

A brief review on preparation and application of MWCNT-based polymer nanocomposites

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Abstract

Technological advancement always seeks new materials with improved functional properties, particularly for smart applications. In this regard, nanotechnology is offering today wide range of novel material designs fabricated by compounding nanofillers into the polymer matrix. Different allotropic forms of carbon can reinforce the properties of polymers for various applications. Reinforcement depends on the dimension, shape, size and compatibility of the nanofiller with the polymer matrix. Chemical modification of filler surfaces and the matrix can selectively localize the filler in the hybrid composites in the desired phase or at the interface by melt mixing or solution casting method, during compounding procedure. In this regard, the conducting nature of the addition of multiwalled carbon nanotubes (MWCNTs) into a polymer matrix fosters the conductivity into the materials. Such nanocomposites can be used for numerous applications such as conducting materials, super-capacitors, light emitting devices, medical purposes etc.,. This review paper focuses on different methods of preparation of MWCNT/polymer nanocomposites, their surface properties, and microbial properties etc.,.

Keywords

Multiwalled carbon nanotubes (MWCNT); Polymer composites; Conductivity; Mechanical strength; Antibacterial properties.

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1 Introduction

The advancing world is the gift of nanotechnology which deals specially with nanomaterial and

nanocomposites. They are multiphase materials in which one or more phases of material is in nanoscale dimensions below 100 nm [1–3]. Generally, a nanocomposite material has dispersing phase

called a matrix and a dispersed or reinforcing phase called a filler. These nanoscaled fillers are embedded in a continuous phase of polymer, metal or ceramics matrix [4]. Almost all varieties of polymers are used as matrices such as thermoplastics; polyurethane (PU) [5], polyethylene (PE) [6], polyvinyl chloride (PVC) [7], thermosets; epoxy polyimide [8] and phenolic resin [9] and elastomers; polybutadiene (PB) [10], ethylene-octene copolymer (EOC) [11]. These fillers may be of 0 dimensional (0D), one-dimensional (1D), two-dimensional (2D) and three-dimensional (3D). 3D particles are cubical or spherical structures spread in x, y and z directions for instance boehmite [11], silica [12] and different kinds of particle. 2D particles are sheet-like layered particles spread in x and y directions for instance graphite. 1D particles are tubular or cylindrical in the structure such as single-walled (SWCNT), multiwalled carbon nanotubes (MWCNT) whereas 0D are the quantum dots which are very minute particles comprising few atoms such as fullerenes, polyhedral oligomeric silsesquioxane (POSS) etc., [9–13].

The filler and matrix phases together generally give the synergetic effect to the composites which would be novel, intense and cumulated. The properties of the composites depend on the types of variables i.e., filler, their nanoscale dimension, loading degree, degree of dispersion, their size, shape, orientation and interaction with the matrix.

Surface area to volume ratio (A/V) with aspect ratio [a = ratio of length (l) and diameter (d)] of filler gives a good relation for the reinforcement in the composite which has been depicted in Fig. 1, in which the change in the aspect ratio of a circular plate with A/V ratio was illustrated. The periodic increase in l of the platelets changes the molecule to a cylindrical form. In region A the platelete on increasing l , structure changes from 1 to 2 and 3. Stage 3 is the one when its $l=d$ and its aspect ratio becomes 1. Further increase in l can be seen in region B, where the structure converts to filamental fibrous 1-dimensional structure, stage 4 [14]. Fibers (such as carbon nanotubes, 1 dimensional) and platelets (such as layered silicates clay, 2 dimensional filler) have higher A/V ratios so they have high reinforcement ability [15]. Therefore increasing order of reinforcement can be illustrated with respect to A/V ratio as $1 > 2 > 3$ [11].

Depending upon the particle size of the filler, electrical, thermal, and optical properties of the nanocomposites can be varied. The interacting phases can even alter the mechanical property of the composite by varying the modulus and strength [16]. Fig. 2 and Fig. 3 show the expanded interfacial surface of the filler to interact with the matrix [14]. The spherical fullerence being 3-dimensional has less tendency for agglomeration, less likely is

for tubular MWCNT and sheet of graphite has least reinforcement and has possibility of agglomeration [17–19].

The nanofillers mostly in practice are aluminosilicates (clays) [20], carbon nanotubes (CNT) [8, 21], nanofibres [22], ultra-dispersed diamonds (nanodiamonds) [23], fullerenes [24], other inorganic nanotubes, nanoparticles of silicon oxide (SiO_2), calcium carbonate (CaCO_3), metal nanoparticles as well as fibers from biological resources. Different allotropic forms of carbon have various effects in the matrix mostly for their strength, conductivity and optical property. Boeing: 787 Dreamliner, the aircraft is an example of mechanical reinforcement. It was made up of carbon fiber-reinforced epoxy resin. Here carbon fiber induced strength and stiffness to the material and epoxy binds the fiber with significant less weight [25]. The conducting materials form the important base of industries in information technology, electronics and electrical industries etc., [26]. Conducting fillers in polymers result the conductive composites which have low density, low weight, high mechanical performance, low cost and easy processability such as biobased polymer (from polylactic acid (PLA) and potato starch) and CNTs are one of the most interesting materials with high strength [8, 27].

The sp^2 hybridization of the carbon in CNT's form an allotropic tubular structure that can be termed into diverse architectures to form SWCNT, MWCNT, nanobud etc. Such carbon materials are conductive even in little amount and open different avenues of applications as polymer composites. Therefore, numerous researchers are in the process throughout the world to achieve optimum best-conducting polymer composites for advanced applications. MWCNT has been incorporated into different polymers such as ethylene vinyl acetate copolymer (EVA) [28], PU block [29], polystyrene (PS)/polyvinylidene fluoride (PVDF) composites [28], polyolefins [29] and polyesters [30–32]. In these systems, electrical-conducting properties, thermal, morphological, mechanical and dielectric properties are enhanced. MWCNT also worked as a compatibilizer between PS and PVDF [30].

The allotropes of carbon/polymeric nanocomposites have significant applications in numerous field nowadays. They can be used as antistatic materials [33–35], super capacitors [29, 35] highly conductive application (electrodes in touch screens and sensors) [28, 36, 37] security application, screen printing, coating materials, organic solar cells [29], printing electrodes, conductive adhesives, current limiting devices, shielding for electromagnetic protection, heater with distributed heat emission and self-regulated heaters [27, 29]. Similarly light emitting device, contact bottoms of computers, laptops, mobiles, I-pads and media techniques etc. X.

Zhao et al., studied carbon black nanoparticles in polystyrene (PS)/polyvinylidene fluoride (PVDF) composites which induce dielectric property. [34] S. B. Kondawar also mentioned that conducting nanocomposites are the noble electrodes for the application as super capacitor [32]. Electronic tattoo are also some noble application of such nanocomposites [38, 39].

2 Preparation of conducting polymer nanocomposites

There are many techniques for compounding polymer matrix with nanofiller. Some of the techniques are explained here [32].

2.1 Solution casting

This is a technique where both polymer and filler are homogeneously dissolved in a solvent separately and mixed followed with sonication. The mixer solution is sonicated and cast to a desired shape and the solvent will be slowly evaporated to get a thin film of the composite [38, 40].

2.2 in-situ polymerization

This is a technique by which the first polymer was synthesized. During the synthesis of polymer, at the stage of prepolymer formation fillers are incorporated and stirred well, stabilized and moulded to the desired shape. Usually, the process is applicable for co-polymer with bigger units [37].

2.3 Internal melt mixing

The dried polymer and fillers are blended into the rotors with specific rotation per minute (rpm), temperature and for a specific time to homogeneous compounding. Depending upon the type of polymer and composites they are pre-cured and post-cured in an oven at a specific temperature. The stable composites are moulded with hydraulic pressure to obtain a plaque [18, 32, 33].

2.4 Twin screw extruder

In this technique, dried polymers and fillers are premixed and put into a conical counter rotating a Brabender with the specific speed (round per minute, rpm) of twin extruders at a higher temperature below the melting point. Composites prepared are cooled with ice water to pelletized or prepared the plates with hydrolytic pressure in the specific diameter of moulds [41–46].

Except for the process described above Peng et al., 2008, explained the chemical synthesis, electrochemical deposition and co-deposition methods

to prepare conducting polymers with carbon nanotubes. These composites have a high charging/discharging ratio so were applicable to prepare supercapacitors, batteries and fuel cells. [36]. material on the GCE electrode and (V) represent the operating voltage.

3 Morphological Features of MWCNT-based Nanocomposites

The morphology of the nanofiller has many effects on the nanocomposites. Their dispersion and interaction with the matrix play a very important role in the mechanical and chemical properties of the materials. Morphology of the composites can be investigated by different techniques; such as Scanning Electron Microscopy (SEM), Optical Microscopy (OP) and Transmission Electron Microscopy (TEM). These techniques are efficient to locate shape, size, distribution of filler and interaction with the matrix.

The expanded graphite has better conductivity in comparison to untreated graphite in composites. This could be due to continuous sea-weeds phase in expanded graphite where as untreated graphite has flakes like interrupted structure [28]. Functionalization of the filler and matrix would increase compatibility between filler and matrix. SEM micrograph of poly(butylene adipate co-terephthalate) (PBAT)/MWCNT-OH shows good dispersion of MWCNT in PBAT polymer which becomes more intense in acrylic acid grafted PBAT. The TEM micrograph of tensile fracture also supports MWCNT-OH get dispersed well where as MWCNT forms agglomerates on 3 wt. % loading of filler in PBAT matrix [31]. Feller et al., also explained SEM micrograph of acrylic acid grafted PBT with OH-functionalized graphene oxide upto 3% shows good dispersion of nanofiller [15, 38]. The exfoliated CNT has conductive network, assigned for the flexible continuous phase of PS matrix [47].

C-X Liu and J-W Choi noticed dispersion of CNT above 1 % cannot be investigated by TEM due to formation of opaque nanocomposite where as SEM can explain uniform dispersion of MWCNT throughout the fracture surface. SEM micrograph of polydimethylsiloxane (PDMS)/CNT upto >5 wt. % fillers showed good dispersion with consistent clusters up to 3 μ m [29].

Polyaniline (PANI)/CNT and polypyrrolidine (PPy)/CNT nanocomposites have CNT coated with homogeneous polymer and formed coralloid network [36]. N. G. Sahoo et.al. observed smooth lattice structure of carbon- carbon bond in TEM image of MWCNT where as some defects are seen in COOH group functionalized MWCNT due to COOH group hanged on CNT surface. The COOH group of CNT is forming ester bond with OH group of polyurethane matrix [37].

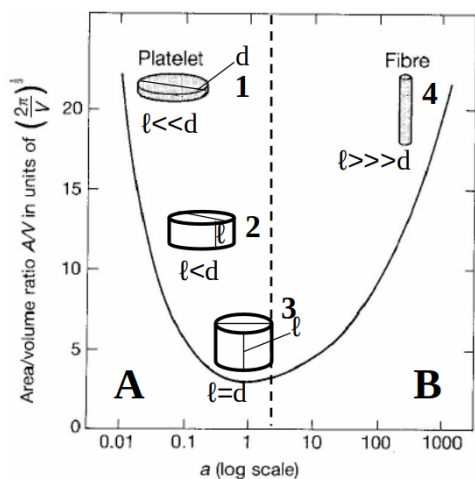


Figure 1: Surface area to volume ratio A/V of a cylindrical particle of a given volume, plotted versus aspect ratio $a = l/d$ [13, 14].

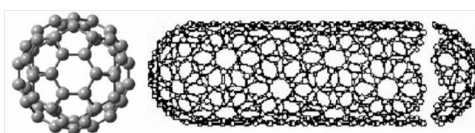


Figure 2: Ideal structures of a fullerene (left) and a carbon nanotube (right) [14, 19].

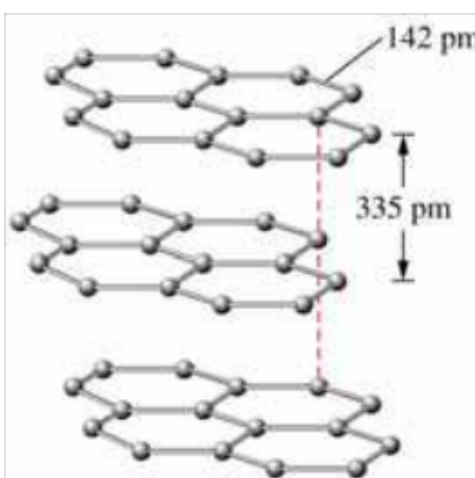


Figure 3: 3D structure of graphene layers in graphite [14, 19].

The SEM micrograph for transverse section cut perpendicular to extrusion direction of PBT60/EEA-CB40 nanocomposite show phase organization where holes and tunnels in black correspond to conductive phase containing carbon black and Poly(ethylene-co-ethyl Acrylate) [15].

The selective localization of carbon black (CB) was observed in morphological study of diphasic PS/PVDF/CB composite with continuous phase of PS by X. Zhao et al., [30]. The interfacial connection between PS and PVDF was not clear in SEM micrograph [28]. Similar type of phase separation

can also be observed in Fig. 4 with two immisible phases of PLA/PBAT/MWCNT nanocomposites. On increasing PBAT composition, CNT migrates towards PBAT phase from PLA although there is no chemical bond reported [43]. MWCNT in PLA/MWCNT composites can also increase chunks and striation in nanocomposites [42]. The MWCNTs also increase roughness and hydrophobicity on the surface of the polymer composites which can be seen in the SEM images of polyhydroxy valerate (PHBV)/MWCNT. The micrographs of nanocomposites of MWCNT varying from 0 %, 1 %, 1.5 %

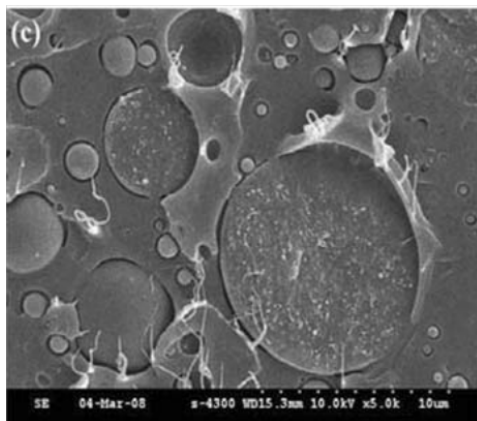


Figure 4: SEM Image of PLA/PBAT/ MWCNT nanocomposite [19].

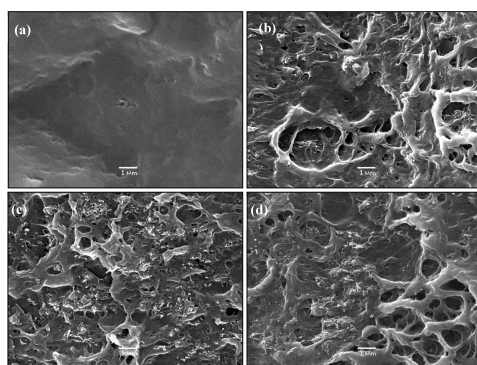


Figure 5: SEM micrographs showing fracture surface PHBV/MWCNT with (a) 0 %, (b) 1 %, (c) 1.5 % and (d) 2 of MWCNT [40].

and 2 % in PHBV show increasing roughness respectively as shown in Fig. 5 (a, b, c and d). The 0 % of MWCNT in nanocomposites found to have smooth surface where as MWCNT in PHBV found to have homogeneous dispersion of MWCNT in matrix and are incompatible to matrix resulting voids into the nanocomposites. The voids cause roughness and increasing with MWCNT see Fig. 5 [40].

The data is further supported by the increasing contact angle value for surface of same nanocomposites as shown in Fig 6a. The increase of $\theta = 55^\circ$ for pure PHBV to 87° on 1 % loading of MWCNT is a significant rise in hydrophobicity. Moreover, the smooth and rough surfaces show a similar trend and significance on rough parts of the nanocomposite surface as shown in Fig 6b [40].

4 Mechanical performance and thermostability of MWCNT-based Nanocomposites

Polymers are class of materials in which one can induce or reinforce any properties such as mechanical, morphological or conductivity. In this regard, different reinforcing inorganic materials such as MWCNT alone or in combination with other materials such as carbon blacks, graphenes, nanodia-

monds, hydrogenated CNT etc., can be incorporated into polymer materials [48, 49]. Fig.7 clearly shows the reinforcing effect of increasing wt.% of MWCNT in polycarbonate. The Young's modulus is increasing with increasing concentration of MWCNT [50].

Other properties like charge barrier, and conducting property can be induced into the polymer matrix by the dispersion of conductive fillers such as CNTs or polymers intrinsically conductive [38, 40, 47]. The practices of researchers are with different types of polymers molded with CNT or conducting polymer to reinforce the mechanical, thermo mechanical properties and conductivity of nanocomposites.

Mixing a filler into the polymer may be a simple technique but the major challenge for the scientist is to make them compatible with the polymer. In comparison to neat filler, functionalized fillers of respective polarity have more good compatibility. N. G. Sahoo et al., synthesized polyurethane and incorporated COOH-functionalized MWCNT by situ polymerization in a urethane matrix. The composite has good dispersion and adhesion of COOH-MWCNT on polyurethane matrix with pronouncing advances thermomechanical property [15]. The

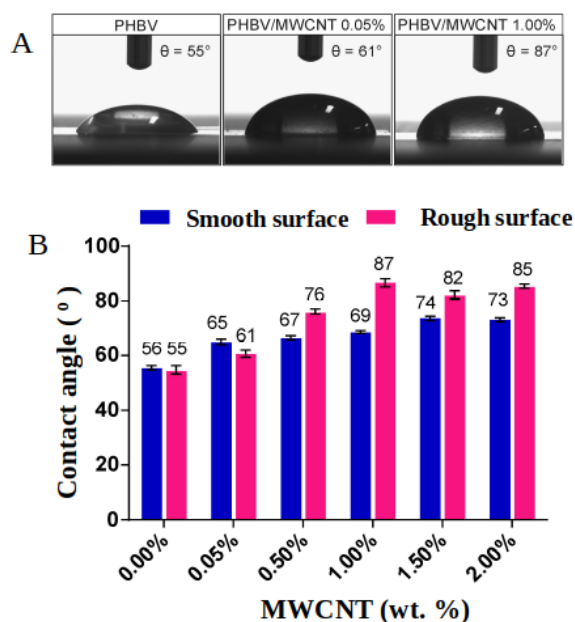


Figure 6: (A) Photographs showing increasing hydrophobicity with increasing MWCNT in PHBV/MWCNT nanocomposites, (B) the bar diagram indicating the effect of increasing contact angle value on increasing MWCNT in PHBV [40].

electrical conductivity in PS/MWCNT composite can be reinforced by the 2 wt-% of loading CNT [47].

5 Electrical Conductivity of MWCNT based Nanocomposites

Carbon material is one of the filler with wide conducting ability. Composites of conducting filler with polymers results in conductive polymer nanocomposite which have low density, low weight, high mechanical performance, low cost and easy processability. Carbon materials carbon black, carbon fibers, carbon nanotubes (CNTs) have conductivity value (σ) ranges from 101 S/cm, 103 S/cm), CNTs are one of the most interesting materials with high strength. The sp^2 hybridization of the carbon form allotropic tubular structure and these structure modified to form single walled carbon nanotubes (SWCNT), multiwalled carbon nanotubes (MWCNT), nanobud etc. Such carbon materials are conductive even in little amount and opens different avenues of applications as composites with polymer. Therefore numerous researches are in the process throughout the world to achieve optimum best conducting polymer composites for different advance applications. MWCNT has been incorporated into different polymers such as ethylene vinyl acetate copolymer (EVA) [28], polyurethane block [29], polystyrene (PS)/polyvinylidene fluoride (PVDF) composites [30], polyolefins [31], polyesters [18, 32] and PLA [51] In these system electrical-conducting properties, thermal, morphological, me-

chanical and dielectric properties are enhanced. MWCNT also worked as compatibilizer between PS and PVDF [?].

The electrical resistivity in Fig. 8 of the PBAT, acrylic acid grafted PBAT with MWCNT and MWCNT-OH composites were measured directly on laminated films (0.1 mm thick) with an Ohm-Stat RT-1000 resistivity meter. The significant decrease in resistivity with the addition of conducting fillers MWCNT and MWCNT-OH by 1 wt-% can be seen in Fig. 8. The electrical resistivity decreases from $1 \times 10^{16} \Omega/\text{sq}$ to $1 \times 10^{10} \Omega/\text{sq}$ differing by 1×10^6 times for PBAT/MWCNT. Similarly, 1 wt-% addition of MWCNT-OH in PBAT-g-AA decreases to $8 \times 10^7 \Omega/\text{sq}$. The difference in resistivity between 1 wt-% and 3 wt-% loading of MWCNT and MWCNT-OH is $1 \times 10^2 \Omega/\text{sq}$ approximately. On adding 5 wt-% of filler the resistivity decreases more to $1 \times 10^1 \Omega/\text{sq}$. Therefore upto 3 wt-% of MWCNT and MWCNT-OH sharply reduce the resistivity where as 5 and more wt-% loading of MWCNT and MWCNT-OH does not reduce in the appreciable amount. Moreover, the PBAT-g-AA/MWCNT-OH composites have more reduction in resistivity in comparison to PBAT/MWCNT. This might have happened due to the ester bond formation between acrylic acid in PBAT and OH-group in MWCNT and ester bonds are always stronger than the hydrogen bond which is in between PBAT and MWCNT [32].

Biodegradable conducting nanocomposites with PLA and MWCNT with increasing concentration of CNT in composite from 0 wt-% to 1.5 wt-% found

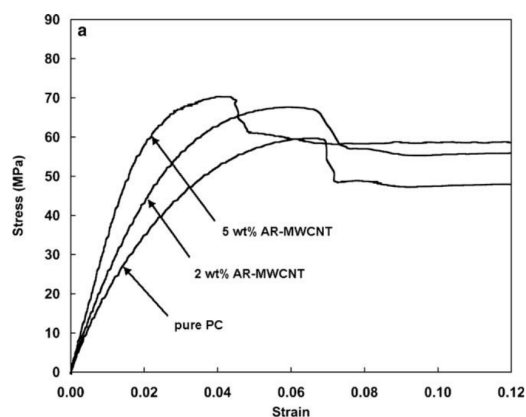


Figure 7: Stress-Strain curve showing reinforcement in polycarbonate by filling 2 wt-% and 5 wt-% MWCNT [50].

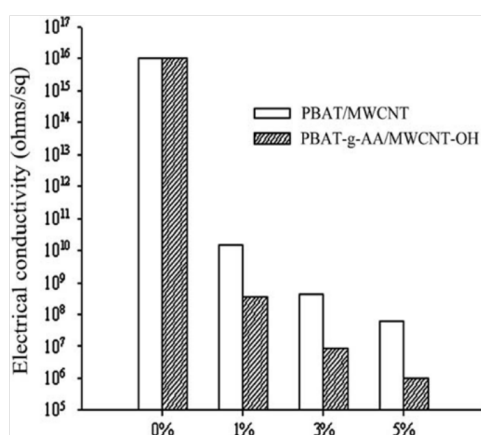


Figure 8: The electrical resistivity of PBAT, PBAT-g-AA, PBAT/MWCNT, and PBAT-g-AA/MWCNT-OH composites [30].

to increase crystallization, thermal degradation and at the same time reported drop in surface resistivity [41]. C.S. Wu and H.T. Liao modified poly (butylene terephthalate) (PBT) by surface grafting with acrylic acid and graphene oxide was modified to acid chloride (GO)-COCl derivative. Composites of such materials have found improved thermal and decreased with graphene oxide (GO) and resistivity property and antistatic property [33].

6 Antibacterial properties

Dispersed CNT's in nanocomposites can induce antibacterial properties. As carbon is inorganic filler spread throughout the matrix renders the bacterial growth showing by increasing hydrophobicity as mentioned above and induce antibacterial property in nanocomposites [40].

Fig. 9 (A) shows the decrease in survival of bacterial colonies on fixed loading of *E. coli* (0.1 mL) with respect to fixed volume and time during incubation with the PBAT-g-AA/MWCNT (3 wt-%). *E. coli* colonies grow on incubation, after

3 hours slowly the composite inhibits the growth of *E. coli*. In Fig. 8(B), 12 hours incubation almost all colonies are disappeared [32]. The infectious bacterial growth of *E. coli* can be observed in the prepared composites of C.S. Wu. The *E. coli* cell count with respect to the exposure time of the composites with bacteria was given in the Fig. 8 A and B. The PBAT and PBAT-g-AA resist the growth of *E. coli* whereas the increase in % incorporation of MWCNT and MWCNT-OH not only decrease in growth of *E. coli* but also suppress the growth of bacteria in the culture to almost zero cell count.

Fig. 10 shows the survival ratio of *E. coli* in contact with the PBAT or PBAT-g-AA surface. Exposure of composite with bacteria upto 6 hours doesnot hinder the growth but after 6 hours exposure result concluded that increase in % of MWCNT and MWCNT-OH in composites started to decrease survival ratio of bacteria showing antibacterial effect of the composites [32]. However, enhanced biodegradation was also observed in PBAT/MWCNT nanocomposites compatibilized with ZnO

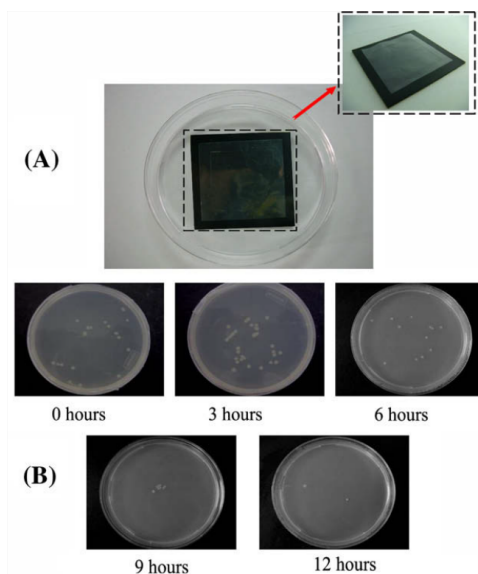


Figure 9: (A) Photographs show PBAT-g-AA/MWCNTs (3 wt. %) samples loaded with a fixed volume (0.1 mL) of *E. coli*. (B) *E. coli* was exposed to PBAT-g-AA/MWCNTs (3 wt.%) for evaluation of antibacterial activity [32].

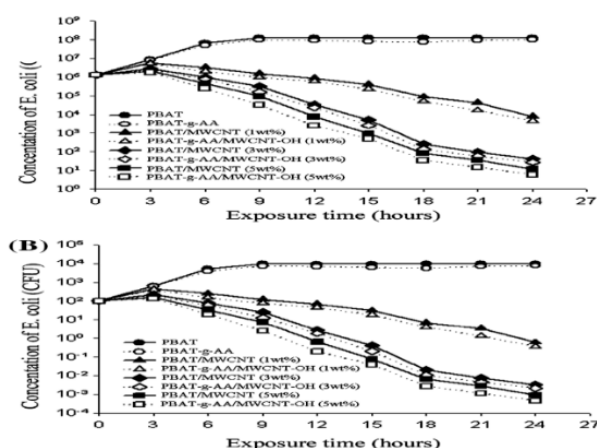


Figure 10: Exposure survival ratio of *E. coli* cells during exposure to PBAT or PBAT-g-AA and its composite surfaces [32].

in more than 0.2 %, indicating bacterial degradation.

7 Conclusion

Carbon in different allotropic states a nanometric dimension of up to 100 nm becomes the nanofiller where the aspect ratio provides an interacting surface for the polymer matrix in the nanocomposite. Nanocomposite can be prepared by simple techniques of compounding. Filler-matrix rearrangement can be investigated by different spectroscopic methods like SEM, optical microscopy (OM), TEM etc., which confirms that significant reinforcement, is achieved only when filler and matrix are compatible with each other. The nano-carbon materials such as MWCNT, nanodiamond, and nanoscopic

graphite can be used to reinforce mechanical properties and thermostability and at the same time are capable of producing synergetic effects in their nanocomposites to reinforce mechanical properties, induce electrical conductivity as well as other functionalities in the different kinds of polymers. All such properties are boon for material science and engineering making the composites nanomaterials applicable in numerous fields in developing novel devices and technologies.

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ing carbon based polymer composites.

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