Role of Metallic and Bimetallic Modified Carbon Nanotubes in the Formation of Polymethyl Methacrylate Composites

Farrukh Bashir[1*], Palwasha A. Hakeem[1], Tajamal Hussain[3], Ayesha Mushtaq[1], Sabeena Rizwan[1], Uzma Jabeen[1], Musarat Riaz[1], Farida Behlil[1], Huma Tareen[1], Mohammad A. Raza[2], Zahid Mahmood[4], Khurrum S. Buzdar[2]

[1] *Faculty of Basic Sciences, Sardar Bahadur Khan Women's University Quetta, 87300, Pakistan.*
[2] *Pakistan Council of Scientific and Industrial Research Laboratories, Complex, Quetta, 87300, Pakistan*
[3] *Institute of Chemistry, University of the Punjab, Lahore 54590, Pakistan*
[4] *Pakistan Council of Scientific and Industrial Research Laboratories, Complex, Lahore 54600, Pakistan*
**Correspondence author: farrukh_chem@yahoo.com*

ABSTRACT

In the realm of nanotechnology, polymer composites, being lightweight and transparent, are used in a multitude of applications. The present work, in particular, focuses on the reinforcement of carbon nanotubes (CNTs) within polymer composites in order to impart conducting properties in a non-conducting polymer. Usually, resistance in the flow of current occurs due to the re-agglomeration of CNTs and in order to overcome this problem, modification of CNTs was carried out through utilization of zinc (Zn) and copper (Cu-Zn) nanoparticles (NPs). Two types of filler: **I** and **II** having Zn/CNTs and Cu-Zn/CNTs were fabricated respectively. In polymethyl methacrylate (PMMA), different wt. % of the fabricated fillers **I** and **II** were dispersed, which corresponds the synthesis of monometallic and bimetallic composites. Methods and techniques like FTIR, XRD, SEM, and LCR meter were used for analyses of these composites. For monometallic and bimetallic composites, the observed conductivity value of filler was 3×10^{-3} S/cm and 1.67×10^{-4} S/cm at 0.1 and 0.05 wt. % respectively. It was concluded that in bimetallic composites, a high conductivity value was achieved at low filler concentration. These lightweight composites with significant properties make them useful in for manufacturing panels or casings in the aerospace and automotive industries.

INTRODUCTION

Engineering at atomic level is known as nanotechnology (1). In 1959, Richard Feynam for the first time introduced the forenamed concept (2). There exist a multitude of reported methods within the realm of nanotechnology (3) such as physical-chemical methods and green synthesis, used for synthesizing NPs (4). Nanoparticles have possible applications in numerous nanoscale devices owing to their small sizes (5). Composites are magnificent materials having a widespread application in advanced technology; there synthesis comprise dispersion of nanoparticles and carbon nanotubes as a filler in the polymer matrix (6). Incorporation of filler enhances the thermal, mechanical and electrical properties of a polymer (7). Nanocomposites, in particular, encompass unique characteristics and hence are applied as sensors, capacitor, and electromagnetic impedance (EMI) shielding; they are also spotted in diverse engineering industries (8).

Since the discovery of carbon nanotubes (CNTs) by Iijima in 1991 (9), the incorporation of CNTs in conducting polymers (CPs) such as Polypyrrol (PPy) and polyaniline (PANI) is chiefly focused by researchers. This incorporation heightens the conducting properties of composites thanks to their unique properties. The application of CPs can be witnessed in rechargeable batteries (10), chemical sensors (11), photovoltaic devices (12), corrosion devices (13), etc. Poly methyl methacrylate (PMMA), a non-conducting polymer (14) implies a special impact in this field because of its light weight (15) and amorphous nature (16), optical clarity, softness (17), biocompatibility (18) and transparent nature (19). Whereas, by incorporating with conducting filler this polymer could be converted into conducting one (17). CNTs, though possess many useful properties (20), yet very naturally come with a disadvantage (i.e. getting re-agglomerated) which can further decrease conductivity thereby giving rise to a resistance in the current flow (21). It has been reported that high surface area and nanometer size are particularly responsible for agglomeration in CNTs (22). Several techniques are reported to tackle this issue in order to obtain high electrical conductivities only through proper dispersion of CNTs in a polymer matrix (23). Different organic compounds like dodecyl benzene sulphonic acid (DBSA) and zwitterionic (ZIS) as surfactants, polyaniline (PANI) as conducting polymer, 3-amino propyl tri ethoxy silane (APTES) as cross-linker, were used to enhance maximum dispersion of CNTs and conductivity of PMMA in CNTs/PMMA composites (24-26). At another place (27), conductivity of PMMA was improved by dispersing series of cobalt (Co) nanoparticles in the absence or presence of DBSA.

Metal nanoparticles encompassing one metal are called monometallic nanoparticles (NPs) while those comprising two different metals are called bimetallic nanoparticles (28). Bimetallic NPs have relatively more ameliorated attributes such as optical, interfacial and high electrocatalytic activities when compared to monometallic NPs that are widely applied in sensing fields (29). Food and Drug

Administration (FAD) of the United States, approved bimetallic nanoparticles for unique food packaging because of their marvelous antibacterial activities (30). Fabrication of monometallic and bimetallic NPs is achieved through the reduction of metal ions (31). Over the past two decades, inorganic nanoparticles and polymer nanocomposites have remained an attraction for both academic and industrial researchers(32). The incorporation of nano-filler into a nanocomposite proved as a synergistic host by improving its conducting properties (33, 34).

In order to overcome the re-agglomeration of CNTs, researchers have tried their hand at a number of processing techniques (35) in an attempt for CNTs' homogenous dispersion so as to attain high electrical conductivity (36). Lanthanum/Cadmium/Polyaniline bimetallic nanocomposites (La/Cd/PANI BNC) were utilized as semiconductor devices and spintronic (37). Through a simple chemical method, multi-walled carbon nanotubes/Polyaniline (MWCNTs/PANI) composites were fabricated by silver-platinum (Ag-Pt) bimetallic NPs, which were proved as very useful electrode materials and could be applied as supercapacitors and biosensors (38). Chen et. al., prepared Cu/CNTs by using different methods like alloying method, co-deposition method, chemical vapor deposition and spark plasma sintering techniques and found that the best results were obtained by using Alloying method (39).

In the present work, fabrication of Zinc and bimetallic copper-zinc (Cu-Zn) NPs with CNTs and their incorporation within PMMA nanocomposites have been investigated in order to impart conducting properties into an insulating polymer. For the syntheses of Zn/CNTs/PMMA monometallic nanocomposites and Cu-Zn/CNTs/PMMA bimetallic nanocomposites, solution casting method has been used. The obtained sample **is** expected to have shown enhanced electrical conductivities as it could be used as capacitors, actuators and sensors.

EXPERIMENTAL

In this section, the fabrication of Zn/CNTs/PMMA and Cu-Zn/CNTs/PMMA bimetallic nanocomposites has been described.

Materials

PMMA and CNTs were purchased from *Sigma Aldrich* whereas; chloroform, sodium borohydride, zinc and copper were purchased from *Daejung.*

Fabrication of Metal Nanoparticles

Metal nanoparticles of Cu and Zn were fabricated through a reduction method (with slight modification) by using sodium borohydride ($NaBH_4$) as a reducing agent (40). One solution comprising of 0.2 M of metal chloride and another 0.4 M solution of $NaBH_4$ were dissolved in 50 mL of distilled water. The resulting mixture was stirred at 100 rpm while gradually adding $NaBH_4$ solution drop-wise. For complete formation of metal nanoparticles, an additional stirring for 30 minutes along with drop-wise addition of sodium borohydride was carried out. Finally, the prepared nanoparticles were washed with 900 mL of distilled water and thereby dried in the oven at 80 °C for 4 hours.

Fabrication of Cu-Zn Bimetallic Nanoparticles

Method as mentioned above was used with slight modifications for the preparation of bimetallic nanoparticles. For the fabrication of Cu-Zn bimetallic nanoparticles, equal amount of both, Cu NPs and Zn NPs (prepared by above method) were dispersed in 25 mL of distilled water. For complete formation of bimetallic NPs, the mixture was stirred for 1 hour at 1000 rpm so that maximum mixing could be provided and then kept in an ultra-sonication bath for 40 min. Later, the mixture of these particles was filtered and dried in an oven at 80°C.

Fabrication of Monometallic and Bimetallic Nanoparticles with CNTs (Filler)

For the fabrication of monometallic filler (Zn/CNTs, as filler I)), an equal amount of Zn-NPs and CNTs were added in 25 mL of distilled water. For maximum dispersion of these particles, this mixture was stirred for 1 hour and then kept on sonication for the next 40 minutes. Later, these particles were filtered and dried in an oven at 80°C. The same procedure was followed for the fabrication of bimetallic filler (Cu-Zn/CNTs, as filler II).

Fabrication of Zn/CNTs/PMMA and Cu-Zn/CNTs/PMMA Nanocomposite Films

For the fabrication of pristine PMMA film, commercially available PMMA was allowed to dissolve in 15 mL of chloroform. With the help of solution casting method, fabrication of thin films of PMMA was carried out. Incorporation of filler I and filler II into a polymer matrix was done respectively with different concentrations. Different amounts of fillers were dispersed separately in a 15 mL solution of PMMA and chloroform and kept on stirring for 1 hour and then sonicated for 40 min for homogenous dispersion of filler in the matrix. By using solution casting method same sized films were fabricated for different compositions by pouring the dispersed solution in same sized petri dishes (25, 27).

Characterization

To study polymer composite interaction, Fourier-transform Infrared (FTIR) spectroscopy was used while for the determination of crystalline and amorphous nature of polymer composite, x-ray diffraction (XRD) was done. For morphological and elemental analyses, scanning electron microscopy (SEM) and microscope were used. Furthermore, for electrical properties of composites (LCR) meter was used.

RESULTS AND DISCUSSION

FT-IR Spectroscopy

In Fig. 1, FTIR spectrum of pure PMMA, monometallic, and bimetallic composites have been shown respectively. From the spectrum of pure PMMA, the peaks at 750 cm^{-1} , 1387 cm^{-1} and 1063 cm^{-1} to 1270 cm^{-1} indicate the α methyl group and ester bond (C-O-C) vibrations respectively (41) . The peaks at 1434 cm^{-1} , 2953 cm^{-1} and 3000 cm^{-1} show the bending and stretching vibrations of C-H, $-CH_2$ and $-CH_3$ groups respectively (42). The characteristic peaks of PMMA between 1720 cm^{-1} and 1725cm^{-1} correspond the stretching vibration of carbonyl group (43). It was observed that the peak intensity of pure PMMA was decreased when filler was reinforced in the polymer matrix for composite fabrication, which corresponds the development of new physical linkage (44).

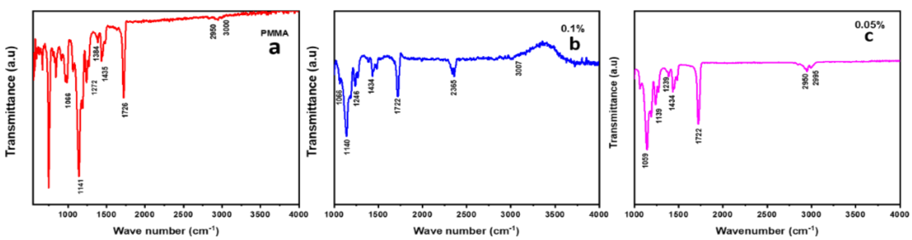

Fig. 1. FT-IR spectrum of pure PMMA (a), monometallic composites (b), bimetallic composites (c) are shown respectively

In both composites the main peak of PMMA got shifted from 1726 cm^{-1} to 1722 cm^{-1}. Correspondingly, peaks from 2950 to 3000 cm^{-1} were shifted to 2365 to 3007 cm^{-1} and 2324 to 2995 cm^{-1} in monometallic and bimetallic nanocomposites (45).

X-Ray Diffraction Analysis

For determination of crystalline structure of fabricated nanocomposites, XRD analysis was done. In Fig. 2, the XRD pattern of pure PMMA, pure CNTs, Zn nanoparticles, Cu nanoparticles, and monometallic and bimetallic composites are shown respectively. For pure CNTs, $2\Theta = 25.6^{o}$ (45). For Zn nanoparticles, the diffraction values of 2Θ were observed at 28^{o}, 36^{o}, 47^{o}, 56^{o} and 70^{o} (43, 45). No sharp peak for PMMA was observed because of its amorphous nature.

Sharpness in the peaks of bimetallic composites as compared to monometallic nanocomposite films can be observed clearly. Variation in the values of 2ϴ from monometallic to bimetallic nanocomposites were appeared, which were converted into 30^o-32^o, 40^o-43^o and, 47^o-49^o respectively.

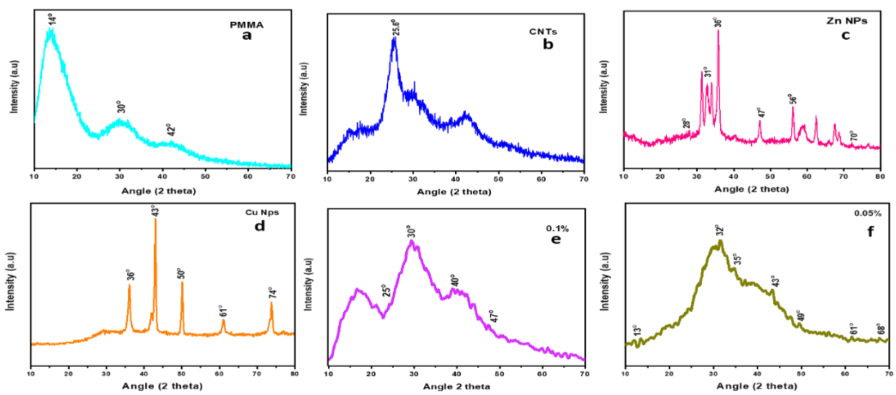

Fig 2. XRD pattern of pure PMMA (a), pure CNTs (b), Zn NPs (c), Cu NPs (d), monometallic and bimetallic composites (e and f) are shown respectively

Crystalline character was established when PMMA was incorporated with monometallic and bimetallic fillers. Table 1 shows the 2ϴ and d-spacing values of Cu NPs, Zn NPs, pure CNTs, PMMA, filler, and monometallic and bimetallic composites.

Table 1. d-spacing values of different materials

Materials	**2ϴ** (d-spacing)
Cu NPs	**36^0** (9.57), **43^0** (17.3), **50^0** (14.9), **61^0** (3.83)
Zn NPs	**27^0** (3.03), **31^0** (5.64), **35^0** (8.17), **47^0** (4.07)
CNTs	**25.6^0** (1.55)
PMMA	**14^0** (1.07), **30^0** (0.76), **42^0** (1.12)
Zn/CNTs	**25.6^0** (1.37), **35.8^0** (11.4), **47^0** (5.27), **56^0** (11.42)
Cu-Zn/CNTs	**26^0** (1.89), **36^0** (8.41), **47^0** (3.81), **56^0** (5.55)
Zn films	**29.5^0** (1.39), **31^0** (1.45), **40^0** (1.84), **47^0** (1.47)
Cu-Zn films	**32^0** (1.09), **43^0** (1.68), **50^0** (1.96)

Scanning Electron Microscopy

In Fig. 3 microscopic images for both monometallic (a) and bimetallic (b) composites and SEM images (c) and (d) are shown respectively.

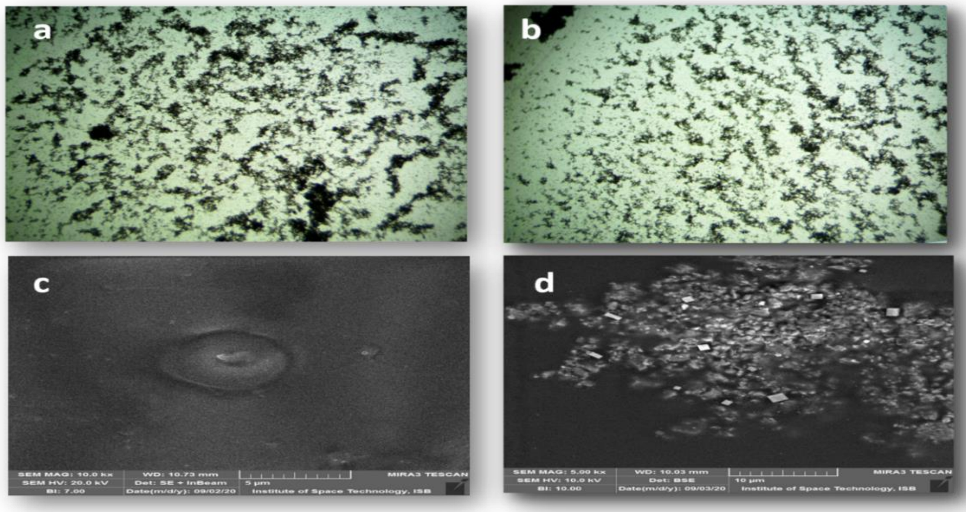

Fig 3. Microscopic images of (a) Monometallic (0.1 wt. %) and (b) bimetallic (0.05 wt. %) composites, and SEM images of (c) Monometallic (0.1 wt. %) and (b) bimetallic (0.05 wt. %) composites

Overall, randomly distributed filler with few agglomerated parts has been observed in SEM images. However, this distribution is more dominant in bimetallic composite which reflected that the modification of CNTs with Cu and Zn significantly helped in reducing the re-agglomerating character of CNTs.

Electrical Conductivities

It has been observed that Cu is considered compatible with the next generation because of its high electrical conductivity and low electrical migration but it is less stable because of its facile oxidation. Similarly, CNTs also possess significant electrical, mechanical, and thermal characteristics but their properties are not exposed well as they re-agglomerate soon after dispersion. Chen et. al., synthesized CNTs/Cu and Cu/CNTs-1 composites in order to form a strong interfacial bonding between them by using different methods and found that the alloying method (AM) was the more feasible one with a high level of electrical conductivity of 92.9% for Cu/CNTs composites (39). On the other hand, researchers also tried to enhance the mechanical and thermal properties of PMMA by introducing noble metals like platinum (Pt), palladium (Pd), Cu and gold (Au) nanoparticles via in situ polymerization. They found Pd to be the best one among these noble metals as it increased the tensile strength of PMMA composite by 18.8% (46). In 2009 a group of researchers, introduced three different sized Cu powder particles in PMMA composites and observed an increase of 12 orders of magnitude in the electrical conductivity of the composites (47). Tajamal and his group studied the impact of cobalt (Co) nanoparticles, with and out the use of surfactant (DBSA), on the percolation threshold of PMMA composites. They found that addition of DBSA helped in the formation of conducting network of Co nanoparticles by reducing the chances of its re-agglomeration in PMMA matrix (27). Similarly, to introduce the conductivity in PMMA, in 2017, another group of researchers fabricated two types of PMMA composites, by using PANI-CNTs and DBSA-CNTs as organic modified fillers (26) . They observed a remarkable increase in conductivity values i.e., 5.6 S/cm and 2 S/cm at 2 wt.% values for both composites respectively. As literature shows both organic and inorganic fillers along with different surfactants have been used to enhance the properties of both CNTs and PMMA, therefore, in present work, the monometallic (Zn) and bimetallic (Zn-Cu), as inorganic modifiers, were doped with CNTs in order to enhance the properties of CNTs and PMMA in CNTs/PMMA composites. It is observed that conductivity was increased by increasing the wt.% of filler. For monometallic composites, at 0.05 wt.%, the observed conductivity value was 8.47×10^{-6} S/cm and it reached to 3×10^{-2} S/cm at 0.1 wt. %. However, it was found to be 1.67×10^{-4} S/cm at 0.05 wt. % for Cu-Zn/CNTs/PMMA. The electrical percolation phenomena of monometallic and bimetallic composites were studied with the help of percolation model which explains the relation between conductivity of composites and the content of filler particles and is described well by the following formula as equation (i):

$$\sigma \, \alpha \, (f - f_c)^t \qquad for \;\; f \geq f_c \qquad (i)$$

Here, σ is the conductivity, f is filler's content and f_c is the critical content at the percolation and t is the exponent of conductivity (26). The graph was plotted between log $(f - f_c)$ and log σ. Values of f_c and t for monometallic composites were obtained as 0.07 wt. % and 1.07 respectively. For bimetallic composites, the obtained values of f_c and t were 0.03 wt. % and 0.918 respectively. Re-agglomeration intensity of CNTs was reduced in both types of composites which supported the establishment of conductive network earlier at low percolation threshold. Electrical conductivities and f_c values of monometallic and bimetallic composites are shown in Figs. 4 and 5 respectively.

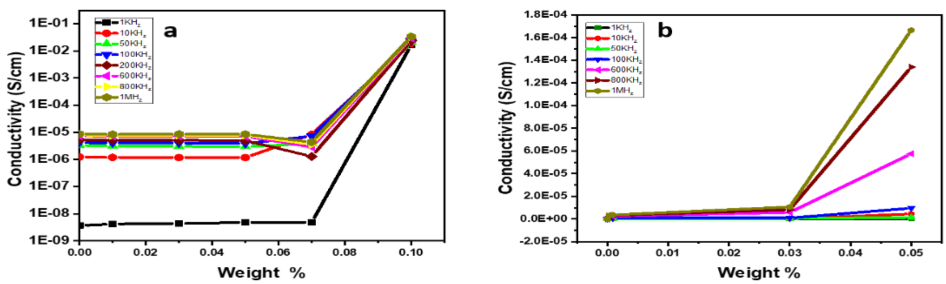

Fig. 4. Conductivities of monometallic (a) and bimetallic (b) composites at different wt. % are shown respectively

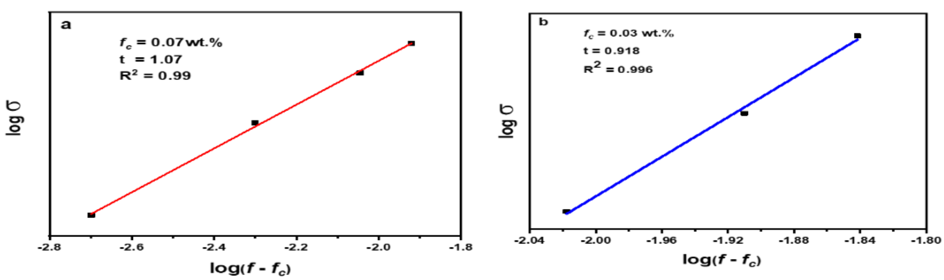

Fig. 5. f_c values for monometallic (a) and bimetallic (b) composites are shown respectively

Researchers are working to enhance PMMA properties because of its cost effective, transparent, ease of processing, biocompatibility and lightweight properties. Table 2, shows the comprehensive understanding of different composites of PMMA and their electrical properties.

Table 2. Comparison of percolation threshold of fillers in different PMMA composites

Composite	Electrical Conductivity	Percolation Threshold	Reference
DBSA-Co Nps/PMMA Co Nps/PMMA	- -	0.5 wt.% 1 wt.%	(27)
PANI-CNTs/PMMA DBSA-CNTs/PMMA	5.6 S/cm 2 S/cm	1.3 wt.% 0.8 wt.%	(26)
PVDF/PS/CNTs-PMMA	8.06×10^{-3} S/cm	0.07 vol.%	(48)
Zn-CNTs/PMMA Cu-Zn/CNTs/PMMA	3×10^{-2} S/cm 1.67×10^{-4} S/cm	0.07 wt.% 0.03 wt.%	Present work

CONCLUSION

In a nutshell, within the present work, the fabrication of Cu and Zn nanoparticles was done by using reduction method. By using these nanoparticles, modification of CNTs was done in order to reduce their re-agglomeration of CNTs in polymer composite. Further, the fabrication of monometallic (Zn-CNTs/PMMA) and bimetallic (Cu-CNTs/PMMA) nanocomposites was done by using Zn-CNTs and Cu-Zn/CNTs as a filler. FTIR, XRD, SEM, and microscopic and LCR meter were used for the characterization of these composite films. In FTIR spectrum, development of physical linkage between polymer matrix and filler attributes the appearance of new peaks as compared to the parent peak of PMMA. Similarly, in XRD pattern, presence of sharp peaks shows that in amorphous PMMA, a crystalline nature got developed. In SEM and microscopic images, proper dispersion of filler in the polymer matrix is shown, which results the improved conductivity of polymer. LCR results showed that an enhanced conducting was observed between CNTs and composites. The maximum observed conductivity values were 3×10^{-3} S/cm at 0.1 wt. % and 1.67×10^{-4} S/cm at 0.05 wt.% of filler for monometallic and bimetallic composites respectively.

Researchers have expressed their demands towards flexible electronic devices. In the present research work low cost and high efficiency films were fabricated which can be applied for developing flexible, rollable, bendable electronic devices and as transistors. PMMA is a biocompatible polymer and its low percolation threshold with controlled conductivity could make it more useful in tissue engineering, diagnostic labs and in drug delivery systems.

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