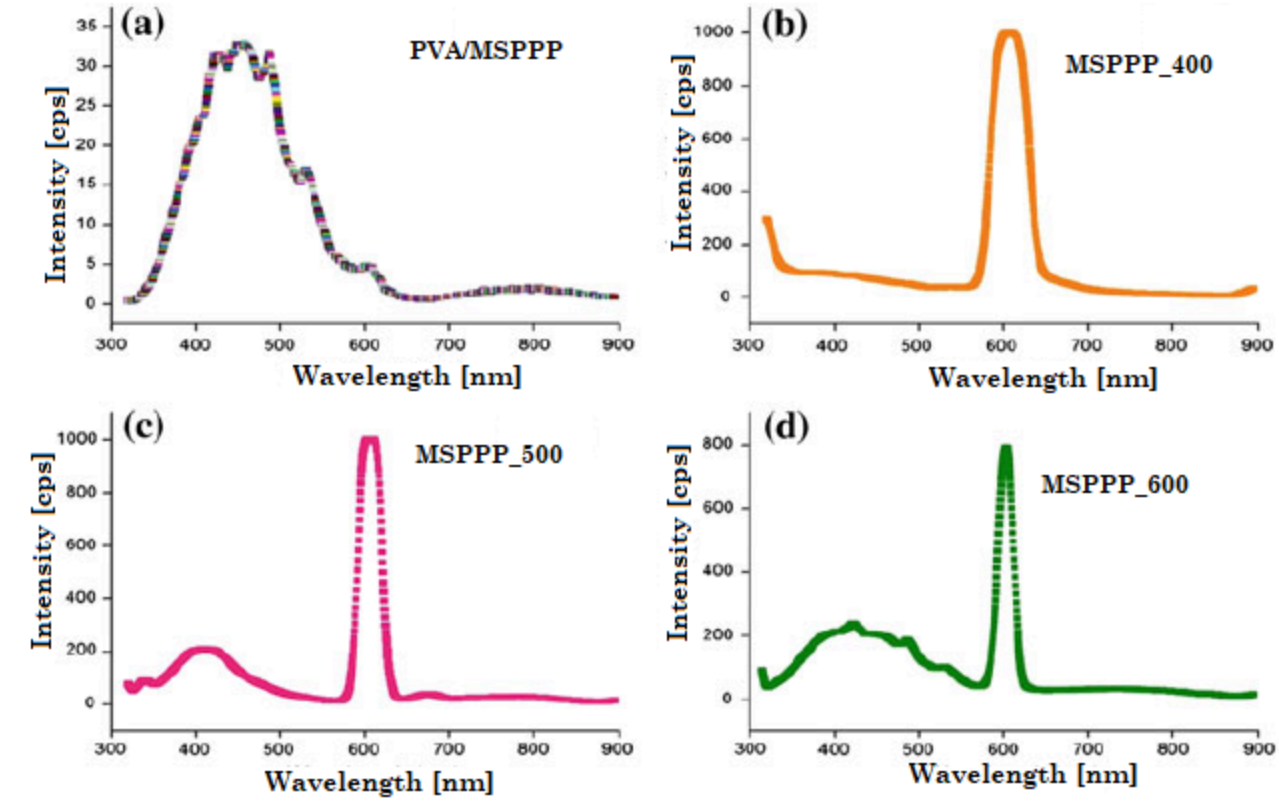


Figure 9

PL spectra of a PVA/MSPPP and b–d MSPPP oxide nanofibers

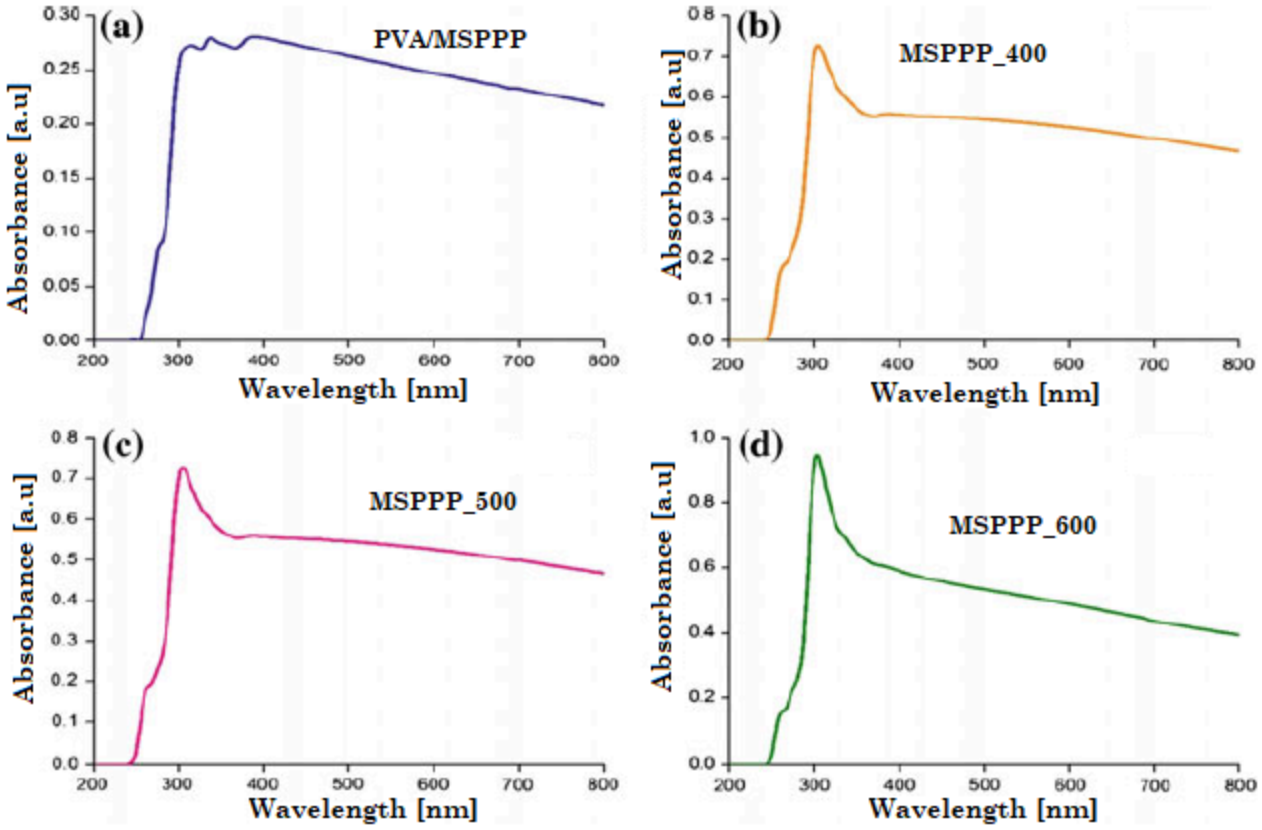


Figure 10

Absorbance spectra of a PVA/MSPPP and b–d MSPPP oxide nanofibers