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Production of graphene and carbon nanotubes using low cost carbon-based raw materials and their utilization in the production of polycarbonate/ethylene methyl acrylate -based nanocomposites

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In the earlier studies carbon nanomaterials were synthesized by using cheaper raw materials such as coal, super clean coal, plastics etc. by the authors research group. Presently, carbon nanomaterials such as graphene and carbon nanotubes have been prepared from cheap carbon-based materials such as dry ice and Pongamia oil. The graphene and carbon nanotubes were also synthesized by catalytic reduction of carbon dioxide (dry ice) in the presence of magnesium metal flame and from pongamia oil using chemical vapour deposition setup respectively. The multiwalled carbon nanotubes (MWCNTs) were synthesized. The synthesized graphene and MWCNTs were characterized by different characterization techniques such as scanning electron microscopy, transmission electron microscopy. Thus, synthesized graphene and MWCNTs have been used as conductive filler in polycarbonate/ethylene methyl acrylate nanocomposites. Different properties i.e. electrical conductivity and electromagnetic inference shielding properties of these nanocomposites have been studied. This study opens new avenues for the fabrication of novel lightweight materials that may be used in communication systems or for other applications such as in the fuel cells, solar cells, supercapacitors, Lithium ion batteries. Several cheaper raw materials have been suggested for the production of CNTs, graphene, carbon nanoparticles, quantum dots etc.

Keywords: Graphene, Carbon nanotubes, Dry ice, Pongamia oil, Polycarbonate

1 Introduction

Conventional reinforced polymer composites have displayed potential for their use in aerospace[1], military[2], automobile[3], sports[4], and energy applications (Lithium ion batteries, supercapacitors, fuel cells or solar cells) etc.[5-8]; however, the demand for multifunctional properties has encouraged the investigation of new materials. Energy industries are using inexpensive fuels and biofuels to replace the use of polluting fossil fuels which produce carbon dioxide besides several other pollutants[9-11]. Some of these cleaner fuels are already being produced at larger scales for safeguarding the environment and halting the climate change. In recent years, numerous studies on the development of advanced polymer composites with improved electrical, mechanical, thermal, and electromagnetic interference (EMI) shielding properties have been performed. Carbon nanomaterials, such as carbon nanotubes (CNTs), carbon nanofibers (CNF) and graphene have been considered as ideal reinforcements for polymer composites due to their exceptional electrical and thermophysical properties coupled with their high aspect ratio, high strength, and stiffness, which leads to significant enhancements in electrical conductivity, mechanical stability, and EMI shielding[12-18]. The properties of these attractive carbon-based systems have been evaluated for the most part independently. As the potential for practical applications is being reported, the development of systems with multifunctional properties has attracted considerable interest from the scientific community in synergistically improving thermal, electrical and structural properties.

Petroleum derived chemicals, such as toluene, xylene or other hydrocarbons or terpenes etc. have been mostly utilized for the production of graphene and carbon nanotubes[19-23]. There is a need to replace the use of these costly feedstocks with inexpensive and waste raw materials, especially when these materials would be required to be manufactured at the larger scales in industries. Authors and their research group have focused on the production of cleaner fuels from coal, petroleum, biomass, waste plastics etc.[24-27]. Cleaner coal technologies were developed to produce the cleaner fuels such as super clean coal and demineralized coal from Indian coals[28-30]. The process

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of organo-refining of coal involving the solvent extraction (N-methyl-2-pyrrolidine and smaller amounts of ethylenediamine, the industrial solvents) was developed to produce super clean coals (SCC) having zero to less than 1-2 % ash contents[31]. This process was also scaled up and even pilot plant studies were completed by a leading steel plant in India for testing this technology at larger scales. Inorgano-leaching of Indian coals using the alkali and acids resulted in the removal of more than 70-80 % of ash contents from the coals to produce demineralized coals[32,33]. Thisprocess had also been scaled up and tested. The use of super clean coal and demineralized coals was made to produce CNTs, mostly single walled nanotubes[34,35] and these CNTs were used for preparing polystyrene based nanocomposites[36,37]. In the separate studies, vegetable oils, such as, Jatropha oil, Pongamia oil which are used for the production of biodiesel through trans-esterification or biodiesel and green diesel and green gasoline through catalytic hydrocracking were also used for the production of CNTs which can be used to produce graphene. The use of waste plastics was also made to produce CNTs by the authors in the past[36].

Even CO_2 which is a greenhouse gas responsible for the climate change was used to produce graphene and CNTs[36-38]. The graphene and CNTs produced by using the inexpensive raw materials or feed stocks were used to produce graphene and carbon nanotubes based polymeric nanocomposites. The use of CO_2 in the preparation of graphene, carbon nanotubes and polycarbonates along with their further utilization in the preparation of polymeric nanocomposites could be of interest in the utilization of CO_2 a greenhouse gas which may help in halting the climate change[15, 39-41]. This may also afford the continued use of coal which is the backbone of power generation and steel production in not only in India but in the world over. It may also be seen that the CO_2 may also be utilized to produce polycarbonates. The authors investigated and compared the effect of graphene and carbon nanotubes on the electrical and EMI shielding properties of polycarbonate / ethylene methyl acrylate (PC/EMA) nanocomposites.

Earlier the authors had used the graphene and MWCNTs which were synthesized by using cost effective raw materials with mainly polystyrene or polycarbonates[15,38-41]. Presently the focus of the studies of the authors has been on the comparative studies of graphene and MWCNTs based PC/EMA nanocomposites.

2 Materials and Methods

2.1 Materials

Commercial grade polycarbonate (PC) LEXAN TM Resin 143 obtained from SABIC Innovative Plastics with melt flow index (MFI) 10.5 g/10min at 300 °C with 1.2 kg load anddensity-1.19 g/cm^3 was used for present study. Ethylene-methyl acrylate (EMA) copolymer is a product of DuPont Packaging & Industrial Polymers under trade name Elvaloy® AC 1330.All the materials were used as received. The magnesium metal ribbons were purchased from Merck. Dry ice slab was purchased from Ashok & CO., Noida. Hydrochloric acid (HCl), toluene and ethanol was obtained from Merck and used as such. The MWCNT used in the study were synthesized by CVD method using pongamia oil as carbon precursor and ferrocene as catalyst.

2.2 Experimental section

2.2.1 Synthesis of Graphene

Graphene was produced by burning of Mg metal ribbon in presence of carbon dioxide (dry ice). Dry ice was taken in two blocks and a hole of diameter of about 5-6 cm radius and depth of about 10 cm was made on any one of the blocks, each block was of 2 kg (Fig. 1). The metal Mg was taken in ribbon form. After the explosive reaction, the mixture of white and black flakes obtained. The following reactions took place during the process:

$$2Mg\ (s) + CO\ 2\ (g)\ 2MgO\ (s) + C\ (s) \qquad \ldots (1)$$

There is a scope of using other metals such as Ni or Zn as co-catalysts which further promotion this reaction and studies in this direction were also extended. Then the mixture washed with dil. HCl to remove the metal oxides and unreacted metal. After the alkali metals are dissolved, the solution is filtered and washed with distilled water to get the graphene.

2.2.2 Synthesis of carbon nanotubes

MWCNTs were grown on the carbon substrates by thermal decomposition of pongamia oil in presence of iron organometallic catalyst like ferrocene using two zone furnace with temperature of 350 °C in first zone and 750 °C in the second zone. The first furnace CVD setup was utilized for vaporizing precursor like pongamia oil and the second furnace of chemical vapor deposition was utilized for pyrolysis of given precursor's vapor. The reaction was allowed to ensue for 4 hr. After completion of process, the CNT was collected and purified by acid treatment for removal of catalyst traces and amorphous carbon.

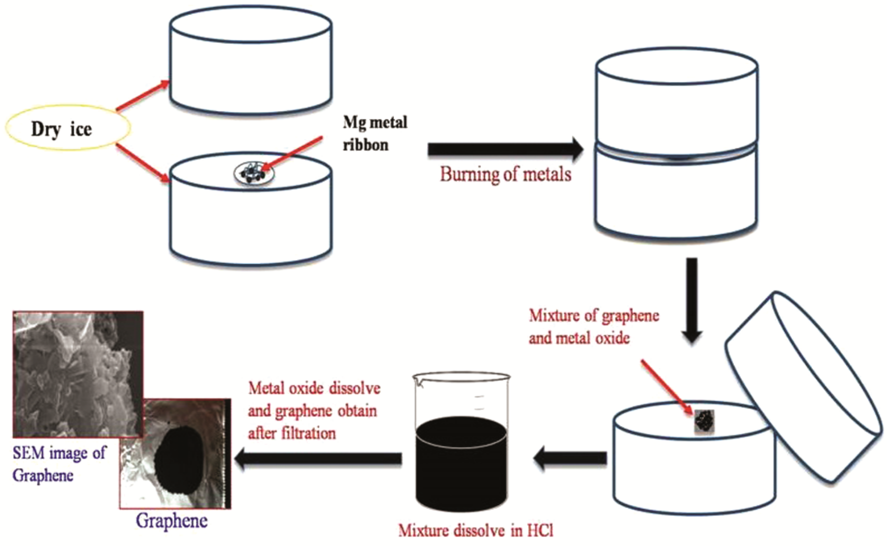

Fig. 1 — Schematic diagram of graphene synthesis.

Table 1 — Details of formulations and sample designation of PC/EMA-G and PC/EMA-MWCNT nanocomposites.

Sample Designation*	Polycarbonate/ethylene methyl acrylate (wt.%)	Graphene loading (phr)	MWCNT loading (phr)
PCE5	100	-	-
PCEG2	100	2	-
PCEG5	100	5	-
PCEG10	100	10	-
PCEG15	100	15	-
PCENT2	100	-	2
PCENT5	100	-	5
PCENT10	100	-	10
PCENT15	100	-	15

(*where PC - Polycarbonate, E - Ethylene methyl acrylate, G – Graphene, NT- Multiwalled carbon nanotubes, Numerical value- graphene or MWCNT content)

2.2.3 Fabrication of graphene and carbon nanotube based polymer nanocomposites

Polycarbonate/ethylene methyl acrylate-graphene (PC/EMA-G) nanocomposites with varying graphene loadings i.e. 2, 5, 10 and 15 phr in PC/EMA (95/5 w/w) blend (Table 1) were prepared by melt compounding method using co-rotating micro compounder (HAAKE MiniLab II) at processing temperature of 300 °C, mixing time of 5 min and screw speed of 100 rpm (Fig. 2).

Polycarbonate/ethylene-methyl acrylate-multiwalled carbon nanotube (PC/EMA-MWCNT) nanocomposites having varying amounts of MWCNTs, i.e. 2, 5, 10 and 15 phr, in PC/EMA blend (Table 1) were prepared by melt blending using micro-compounder (Model: HAAKE Minilab II) at processing temperature 270 °C, screw speed 100 rpm and mixing time 5 min. The recirculating melt is diverted towards micro injection molding unit (Thermo Scientific HAAKE Mini) for the preparation of test specimens (Fig. 2).

3 Characterization Techniques

The morphological characterization of carbon nanofiller was done using scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The surface morphology of carbon nanofiller were performed using SEM (Zeiss EVO 50) at the accelerating voltage of20kV. For SEM analysis, cryogenically fractured specimens of composites were used. TEM images were recorded using FEI Tecnai TF20 operating at the accelerating voltage of 200 kV.

Purity of graphene and MWCNT were observed by using Raman spectroscopy (Labram HR 800 Evo Raman Spectrometer) with Argon laser of wavelength 514.5 nm. The crystallinity of the filler materials were examined by X-ray diffraction ((PW3050/60 Xpert Pro Netherlands).

The electrical conductivity (EC) is the measure of electric current carrying capacity of a material. The direct current (DC) electrical conductivity of nanocomposites was performed on the molded bars and determined by using Keithley semiconductor characterization system in two-probe configuration. For electrical conductivity measurement, silver pasted composite bars were used to make electric contacts. Minimum five samples for each composition were tested. Electromagnetic interference (EMI) shielding

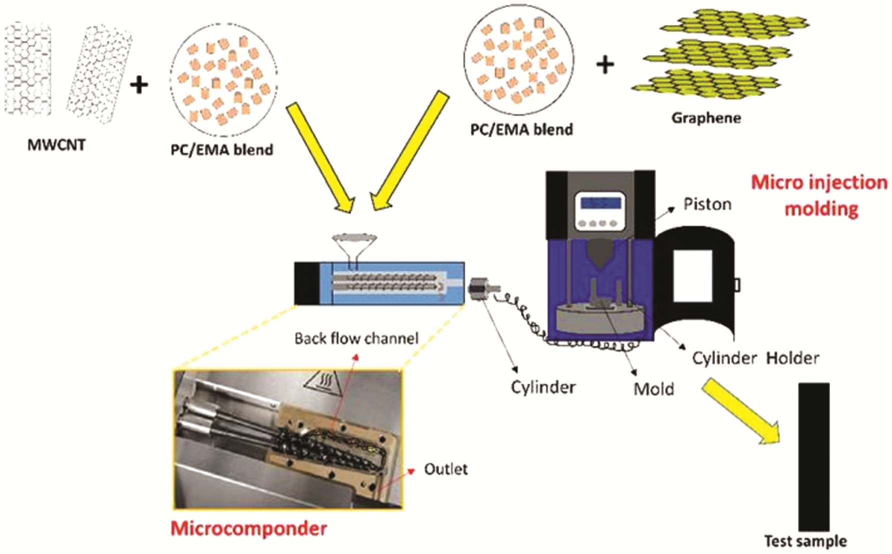

Fig. 2 — Schematic diagram for fabrication of PC/EMA-G and PC/EMA-MWCNT nanocomposites.

is defined as the ability of a material to carry out a certain level of attenuation in order to act as a barrier against the propagating electromagnetic waves. Shielding efficiency is measured in terms of the ratio of incident power to the outgoing power upon transition across the shield material and expressed in terms of decibel (dB). Electromagnetic interference shielding effectiveness of nanocomposites was measured by recording the scattering pattern on Agilent E8362B Vector Network Analyzer (VNA) as shown in Fig. 3 using two port measurement technique in X-band (8.2-12.4 GHz).

4 Results & Discussion

4.1 Morphological characterization of graphene and multiwalled carbon nanotubes

The graphene synthesized was characterized by using SEM and TEM. From the SEM images of the graphene samples are shown in Fig. 4 (a), the morphology of graphene is also revealed with length of 20–100 nm. TEM image of obtained graphene are shown in Fig. 5 (a). From TEM images, it can be seen that the graphene sheets are seen as superimposed on each other and their sizes are typically between 26 & 80 nm which analyzed by Image J software.

Morphology of the multiwalled carbon nanotubes sample formed by catalytic decomposition of Pongamia oil was examined by SEM (Fig. 4 b) and TEM (Fig. 5 b) analysis. SEM and TEM micrographs disclosed that the diameter of MWCNT was in the range of 150–200 nm with average value of 125.8 nm and length is in the range 5-10 µm.

Fig. 3 — Vector network analyzer (VNA).

4.2 X-ray diffraction pattern of graphene and multiwalled carbon nanotubes

The X-ray diffraction (XRD) pattern of the graphene material synthesized from Mg metals as the reductant and CO_2 as the oxidant are shown in Fig. 6 (a). Two characteristic peaks were obtained in XRD. A broad graphitic (002), corresponding to an inter-layer spacing of about 0.34 nm was obtained at $2\theta = 26.10$. The graphitic (002) peak in the XRD pattern is presenting that multilayer graphene produced. The other two additional peaks were obtained due to the {200} reflections of MgO obtained at 43.02.

XRD pattern of produced MWCNTs are shown in Fig. 6 (b) which explains a sharp peak to (002) plane at approximately $2\theta = 26°$ and was obtained from the configuration of the concentric cylinders of graphitic carbon. The peaks at approximately 43° were due to

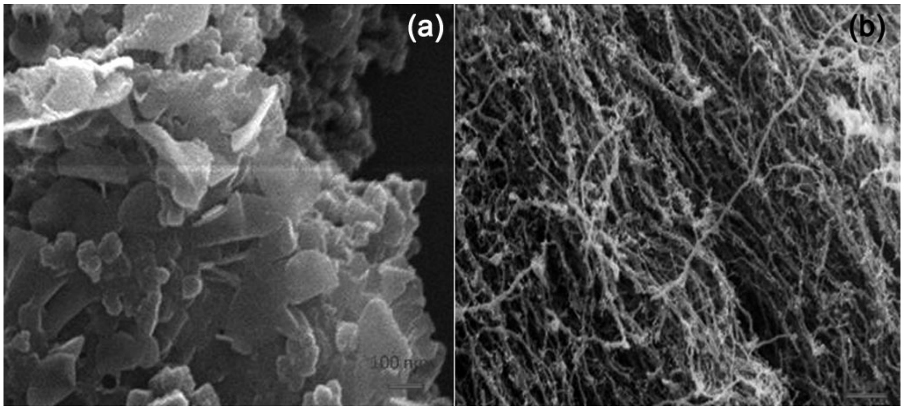

Fig. 4 — SEM images of (a) graphene and (b) multiwalled carbon nanotubes.

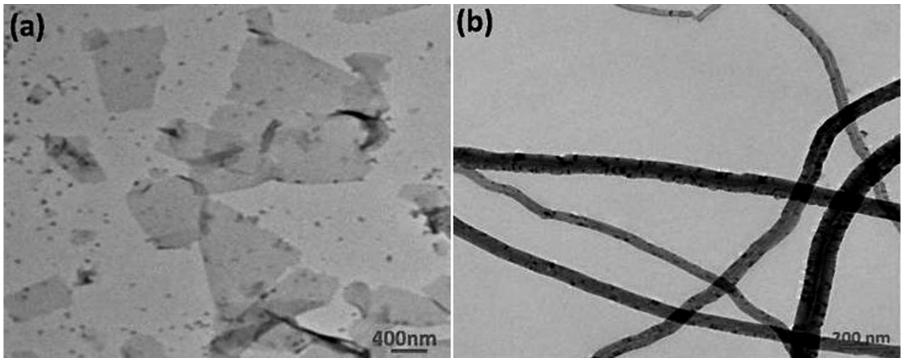

Fig. 5 — TEM images of (a) graphene and (b) multiwalled carbon nanotubes.

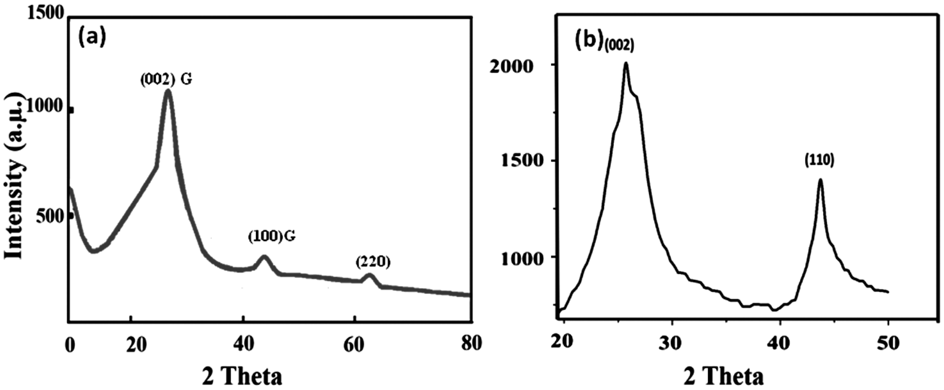

Fig. 6 — X-ray diffraction pattern of as-prepared (a) graphene and (b) MWCNT material.

the (110) and (100) graphitic planes adding minute amount of catalyst unit summarize within the walls of the MWCNTs. XRD pattern of MWCNTs showed that one dimensional multi walled carbon nanotubes have crystalline structure.

4.3 Raman spectrum of graphene and multiwalled carbon nanotubes

Raman spectroscopy is a most important technique for characterizing graphene and MWCNT because it can interpret fine structural and electronic information with disorder and defect structures in graphene. We acquired Raman spectra using 514 nm radiation. Three main characteristic peaks, the D, G and 2D obtained in Raman spectrum of graphene (Fig. 7 a). D band was obtained due to the in-plane breathing vibration mode, and the G band was resulted due to the doubly degenerate in plane vibration mode, which results in two peaks at about 1,344 and 1,575 cm^{-1} respectively. The most noticeable peak was the 2D band obtained at 2,686 cm^{-1}, which has been used to differentiate between monolayer, bilayer, and few-layer graphene on the basis of the position and shape of the 2D band. The graphene obtained here have typically fewer than five graphene layers.

Raman spectrum for produced MWCNTs exhibited characteristic D and G bands located at 1,352 cm^{-1} and 1,580 cm^{-1} respectively, as shown in Fig. 7 (b). The D-band is related to the sp^3 state of carbon and can be used as an evidence of disturbance of the aromatic π-electrons (sp^2 hybridized) of carbon nanotubes, while G band is linked to the graphitic configuration (sp^2 carbons).

4.4 Morphological characterization of PC/EMA nanocomposites

The morphologies of the PC/EMA nanocomposites were investigated by using SEM and TEM analysis. The SEM image (Fig. 8a) of PC/EMA-5 phr graphene nanocomposites reveals that the graphene show uniform dispersion in the PC/EMA matrix. Fig. 8b which represent the SEM micrograph of the

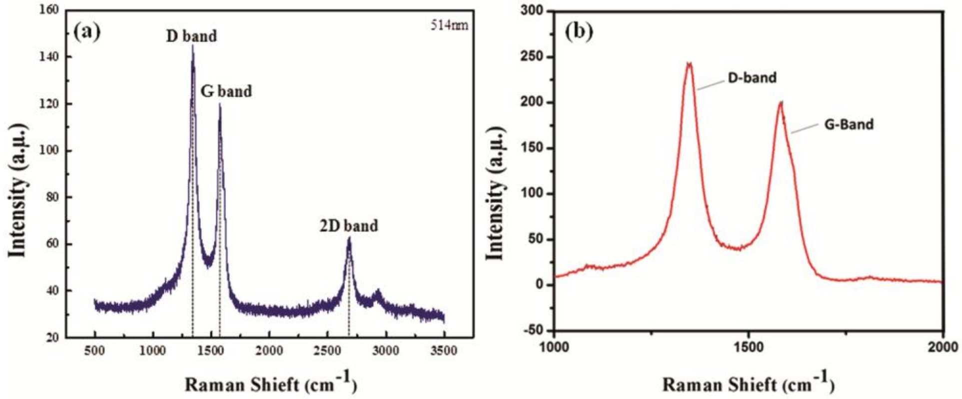

Fig. 7 — Raman spectra at 514 nm of as-prepared (a) graphene and (b) MWCNT material.

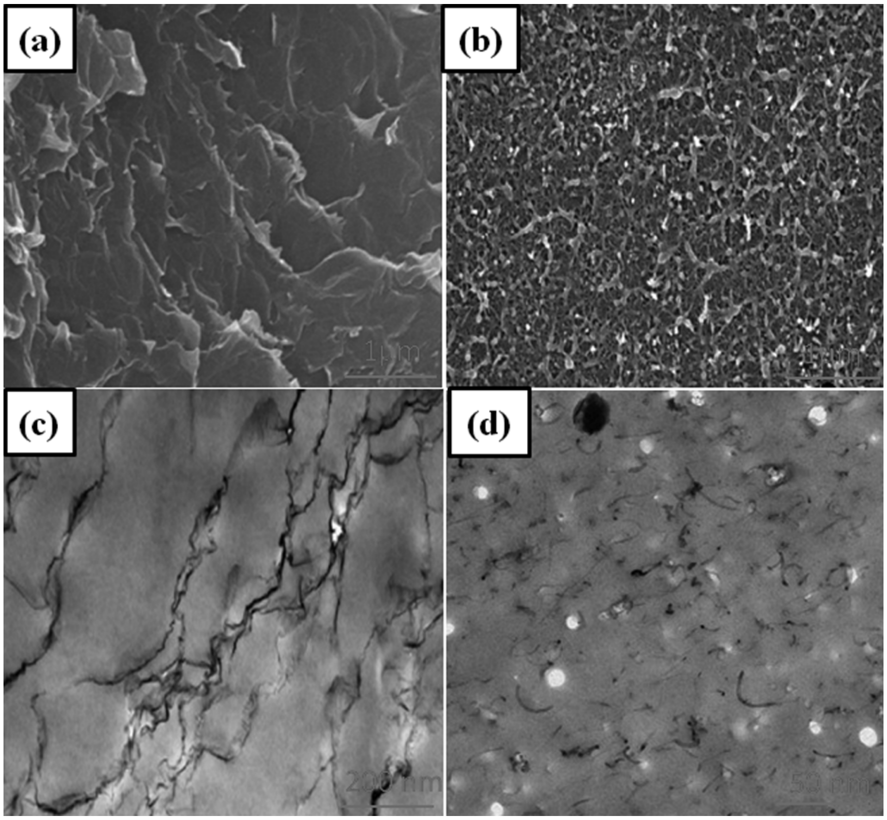

Fig. 8 — SEM images of PC/EMA nanocomposites with (a) 5 phr graphene and (b) 5 phr MWCNT; TEM images of PC/EMA nanocomposites with (c) 5 phr graphene and (d) 5 phr MWCNT.

PC/EMA-5 wt% MWCNT nanocomposites, some agglomerations of MWCNT were found. Thus MWCNT does not disperse as uniformly in the PC/EMA matrix as compared to graphene. This was also confirmed by TEM micrograph of PECNT5 composite shown in Fig. 8d. Some dark spots were noticed in the image, which represents MWCNT agglomerations. TEM micrograph of PEG5 composite is much different, as indicated in Fig. 8c, there was no evidence of multilayer stacks of graphene. Therefore, graphene was homogeneously dispersed in the matrix, and a continuous network structure was formed. This explains why the electrical conductivity of the PC/EMA-graphene nanocomposites is distinctly superior to the PC/EMA-MWCNT nanocomposites.

4.5 Comparison of electrical conductivity of the PC/EMA-graphene and PC/EMA-MWCNT nanocomposites

Figure 9 shows DC electrical conductivity (EC) of PC/EMA-graphene and PC/EMA-MWCNT nanocomposites with different filler loadings (2-15 phr). The conductivity of both graphene and MWCNTs based composites increased with increasing filler content. PC/EMA-graphene composites display a sharp rise in EC below 2 phr loading of graphene (Fig. 9), which is signature of formation of 3D conducting networks or percolation pathways. Thus, these nanocomposites showed percolation threshold below 2 phr loading of graphene. PC/EMA-graphene nanocomposites showed EC about 0.49 S/cm at 2 phr loading of graphene. Maximum EC was observed to be 9.9 S/cm at 15 phr loading of graphene. In case of PC/EMA-MWCNT nanocomposites, the percolation threshold was observed below 2 phr loading of MWCNTs and maximum EC was observed about 2.3 S/cm in 15 phr MWCNT loaded nanocomposites. As we have compared the EC of both nanocomposites, PC/EMA-graphene composites showed better EC than PC/EMA-MWCNT nanocomposites.

4.6 Comparison of electromagnetic shielding results of the PC/EMA-graphene and PC/EMA-MWCNT nanocomposites

EMI-SE is defined as the attenuation of the propagating electromagnetic waves produced by the shielding material. EMI shielding refers to the reflection and absorption of electromagnetic radiation by the material. The presence of charge carriers in material helps in electromagnetic wave reflection via reflection mechanism electromagnetic wave penetrate through the material and get attenuated via the absorption. Electromagnetic shielding effectiveness (SE) of nanocomposites in the X-band frequency range of 8–12 GHz are represented in Fig. 10. According to electrical conductivity results, the highly conductive PC/EMA-graphene composites show higher EMI shielding effectiveness than PC/EMA-MWCNT composites. The PC/EMA-graphene composites show higher EMI shielding effectiveness about -36 dB at 15 phr graphene content (Fig. 10). In contrast, PC/EMA-MWCNT composites showed highest EMI shielding about -27 dB at the same loading of MWCNT. Thus EMI SE of the PC/EMA-graphene composite has ~9 dB higher than that of the PC/EMA-MWCNT composite at 10 phr loading of filler. Therefore, the improvement in EMI SE in PC/EMA nanocomposites with graphene content may result from the improved reflection and absorption capability of the conducting network of graphene

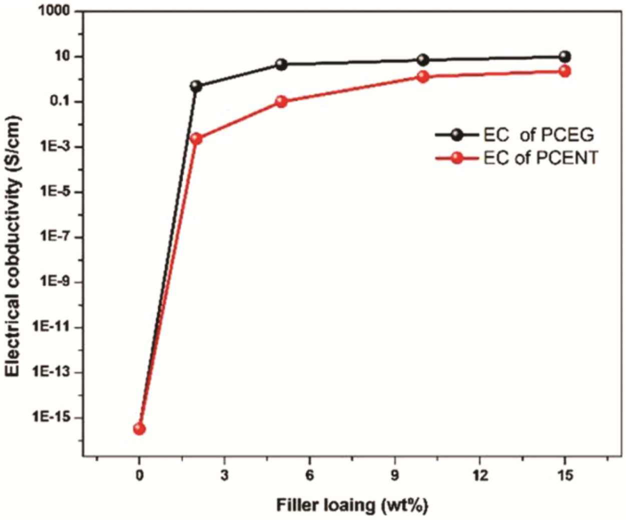

Fig. 9 — Electrical conductivity of PC/EMA-graphene and PC/EMA-MWCNT nanocomposites.

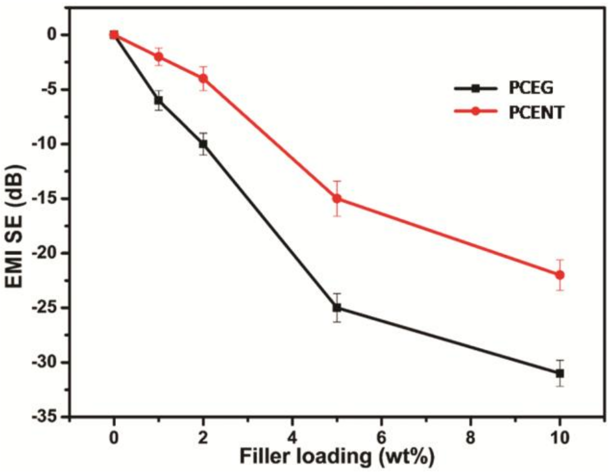

Fig. 10 — EMI SE of graphene and MWCNTs based PC/EMA nanocomposites.

sheets in PC/EMA matrix. Usually, the target value of the EMI SE required for commercial applications such as mobile cell application, laptop and desktop computers is in between 20-30 dB. Therefore, the results show that the target value of SE was reached by incorporating 5-15 phr graphene in PC/EMA matrix. These PC/EMA-graphene nanocomposites are very promising for use as an effective and practical EMI shielding material due to their low cost, light weight and high shielding performance properties[16-18, 38-42].

5 Conclusions

The studies have also successfully explored the potential of graphene and multi-wall carbon nanotubes as conductive reinforcements in PC/EMA nanocomposites. Graphene nanomaterial has been successfully synthesized by catalytic reduction of dry ice (carbon dioxide) in the presence of Mg metal flames. Earlier Jatropha oil was also successfully used for the production of CNTs. Presently the MWCNTs have also been synthesized from the Pongamia oil using CVD method. These nanomaterials have been characterized by different techniques such as SEM, TEM etc. Thus synthesized graphene and MWCNTs were used as conductive filler in the preparation of PC/EMA-graphene and PC/EMA-MWCNT nanocomposites. PC/EMA-graphene composites show enhanced conductive and EMI shielding in comparison to the PC/EMA-MWCNT composites. The highest electrical conductivity about 8.9 S cm^{-1} was achieved for nanocomposites having 15 phr loading of graphene. The highest EMI SE was found to be −36 dB in X-band (8–12 GHz) for 15 phr graphene loaded PC/EMA nanocomposites. In contrast, 15 phr MWCNTs loaded PC/EMA nanocomposites showed highest EMI shielding about -27 dB. Thus 15 phr loaded PC/EMA-graphene composites graphene composites show better electrical and EMI shielding properties. From these studies, it can be concluded that the electrically conducting lightweight shield material can be prepared using PC/EMA-graphene nanocomposites.

Since there is a need to find out a low cost raw material and feedstock for the production of graphene and CNTs at larger scales in industries the following low cost feed stocks may be used for the production of CNTs, graphene and carbon nanoparticles: coals, coke, coke breeze, wood charcoal, coal tar, coal tar pitch, turpentine oil, pine oil, palm oil, Jatropha oil/Pongamia oil/ other seed oils, etc., agro residues/ algae/ biomass/ pyritic oil, carbon dioxide/ dry ice, biogas/ sewage gas, etc., organic wastes including waste cooking oils/cigarette butts, waste plastics - polyethylene, polystyrene, bakelite etc., lignin – black liquor, wood or algal extracts, petcoke / waste refinery gases, petroleum vacuum residue, waste carbons from electrodes of batteries and other sources, anthracite etc. These raw materials may be used for the production of carbon nanocomposites and grapheme at larger scales at lower processing costs by industries. The use of welding equipment and fluidization processes for the production of carbon nanomaterials using low cost raw materials such as low ash coals, anthracites, SCCs, biomass, waste plastics etc. would help in reducing the cost of production of carbon nanomaterials for use in industries in future. However, further research may have to be extended in this direction to study the use of some other feedstocks besides coal, super clean coal, anthracites, seed oils or used cooking oils, waste plastics etc.

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