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Effect of Graphene Oxide and Multi-Walled Carbon Nanotubes on the Structure and Properties of Pitch Derived Carbon Foam composites

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Abstract

Multi-walled carbon nanotubes (MWCNTs) and graphene oxide (GO) reinforced carbon foam (CF) composite were prepared by direct pyrolysis of MWCNTs, GO and mesophase coal tar pitch. The effect of additive amount of the mixture of MWCNTs and GO on the microstruture and properties of carbon foam was analzyed by transmission electron miscroscopy (TEM), scanning electron microscopy (SEM), X-ray diffraction (XRD), Four-probe resistance meter, universal testing machine, and laser thermal conductivity tester respectively. The result shows that MWCNTs and GO had significant impact on the microstructure of carbon foam. Futhermore, the electrical, mechanical and thermal properties of carbon foam composites were significantly enhanced by increasing the additive amount. Maximum compressive strenght of 19.2 MPa and Young's modulus of 56.8 MPa of CF composite were observed. Similarly, Highest thermal conductivity of 30.91 W/m.K and electrical conductivity of 27.2 $\times 10^3$ S/m were observed at 2 wt. % of MWCNTs-GO additive loading.

Key Words: Carbon foam, multi walled carbon nanotubes, Graphene oxide, electrical, mechanical and thermal properties.

1. Introduction

Graphene are two dimensional (2D) allotrope of carbon, having a single layer of carbon atoms arranged in hexagonal pattern. It is well known material due to its characteristic electrical and mechanical properties. Moreover, graphene has versatile applications in electrochemical-electrodes, solar cells, sensor and many other materials. Graphene can be synthesized by various methods such as, micro chemical exfoliation of graphite, solvo-thermal synthesis, reduction of graphene oxide, epitaxial growth on electrically insulating surface, and chemical vapor deposition. In addition, currently 3D graphene can be prepared by chemical vapor deposition and show better conductivity. It shows better electrochemical properties due to large surface area [1, 2]. While graphene oxide (GO) is amphiphilic molecule having both aliphatic and aromatic structural

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arrangement. In addition, GO exhibits both hydrophilic and hydrophobic properties. Hydrophilicity of GO is due to of the presence of oxidized Sp^3 carbon atom in the aliphatic region. While hydrophobicity is because of aromatic region having unoxidized benzene rings having Sp^2 carbon atoms. GO demonstrated chemical functionalization and high dispersion due to oxygen containing functional groups. Due to π-π supramolecular interaction, the aromatic region provides active site for the interactions with other molecules. Because of these intriguing chemical and physical properties, GO is used to prepare composite with other nanostructures [3]. Similarly, Carbon nanotubes are one dimensional (1D) allotropes of carbon having excellent electrical, mechanical and thermal properties. Multi-walled carbon nanotubes have many layer of graphene. Carbon nanotubes have many applications in energy storage and conversion devices [2]. Both these nanoparticles show characteristic properties in Nano composite.

Carbon foam (CF) is a porous spongy material consisting of interconnected network of atoms. It has many attractive properties, such as, light weight, low density, low cost, chemical inertness, high surface area, high thermal stability, adjustable electrical, thermal conductivity, high strength and corrosion resistance [4, 5]. Due to these properties CF have many tremendous applications including oil removal, catalyst support, electromagnetic shielding, as electrode in electrochemical cells, energy storage, water purification, radar absorption-filter, heat exchanger, and as matrix for sensor [6, 7]. Wang et al. examined the effect of clay on the mechanical and thermal conductivity of CF. The outcome showed that when clay is mixed with CF, the mechanical strength of CF increases but thermal conductivity decreases by addition of clay [8]. Moreover, Chen et al. prepared CF by the direct carbonization of melamine foam. He indicated that CF shows low mechanical properties, but desirable electrical conductivities as well as high surface area and high porosity [4]. In another report, Liu et al. studied the effect of MWCNTs on the property of pitch derived CF and found that the electrical conductivity of CF increases with increasing MWCNTs but the thermal conductivity first increases then decreases [9]. Kim et al. synthesized CF from carboxymethyl cellulose (CMC) and Ag, Al and carbon nanotubes (CNTs), also graphene was added in the foam, then studied the effects of these on the thermal conductivity of CF [10]. Kumar et al., grown MWCNTs on CF by using the method of chemical vapor deposition and found that thermal conductivity of CF rised by 75% due to the addition of MWCNTs [11]. In another report, Kumar et al. studied the effect of nanosized iron particles from ferrocene on CF prepared from coal tar pitches and observe that nanosized iron particles work as a catalyst and there is better graphitization of CF. They further observed that the thermal and electrical properties as well as electromagnetic interference shielding effectiveness increases with increasing nanosized iron particles [12, 34]. Yu et al. synthesized ultralight flexible CF from melamine foam using the method of carbonization. They further investigated the effect of temperature on the porosity and thermal property of ultralight flexible CF. They observed that with increasing temperature, the pore diameter as well as thermal properties are badly affecting. With increases pore diameter thermal conductivity increases, so better results could be achieved by modifying the structure [13].

Yadav et al. synthesized high thermal conducting CF by dissolving water slurries of different concentration of mesophase pitches using a template of polyurethanes foam and graphitization is done with the help of different heat treatment. They observed that graphitized CF show better pore structure and excellent thermal conductivity up to 60 W/m K. It also show double specific thermal conductivity compare to copper [14]. Zhang et al. synthesized ultralight CF for achieving high mechanical strength and electromagnetic interference shielding effectiveness by carbonization of phthalonitriles-based polymer. He found that the obtained CF had high compressive strength, low density and high performance [15]. Peng et al. worked to achieve better

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performance of supercapacitor based on ultralight and elastic three dimensional (3D) porous melamine foam (MF)-derived macroporous carbon (3DPMFDMC)/reduced graphene oxide (rGO)/polyaniline (PANI) nanocomposites (denoted as 3DPMFDMC/rGO/PANI) nanocomposites. The 3D MFDMC/ rGO prepared by using the method of carbonization at high temperature and MF immersed in GO and GO reduce. By chemical oxidation polymerization, polyaniline disperse on 3D MPDMC/rGO. They observed that nanocomposite show high electrical conductivity, large surface area, high performance and high efficiency [16, 35]. Meng et al. prepared CF using the Ni as a template and CF deposited on Ni-template. They observe that certain properties of CF enhanced by this method such as porosity, high thermal conductivity and high electrical conductivities [17]. Moreover, Caicedo et al. used the method of blow foaming to synthesize CF from pyroligneous acids, obtained from angustifolia Kunth and CF studied at different carbonization temperatures, and mechanical property investigate by stress strain test, which indicated that CF have high mechanical property [18]. All these Nano particles enhanced one property but at the same time badly affected other properties. More work is required to get the desirable properties.

In this study we have incorporated the various ratios of GO and MWCNTs as additives in mesophase pitches to investigate the effect of GO and MWCNTs on the microstructure of CF composites. The fabrication and reinforcement effect of these additives were further checked in terms of mechanical, thermal and electrical, properties of CF composites.

2. Experiment

2.1 Material

The carbon foam was prepared using coal tar pitches that were provided by Wuhan Steel Corporation limited, China. The physical characteristics of coal tar pitches are given in Table-1. Graphite flakes and the other chemicals used, such as, $KMnO_4$, H_2SO_4, H_3PO_4, HCl, H_2O_2, $(H_3C)_2$ CO and C_2H_5OH, $(C_2H_5)_2O$ were purchase from Sigma Aldrich (Pvt.) Ltd., Australia. Multi-walled Carbon nanotubes were purchased from Tsinghua University, China. Nitric acid and sulfuric acid were used for the purification of carbon nanotubes.

Table.1: Physical characteristics of commercial coal tar pitches.

Soft point	Quinoline insoluble	Benzene insoluble	C/H (atomic)	Carbon yield
(˚C)	(wt. %)	(wt. %)	(ratio)	(wt %)
81	6.55	20.3	1.72	55.1

2.2 Preparation of Graphene oxide

The preparation of GO involve the oxidation of graphite flakes as the method describe by Marcano et al. [19]. In this method H_2SO_4 and H_3PO_4 were used in 9:1. This mixture was mixed with graphite flakes. The KMnO4 in 6 equal portions was slowly added to this mixture. Then mixture was heated at 50˚C for 12 h with continuous stirring. Furthermore, the mixture was cool to room temperature and put on ice with 3ml of 30 % H_2O_2. Then the mixture was passed through

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a metal U.S. Standard Testing Sieve, and then filtered through polyester fiber mixture. The centrifugation of filtrate was done, then by using 200 mL of water, 200 mL of ethanol, and 200 mL of 30 % HCl solid material washed. After every wash mixture passes through U.S. Standard Testing Sieve and filtered by polyester fiber. The mixture obtained so was coagulated by using 200 mL ether and filter by using PTFE membrane. The material dried under high vacuum for an overnight [19].

2.3 Purification of MWCNTs

For the purification of MWCNTs concentrated sulfuric acid and nitric acid were used, in ratio of 3:1 by volume. The MWCNTs was added into mixture of concentrated nitric and sulfuric acid solution. The slurry was mixed by stirring it through ultrasonic machine at 60˚C for 8 hours. The MWCNTs was then thoroughly washed through deionized water until the pH-7 was obtained. The dried MWCNTs resulted after filtration and kept at 90˚C for 10 hours [9].

2.4 Preparation of carbon foam composite

The commercial coal tar pitches were used for the preparation of CFs. First of all raw coal tar was subjected to pretreatment to form mesophase pitches. For this purpose about 120 g of coal tar was taken and grounded to fine powder to get a particle of 70 µm, then heated to 430˚C for 5 h in nitrogen atmosphere. The pretreated pitches were again heated and grounded to fine powder [20]. Then CF composite was prepared by adding different amount of GO and MWCNTs in CF. The slurry of pretreated pitches and nanoparticles was dissolved in ethanol, so that nanoparticles evenly dispersed with pretreated pitches. To remove ethanol, mixture was heated to 80˚C. The dried mass obtained was grounded to obtain 70 µm sizes. Then by adding different amounts of nanoparticles, composite of CF-MWCNTs and CF-MWCNTs-GO were prepared. The blank sample containing only CF was also prepared. For measuring properties, the 1, 2, 4 wt % of MWCNTs were mixed in pretreated pitches to get MWCNTs-CF composite. While for preparing CF-MWCNTs-GO, the MWCNTs and GO were added in additive amounts to the pretreated pitches to form 1, 2, and 4 wt % composites. In reaction vessel containing 3 MPa nitrogenic atmospheres, foaming of MWCNTs-GO-pretreated pitches was carried out. The mixture was heated up to 500˚C for 2h. Then the process of carbonization at 850˚C and graphitization at 2400˚C of CF containing nanoparticles was carried out in nitrogenic atmosphere for 2h and 1 h respectively, with heating rate of 2˚C per minute. CF containing MWCNTs and GO as nanoparticles were obtained cooled and then characterized.

3. Characterization

To investigate the microstructure and morphology of CF containing GO and MWCNTs, Transmission Electron Microscopy (TEM) (Hitachi H-600) was used. The Scanning Electron Microscopy (SEM) (XL30) was used to investigate the cell structure, size of cell and also to investigate the dispersion of nanoparticles in CF. To analyze the crystal-structure information of CF sample X-ray diffractometer (XRD) (D/Max 2500 V PC-1, Cu-Kα radiation) was used with the scanning rate of 2/min. Mechanical properties, such as, compressive strength and young's modulus were measured by Universal Testing Machine having crosshead speed of 5 mm min^{-1} and

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load cell of 100 N. Sample was compressed between two stainless plates. The compressive yield strength (σ) was calculated by using the following equation:

$$\sigma = F/A \qquad \text{Eq. (1)}$$

In this equation **"F"** is the load at yield and **"A"** is area of cross section. Similarly, thermal conductivity was measured by Netzsch LFA 457 conductivity tester, which uses laser flash technique. Test were performed between 25 and 900˚C. Thermal conductivity (λ) was measured by following equation

$$\lambda = \rho C \alpha \qquad \text{Eq. (2)}$$

Where **"ρ"** is the sample density, **"C"** is heat capacity and **"α"** is the thermal diffusivity. The electrical conductivity was measured by using four-probe resistance meter (SX 1944, china). The room temperature electrical conductivity was calculated by taking reciprocal of electrical resistivity. And electrical resistivity was measured by four-probe method. The both sides of sample were measured and two measured values were averaged.

4. Result and discussion

4.1 Transmission Electron Microscopy

Transmission electron microscopy was used to investigate the particles size and their average range. For removing impurities on MWCNTs, acid treatment was performed. **Fig-1a** shows the TEM result of purified MWCNTs. Due to impurities present on CNTs there are clusters of MWCNTs that reduce after acid treatment and fine arrangement of nanofiller obtained in nanometer range. **Fig-1b** shows hybrid composite of GO and MWCNTs in CF. TEM result shows that GO and MWCNTs are uniformly dispersed in CF. This uniform dispersion of GO and MWCNTs have great impact on the properties of CF [9].

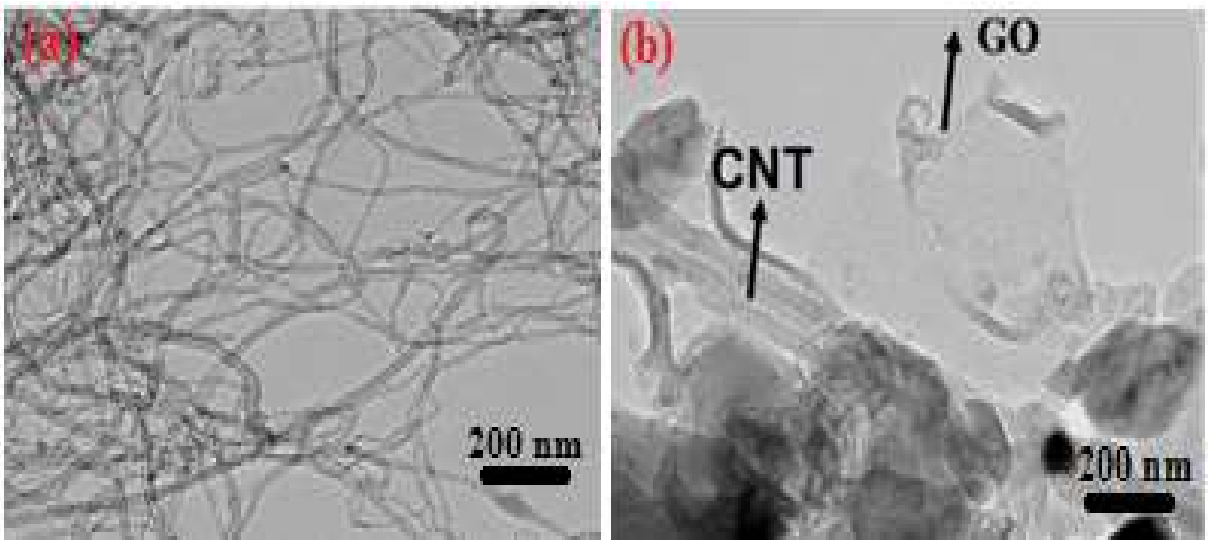

Fig-1: TEM results of purified MWCNTs (a) and hybrid MWCNTs and GO dispersed CF composite (b).

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4.2 Scanning Electron Microscopy

The scanning electron microscopy was used to examine the morphology of CF and the dispersion of nanoparticles in CF. **Fig-2** Shows the SEM images of CF **(a)** and SEM images of CF after addition of GO and MWCNTs **(b)**. **Fig-2a** shows the CF without any nanoparticles. Cell size and structure of CF are not uniform in pure CF. **Fig-2b, c, d** shows the SEM images of 1, 2 and 4 w % of MWCNTs in CF. It can be observed that after the addition of MWCNTs in CF, cell size and structure becomes more organized and uniform. The MWCNTs act as cracks barrier that lead to reduction of cracks in structure of CF. **Fig-2e, f, g** shows the 1,2,4 wt % CF/MWCNTs-GO hybrid composite. It can be seen that prominent changes occurs in microstructure of CF after the addition of MWCNTs and GO in CF. Due to uniform and homogeneous dispersion of MWCNTs and GO in CF, cellular structure become more uniform and spherical, also imperfections greatly reduced. The reason behind this, is the reduction of viscosity of pretreated pitches during foaming process [33]. When MWCNTs and GO introduce in CF viscosity of pretreated pitches reduced at the foaming temperature, as a result bubble easily grows, combine and uniform CF structure form. SEM results shows that MWCNTs and GO greatly improved the morphological behavior of CF such as cellular structure, porosity, size and well organize homogeneity [9, 20].

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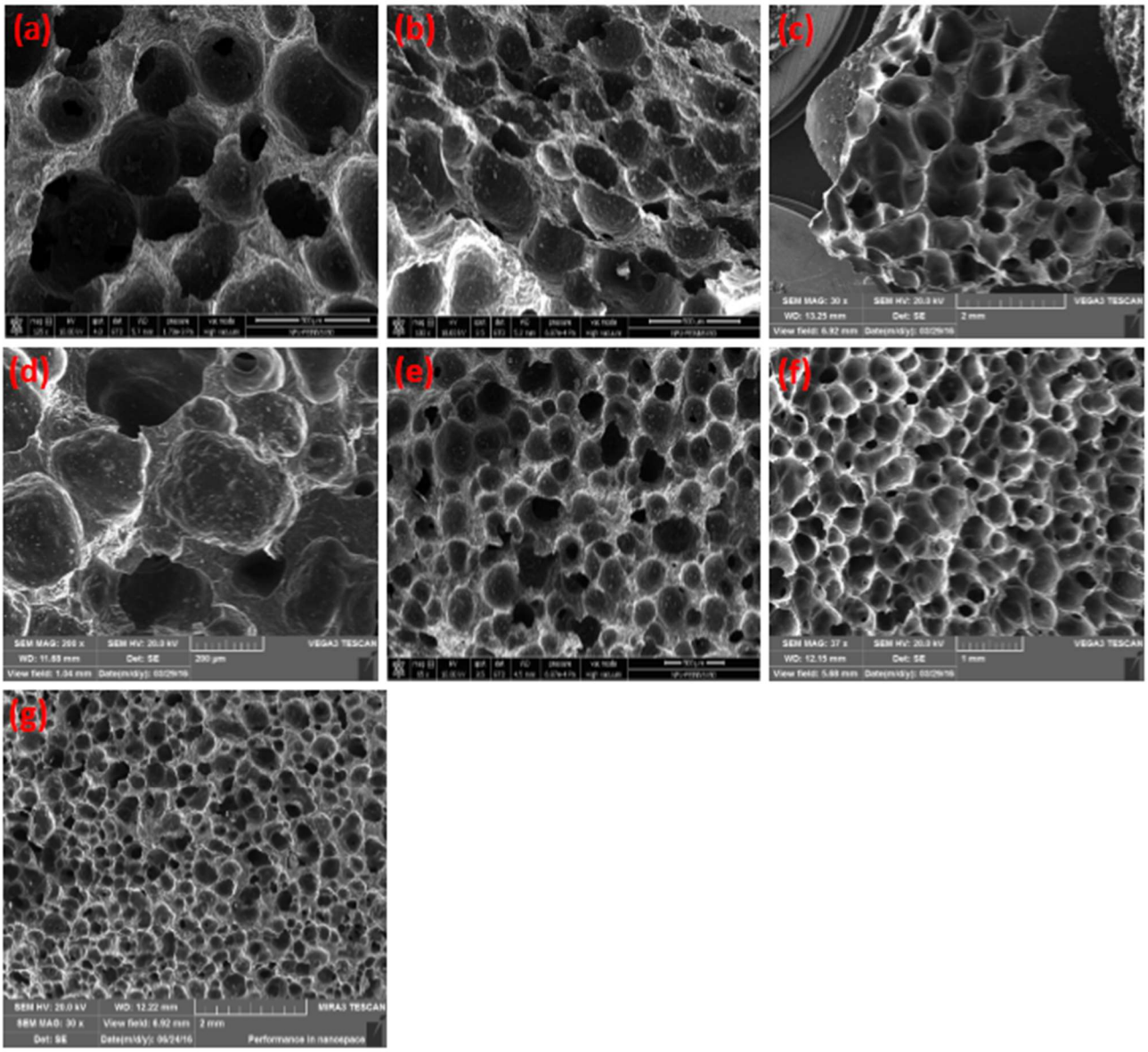

Fig-2: SEM images of pure CF (a), 1, 2, 4 wt. % CF/MWCNTs (b, c, d), while 1, 2 and 4 wt. % CF/MWCNTs-GO hybrid composites (e, f, g).

4.3 X-ray Diffraction

The XRD technique was used to examine the crystal structure, phase, purity and peak shift upon the formation of hybrid composite. **Fig-3** shows the XRD Spectra of pure CF, CF/ MWCNTs and CF/MWCNTs/GO. Pure CF shows peak 27.5˚, 44.5˚, 56.3˚, 78.5˚corresponding to planes 002, 101, 004, 110 which confirm the crystalline structure of CF. The XRD of CF/MWCNTs shows more intense peak which shows that the crystal structure much more improved than pure CF [21]. While CF/MWCNTs/GO shows high intensity peak which shows MWCNTs and GO more improve CF crystal structure. There is one additional peak at 11.5˚corresponding to plane 001 which shows the oxygen-containing functional group in GO [22].

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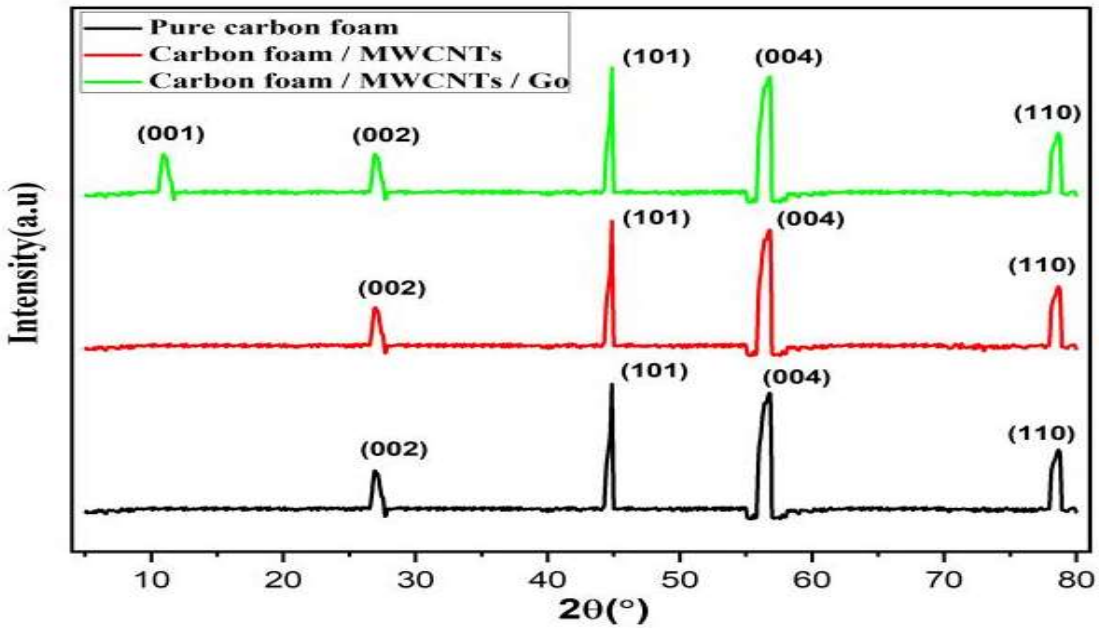

Fig-3: shows the XRD spectra of carbon foam with different amount of GO/MWCNTs.

4.4 Mechanical properties

The Mechanical property of CF composite containing GO and MWCNTs as additive, were observed in term of compressive strength and young's modulus. **Fig-4 (a) and (b)** shows the compressive strength and Young's modulus values of CF/MWCNTs and CF/MWCNTs-GO composites. It can be observed from the **Fig-4(a)** that compressive strength of CF becomes much improved after the addition of 1 to 4 wt. % MWCNTs and GO. **Fig-4 (a)** clearly reveals that initially the compressive strength of pure CF was found 6.8 MPa while after the additive amount of MWCNTs reaches up to 2 wt. %, maximum compressive strength of 15.3 MPa was observed. The main reason for increase of compressive strength of CF after addition MWCNTs is due to the modification of MWCNTs that hinder the propagation of cracks in cell structure and improve interfacial bonding. Moreover, further addition of MWCNTs reduces the compressive strength due to cluster formation. It can also be seen that, when both MWCNTs and GO are synergistically introduce in CF matrix maximum compressive strength of 19.2 MPa resulted. This indicates that both MWCNTs and GO enhances the compressive strength of carbon foam [11, 20]. Furthermore, **Fig-4(b)** indicates the mechanical properties of CF composites containing various loading of MWCNTs and GO as additives in term of Young's modulus. The pure CF shows only 12 MPa of Young's modulus. After the addition of MWCNTs maximum value of Young's modulus was observed and the value of 40 MPa was observed when 2 wt. % MWCNTs were added. After that MWCNTs and GO synergistically introduce and value reaches to 56.8 MPa when 2 wt. % of MWCNTs-GO were applied. This highest value of Young's modulus shows that very high energy is needed to break CF composite having MWCNTs and GO as additives [23, 24].

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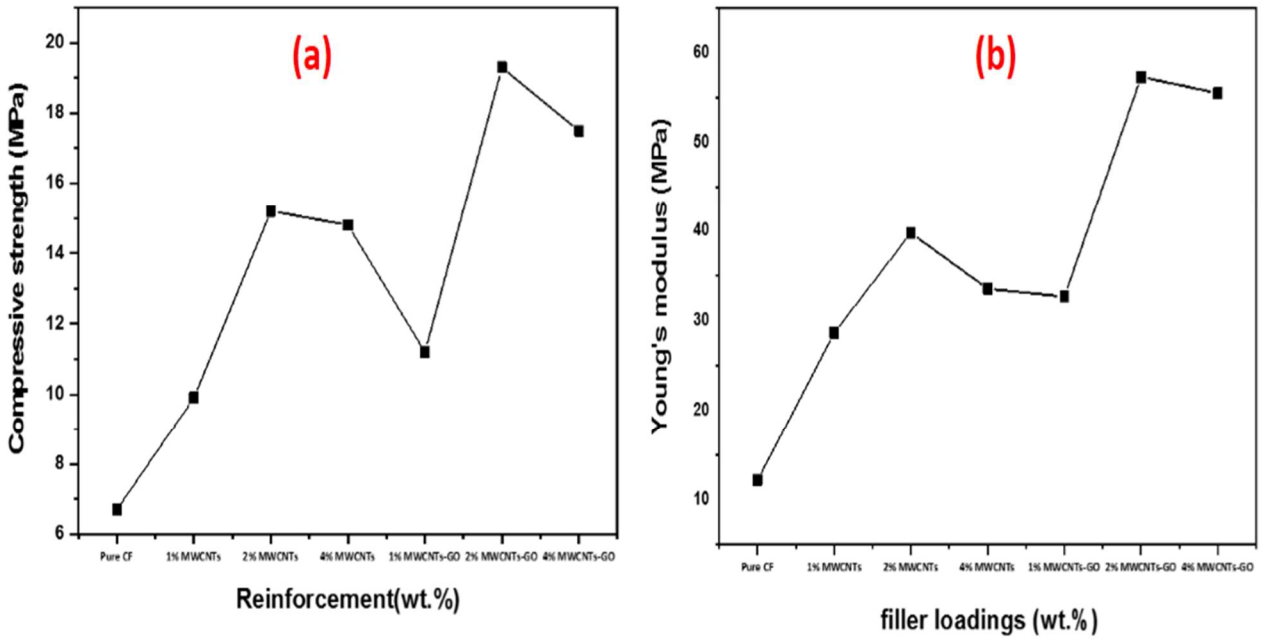

Fig-4: Compressive strength (a) and Young's modulus (b) of CF composites having variable reinforcements amount.

4.5 Thermal properties

Fig-5 shows the thermal conductivity of CF containing different amount of GO and MWCNTs as additive. As the temperature is rises up to 900 ℃ with loading of GO and MWCNTs the thermal conductivity of carbon foam enhances. It can be clearly observed that thermal conductivity of pure CF is low. Maximum thermal conductivity of 28.93 W/m K is observed when there is a loading of 2 wt.% MWCNTs at a temperature of 900 ℃. While 4 wt. % MWCNTs shows low thermal conductivity due to non-homogeneous dispersion of MWCNTs in CF. The maximum thermal conductivity of 30.91 and 29.1 W/m K was shown by CF containing 2 and 4 wt % of GO and MWCNTs as additives. The incorporation of GO along with MWCNTs enhance the thermal conductivity of CF due to the excellent thermal properties and homogeneous dispersion of nanofiller. This study shows that CF shows excellent thermal conductivity when GO is used synergistically with MWCNTs [25, 26].

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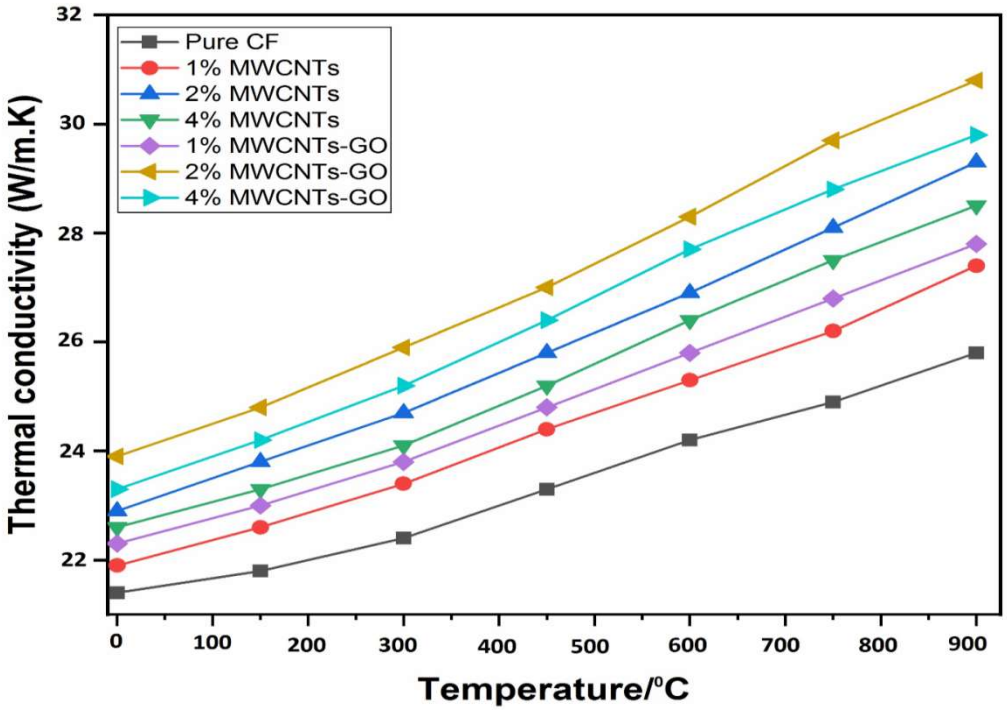

Fig-5: Thermal conductivity of CF composites with various nanofillers loadings.

4.5 Electrical properties

Fig-6 shows the electrical conductivity of CF at various concentrations of nanofiller. It can be seen that electrical conductivity of pure CF is 7.1×10^3 S/m which shows that pure CF is poor conductor. After the addition of MWCNTs electrical conductivity increase to several order of magnitude. The result shows that maximum electrical conductivity of 22.1×10^3 S/m was observed when 2 wt. % MWCNTs added as additive amount. This enhancement in electrical conductivity is due to the conduction path of electron [31, 32]. After that electrical conductivity deceases due to nonhomogeneous dispersion of MWCNTs. **Fig-6** also shows that when GO added in additive amount along with MWCNTs the electrical conductivity enhanced up to 27.2×10^3 S/m due to core structure and surface layer that function as charge transfer channel to improve electrical conductivity of CF composite. This electrical conductivity result indicates that consolidation of GO along with MWCNTs had much better effect on electrical properties of CF composites [27-30].

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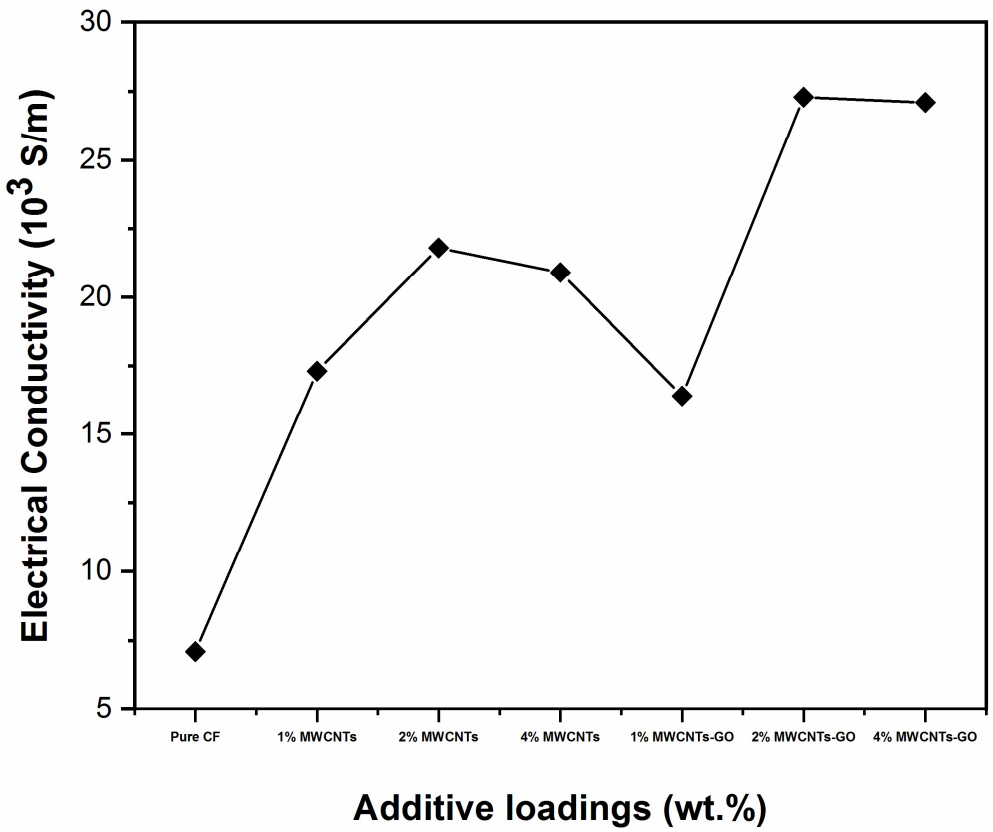

Fig-6: Electrical conductivity of CF composites at various amount of nanofillers contents.

5. Conclusion

In this work preparation and properties of carbon foam (CF) containing additive amount of MWCNTs and GO were studied. The microstructure investigation showed that both MWCNTs and GO has significant impact on the pore size and cell structure of carbon foam. Result revealed that electrical, mechanical and thermal properties increases gradually with increasing nanofiller additive content. The maximum compressive strength and young's modulus of 19.2 and 56.8 MPa respectively were shown by CF at 2 wt % MWCNTs-GO as additive. In addition, the highest thermal conductivity of 30.91 W/m.K at 900 ° C and electrical conductivity of 27.2×10^3 S/m was observed at 2 wt % MWCNTs-GO loading.

Deceleration of Interest Statement

On behalf of all authors, the corresponding author declared that this work is the original work of authors and all standards were followed accordingly and there is no conflict of interest

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