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Exploring the properties of lead oxide and tungsten oxide based graphene mixed nanocomposite films

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This work reports the possibility of mixing metal oxides along with graphene and polymer to form Metal Oxide-Graphene-Polymer films. The metal oxides used in this work were lead Oxide (PbO) and tungsten oxide (WO_3). Polyvinylidene fluoride (PVDF) is the polymer, which is used as matrix in preparation of these metal oxide and graphene mixed nanocomposite films. Metal Oxide-Graphene-PVDF films were developed using a chemical technique called solvent casting. The prepared films were characterized using techniques like FE-SEM, XRD and FT-IR to understand its morphological, crystalline and functional properties. I-V characteristics were analyzed using four-probe techniques. The developed Metal Oxide-Graphene-PVDF films have potential applications in a wide range of fields and particularly for electronic applications. The ability of these films to be used as electronic components in devices was explored.

Keywords: graphene, polymer, metal oxide.

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1. Introduction

Graphene is a single layer of carbon atoms peeled from graphite as flakes. Graphene has numerous extraordinary properties which make it exceptionally valuable for different applications [1]. Graphene is preferred compared to carbon Nano tubes, carbon Nano fibers and other carbon materials due to its aspect ratio, high surface area, high tensile strength, good thermal conductivity, good electrical conductivity, transparency and flexibility [2,3]. Graphene is capable of being blended with a wide range of polymer matrices including, epoxy, polystene, polyaniline, nafion, poly (vinyl alcohol), polyurethane, polyvinylidene fluoride, poly (3,4-ethyldioxythiophene), polyethylene terephthalate and polycarborate [4]. Such composites were also shown to exhibit superior properties compared to base polymer materials, as a result of graphene's novel properties. In this present work, graphene along with metal oxides like lead oxide and tungsten oxide were mixed separately with PVDF polymer matrix in order to explore the conductivity behavior of graphene based nanocomposite films before and after mixing with various metal oxides, as previously mentioned.

2. Materials and methods

Functionalized graphene were purchased from Ad Nano Technologies with 99 % purity. Lead Oxide and tungsten Oxide were purchased from Sigma Aldrich with 99.4 % purity. Polyvinylidene Fluoride (PVDF) was purchased from Sigma Aldrich with 99 % purity. Dimethylformamide (DMF) of laboratory grade with 98 % purity was purchased from Sigma Aldrich.

FESEM and EDX images were examined using F E I Quanta FEG 200 – High Resolution Scanning Electron Microscope with EDX system. The prepared samples were examined at 50 μm range with 1000–2000 x magnification at low vacuum. XRD measurements are carried out using X'pert Pro Powder X-Ray Diffractometer system. The system used the high speed solid-state X'celerator as a detector type at the range of 0 – 100 °. FTIR pattern is measured using ALPHA-T FT-IR Spectrometer. The spectral range used for analyzing is 400 to 1000 cm^{-1} range. The spectral resolution used is better than 2 cm^{-1}.

3. Preparation protocol of nanocomposite films

3.1. Preparation of 2wt% of graphene-PVDF nanocomposite

Graphene (0.04 mg) and PVDF (1.96 mg) is mixed with DMF solution and sonicated for 1 hour to get proper mixing in the solution. Obtained graphene-PVDF solution is magnetically stirred for 1 hour by maintaining the temperature between 45 to 65 ° C to get homogenous dispersion in the solution [5]. Finally the solution is kept in hot air oven for 6 hours by keeping constant temperature at 60 °C.

3.2. Preparation of 2wt% of graphene-PVDF-PbO nanocomposite

Graphene (0.04 mg), lead Oxide (0.04 mg) and PVDF (1.92 mg) is mixed with a DMF solution and sonicated for 1 hour to get homogenous mixing in the solution. The obtained graphene-PVDF-PbO solution is magnetically stirred for 1 hour by maintaining the temperature between 45 to 65 °C to get proper dispersion in the solution. Finally, the solution is kept in hot air oven for 6 hours by keeping constant temperature at 60 °C.

3.3. Preparation of 2wt% of graphene-PVDF-WO_3 nanocomposite

The procedure used is similar to that of graphene-PVDF-PbO nanocomposites. Here lead Oxide is replaced with tungsten Oxide.

4. Results and discussions

Figure 1 shows FESEM images of (a) graphene-PVDF, (b) graphene-PVDF-PbO and (c) graphene-PVDF-WO_3 respectively. Superior bonding is noticed which shows the homogenous mixing in the prepared nanocomposites whereas the EDX pattern shows the presence of chemical compounds present in the nanocomposite. EDX images in Fig. 1(d) shows the presence of Carbon and Fluorine in the graphene-PVDF film, in Fig. 1(e) shows the presence of Carbon, Fluorine and Lead in the graphene-PVDF-PbO film and in Fig. 1(f) shows the presence of Carbon, Fluorine and Tungsten in the graphene-PVDF-WO_3.

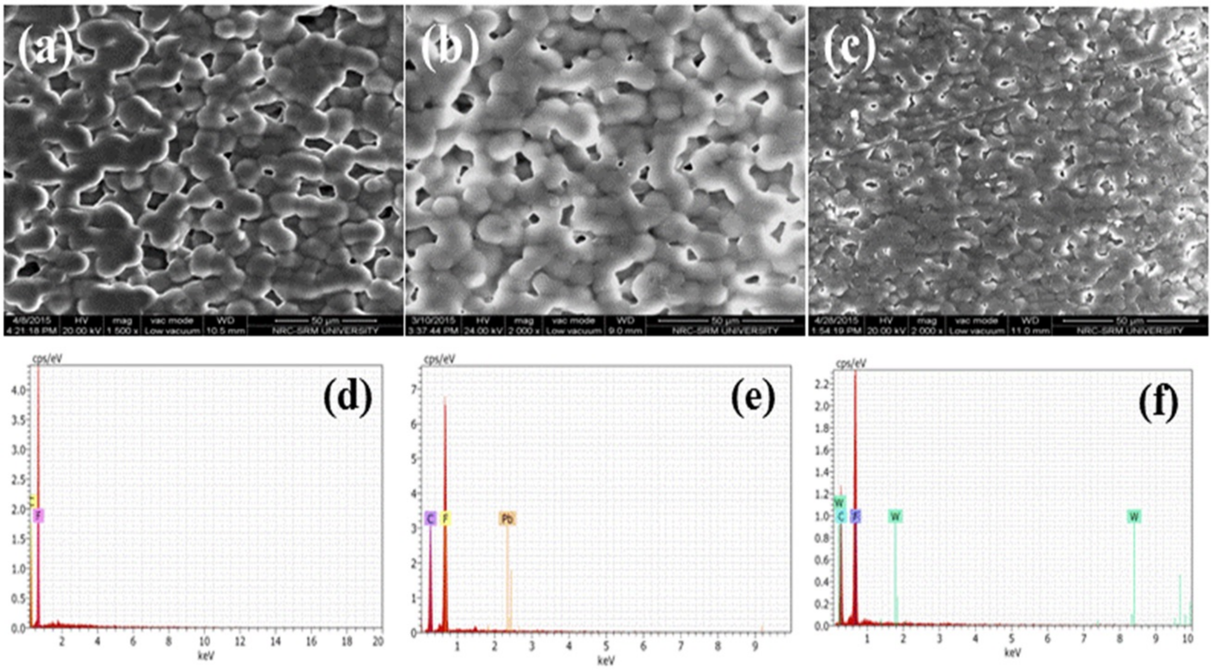

FIG. 1. FESEM images of (a) graphene-PVDF, (b) graphene-PVDF-PbO & (c) graphene-PVDF-WO_3 and EDX images of (d) graphene-PVDF, (e) graphene-PVDF-PbO & (f) graphene-PVDF-WO_3

Figure 2 shows the XRD pattern of (a) graphene-PVDF, (b) graphene-PVDF-PbO and (c) graphene-PVDF-WO_3 respectively. Fig. 2(a) shows the 2θ peaks at 20 ° (110) and 40 ° (002), which confirms the presence of PVDF and at 26 ° (021) as graphene (JCPDS Card No. 00-41-1487). Fig. 2(b) shows the 2θ peaks at 20 ° (110) and 40 ° (002) which confirms the presence of PVDF, at 26 ° (021) as graphene and at 31 ° (111) and 48 ° (220) as lead (JCPDS Card No. 00-38-1477) [6]. Fig. 2(c) shows the 2θ peaks at 20 ° (110) and 40 ° (002), which confirms the presence of PVDF, at 26 ° (021) as graphene and at 18 ° (100), 37.5 ° (021), 45 ° (020) and 48 ° (124) as Tungsten (JCPDS Card No. 00-43-1035) peak [7]. Fig. 3(a) shows the peaks of graphene-PVDF at 440 cm^{-1}, 485 cm^{-1} and 510 cm^{-1}. Fig. 3 (b) shows the peaks of graphene-PVDF-PbO at 430 cm^{-1}, 470 cm^{-1}, 500 cm^{-1} and 865 cm^{-1}. Fig. 3(c) shows the peaks of graphene-PVDF-WO_3 at 427 cm^{-1}, 465 cm^{-1} and 860 cm^{-1}. These peaks show the presence of aromatic C–C bonds between (400 – 600 cm^{-1}), C–O bonds between (700 – 1000 cm^{-1}) and C–H bonds between (400 – 650 cm^{-1}), which is due to the presence of carbon fillers.

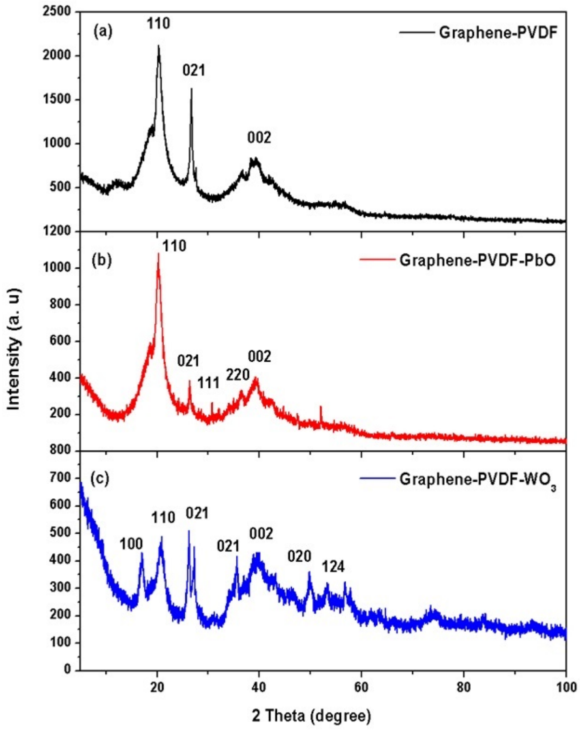

FIG. 2. XRD Pattern of (a) graphene-PVDF, (b) graphene-PVDF-PbO and (c) graphene-PVDF-WO$_3$

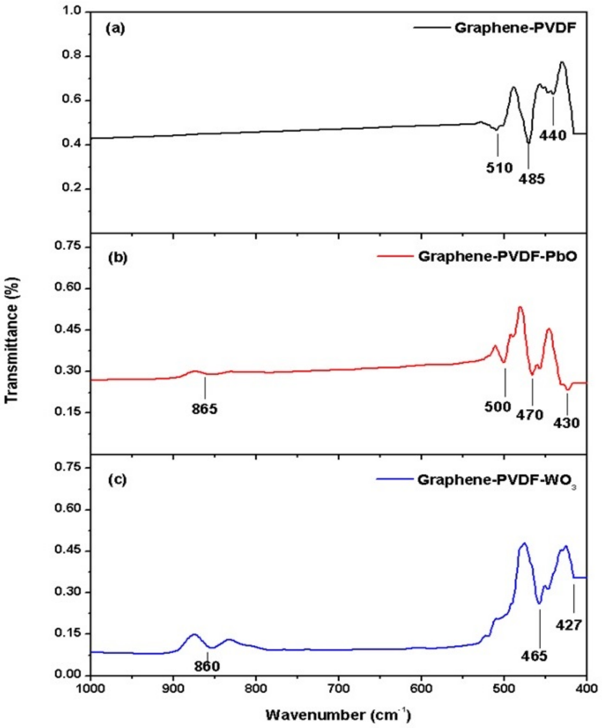

FIG. 3. FTIR Pattern of (a) graphene-PVDF, (b) graphene-PVDF-PbO and (c) graphene-PVDF-WO$_3$

Figure 4 shows the I-V Characteristics of (a) graphene-PVDF, (b) graphene-PVDF-PbO and (c) graphene-PVDF-WO$_3$ were obtained using four probe techniques. Temperature is kept as constant at room temperature and voltage is varied between 0 V to 1.4 V. Current measurements are carried out across all the three types of nanocomposite films, as shown in Fig. 4. Graphene-PVDF-WO$_3$ shows better conductivity when compared to graphene-PVDF-PbO and graphene-PVDF nanocomposite. This indicates metal oxide graphene based nanocomposite shows better conductivity due to incorporation of metal oxides in graphene based nanocomposites.

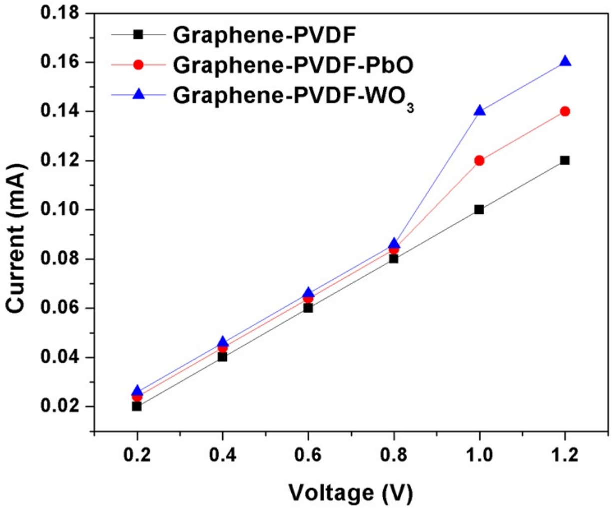

FIG. 4. I-V Characteristics of (a) graphene-PVDF, (b) graphene-PVDF-PbO and (c) graphene-PVDF-WO$_3$

5. Conclusions

Three different nanocomposite films, namely graphene-PVDF, graphene-PVDF-PbO and graphene-PVDF-WO_3 were prepared. SEM shows the morphological variations in all types of films. EDX shows the presence of elemental compositions such as carbon, fluorine, lead and tungsten of each film individually. XRD spectra shows the peaks of PVDF at 20 ° & 40 °, graphene at 26 °, lead at 31 ° & 48 ° and tungsten at 18 °, 37.5 °, 45 ° & 48 °. FT-IR spectra show the presence of various functional groups like aromatic C–C bonds, C–O bonds and C–H bonds. Developed nanocomposite films will have utility in flexible electronics in the near future.

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