Experimental Design of AL2O3/MWCNT/HDPE Hybrid Nanocomposites for Hip Joint Replacement

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Abstract

A hip fracture can be considered a major health issue facing the elderly fraction of humans. The loads exploited by the lower limbs are very acute and severe; in the femur, they can be several folds higher than the whole weight of the body. Mechanical properties like strength and hardness are challenging parameters which control the selection of the joint. In this research work, a modification has been performed by adding multi-layer carbon nanotubes to the High-density polyethylene thermoplastic polymer together with alumina. Prepared material well physiochemically characterized to check the morphology and the structure. Thermal gravimetric analysis (TGA) dedicates that the percentage of crystallization has been increased to 6% after adding Multi-wall carbon nanotubes (MWCNT)to the polymer. According to mechanical properties results, MWCNTs successfully increase in young's module and hardness of the polymer. Optimization of the materials ration has been done and it has been found that hybrid composite with structure (2.4% AL_2O_3 and 0.6% MWCNT) obtains high mechanical properties compared to other ratios with 3% MWCNTs and 5% MWCNTs. Also, moisture absorption decreased to 90% at with 5% MWCNT ratio. Experimental results of MTT assay of a normal human epithelial cell line (1- BJ1) over AL_2O_3/MWCNT@ HDPE showed a low value of cytotoxic activity, which proves that it is proper for medical use.AL_2O_3/MWCNT@HDPE composite showed promising results for further studies of artificial joints.

1. Introduction

Hip joint represents a major health challenge facing medical society nowadays. The hip prosthesis is the greatest key factor of biomaterial fabrication to meet the requirements of joint arthroplasty. Massive loads experienced by the limbs are very dangerous. Abnormal combinations of mechanical and physical characteristics are the main parameters that control the quality and feasibility of the hip joint. High strength, toughness, tailored stiffness, resistance to impact and corrosion are examples of those combinations relevant for tissue substitute structure. Many polymers are broadly utilized particularly to be applied in this direction due to wide classification of chemical compositions, characteristics, feasibility and biocompatibility[1-3].

While joint replacement represents attractive cases in the specialization of orthopedic surgery but maintaining the design and structure for a long time is still a big challenge. The mean half life time of the hip joint is usually among the range of 15-20 years. Since the great discovery of polymer composites by the Toyota research group[4], a new track of research has been assembled in the field of joint replacement. Previously, researchers used to apply inorganic nanomaterials as loading compounds in the preparation of composites with very high processing and manufacturing costs [5]. Also preparing composites based inorganic compounds will produce heavy material which not applicable for medical use. Nanotechnology also displays a new opportunity to improve the performance of the composites together with the carbon compounds because of the unique characteristics of the nanoparticles. Nanocarbon materials like fullerene, carbon nanofibers and carbon have earned great attention in polymer science.

This research focuses mainly on studying the effect of impeding MWCNT inside the pores and over the surface of HDPE polymer. Carbon nanotubes or single layers of graphene rolled into a cylinder shape, have been commonly applied and evolved into diverse research directions: mechanics, and even medical applications. CNT usually exists in 2 phases, single-walled and multiwalled, with different characteristics properties [6-11]. There are different types of techniques that have been improved for synthesizing CNT which mainly involve vapor phase routes. Commonly, three methods are being applied for producing CNTs the first one is the chemical vapor deposition, the second one is the laser-ablation and carbon arc-discharge system[12]. A combination of carbon nanostructures and polymer matrices widely studied to adjust the mechanical properties and to extend their applications[13-14]. HDPE exhibits good mechanical behavior includes high strength density ratio, lightweight and chemical resistance, however it shows low hardness with long run use [15-19]. Improving those properties has been retained in different research work recently by adding various types of fillers such are talc, nano-Hydroxyapatite and nano aluminum oxide [20-21]. The results exploit a smooth increase in that the degree of crystallinity and modulus of the composites, followed by a decrease at higher ratios[22-23]. Also, the friction coefficient of composites reduced and the hardness increased with the increment of carbon fibers [24-25]. HDPE/HA composites have been assembled and commercialized under the name of HAPEX™, showing very promising mechanical properties like human being bone [26-27]. It has been retained that by slightly replacing the HA content with alumina tribological characteristics of HAPEX™ are significantly improved [28-29]. Recently, carbon Nanotubes and graphite have been excessively used to develop composites for some applications [30-41].

In this research work, different ratios of MWCNT incorporated inside the matrix of HDPE in attempts to improve the hardness and other mechanical properties of polymer for a hip joint application. AL2O3/MWCNT@ HDPE composite has been prepared by the wet chemical method, completely characterized to check the structure and the morphology. Using the advantage of MWCNT solid lubricant efficiency for improving the overall mechanical properties of HDPE as a bearing material. Also, the influence of adding MWCNT over the normal cell (Cytotoxic activity test) has been studied to confirm the feasibility for medical use.

2. Materials And Methods

High-density polyethylene (the tensile strength = 27.5 MPa) and (density = 0.944 g/cm3) was purchased from Nanotech company. MWCNTs and nano alumina (Al_2O_3) used in this research were purchased Middle east company for the petrochemical industry. The properties on MECNT have 8–13 nm diameter, 3–15 μm length and purity 99.87%. The physical properties of nano-Al_2O_3 are density at RT: 3.9 g/cm3 and particle size 20–30 nm and purity of 99.95%.

The first composition of MWCNT@ HDPE and the second composition of AL_2O_3/MWCNT@ HDPE composite have been prepared by wet chemical approach as presented in Fig. 1. An ethanolic mixture of MWCNT and high-density polyethylene has been stirred for 30 minutes [42–43]. Alumina has been added to the reaction in the presence of TWEEN as a pore directing and capping agent. Different ratios of MWCNT introduced in attempts to obtain the highest performance [44].

Then the composite has been stirred for 1 hour. After that, the powder separated and dried well to remove excess ethanol [45–46] and the surfactant. 185°C temperature has been applied for about 120 min. Following the extraction of AL_2O_3/MWCNT@ HDPE composite from the inward blender, hot pressing has been done to design the specimens into the desired shape. Then, the specimens have been bitten by rapid cooling to room temperature.

2.1. Material characterizations

X-ray diffraction technique has been utilized to check the structure of phase material and the crystallinity of prepared material [47]. The XRD measurement has been performed using (Siemens XRD D5000) and a copper X-ray source within the range from 0° to 70°. The voltage and current adjusted to be 40 kV and 50 mA, respectively. The morphology and microstructure characterization of AL_2O_3/MWCNT@HDPE hybrid composites have been studied using SEM and TEM systems [48–50].

Mechanical testing is applied to exploit the characteristics like hardness, modulus, fracture toughness or yield strength. Uniaxial compression and tensile testing are typically measured to investigate bulk powders to acquire elastic modulus [51]. The hardness test uses an indenter probe that is located over a surface under a specific weight load. Usually, the size of the indentation is calculated to determine hardness. Microhardness testing is considered an industry standard for quality control for hardness data [52]. Tensile measurements have been measured on a universal testing machine with a speed of 5 mm/min. Percentages of the MWCNT have been adjusted to be 1–5%. All samples were tested in the same environmental conditions. The Vickers microhardness tests were dedicated to investigating the influence of MWCNTs on the mechanical properties as well. The total load is normally applied between 10 and 15 seconds. The zone of the slanting surfaces of the indentation has been investigated and the Vickers hardness which is calculated by the (kg) weight load by the square (mm) region of indentation [53].

TGA test estimates the weight change of a material, either as an element of expanding temperature, or isothermally as a function of time under nitrogen or helium. The degeneration conduct of polymers and their hybrid composites is usually determined by 3 parameters: the first one is the temperature at which the framework starts to debase, the degradation temperature T_d, "temperature at which the most noticeable debasement rate happens and degradation rate Dr. TGA, which is found in the subsidiary weight reduction as an element of temperature curve[54–55]. TGA analysis was performed using (DTG – 60 SIMULTANEOUSDTG- TG SHIIMAADZU, JAPAN) for each sample and AL_2O_3/MWCNT@HDPE composite. The specimens have been heated from 25 °C to 750 °C at a rate of 10 °C/min in a nitrogen atmosphere. Flexural and mechanical characteristics usually decrease the water absorption ratio increases. Tensile modulus was found to decrease at a higher level of water absorption which considered a sensitive property of the fiber. Flexural modulus decreases in higher fiber content specimens after water absorption. Therefore, it is a requirement to investigate the effect of adding MWCNT over moisture absorption of HDPE.

Water absorption examination (ASTM D750-95) has been carried out by a total inundation of 5 samples in distilled water at room temperature for 72 h. The water absorption was calculated by weighing the samples versus time. Since this application for medical use, it is very important as well to check the feasibility of material to be applied inside a human body. Cell viability test has been dedicated by the mitochondrial-dependent reduction of yellow MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide) or MTT essay to magenta formazan [56].

All measurements have been done in an antiseptic zone applying a laminar flux cabinet biosafety class II level (Baker, SG403INT, Sanford, ME, USA). Cells have been suspended in DMEM-F12 medium with 1% antibiotic-antimycotic mixture (10,000 U/ml Potassium Penicillin, 10000 µg/ml Streptomycin Sulfate and 25 µg/ml Amphotericin B) and 1% L-glutamine at 37 ºC under 5% CO_2. Cells have been refined for 5 days, then implanted at a concentration of $10*10^3$ cells/well in unprecedented plenary outgrowth medium in 96-well microtiter plastic plates. After 48 h of incubation, the medium has been aspirated, 40 ul MTT salt (2.5 µg/ml) was added to each well and hatched for further four hours at the same conditions[57−58]. Absorbance has been estimated using a microplate multi-well reader (Bio-Rad Laboratories Inc., model 3350, Hercules, California, USA) at 595 nm and a reference wavelength of 620 nm.

3. Results And Discussion

Figure.2 dedicates the XRD diffraction pattern of HDPE, MWCNT and HDPE/MWCNT hybrid composite. HDPE presented with two characteristic peaks at 21.4° and 23.2°. These peaks were also detected in hybrid composite, but the intensity was greater, hence, more crystalline. This could happen as a result of the interaction and crystallization behavior of MWCNT with HDPE during the blending process. The X-ray diffraction (XRD) pattern of MWCNTs is shown as well in Fig.2. MWCNT exhibits 2 peaks at around $2\theta = 26^\circ$ and other broad peak centered at $2\theta = 43^\circ$ corresponding to the (002) and (100) Bragg reflection planes having the interlayer spacing of 3.213 and 2.012A respectively [59].

Microscopy images have been collected with its two types the scanning and transmitting to further investigate the nanocomposite structure and the matrix distribution as shown in Fig. 3. Scanning electron images of the nanocomposite samples shows clearly the MWCNT among the polymer matrix. In the processing of HDPE and MWCNT blends by the melt blinder, the polymer chains of HDPE are overlapped with each other as shown in SEM figures. Also, HETEM confirms the MWCNT structure very well and nano-scaled design of the other material which composes the material. The homogenous distribution of the MWCNT over the whole matrix can be also observed.

3.2 Mechanical properties

Figure 4. (a) exploits the stress-strain curve for AL_2O_3/MWCNT@ HDPE Nanocomposite. It can be seen clearly that the composite specimens tend to reach the highest point around 0.3. It also shows the strain of samples with a higher concentration of MWCNT more than others with lower concentrations. Table.1 illustrates the overall view of different parameters that control the mechanical direction, i.e. elastic modulus, tensile strength, and ductile. The maximum value of young's modulus reaches up to 6.25% when MWCNT with 5% applied compared with 7 % compared with pure HDPE. The percentage of ductility reaches its max value at 5 wt.%CNT. Furthermore, toughness has been calculated from the area under the load versus stroke curve, the toughness of the pure polymer was about 160 MJm^{-3} which is less than in the case of (1%) MWCNT impeded into the polymer. The 1 wt.% CNT loaded sample reached the max value of about 610 MJm^{-3}. the toughness decreased sharply with the increase of CNTs in the HDPE matrix as a result of the CNT agglomeration is low with increasing condensation of filler content.

Good dispersion between polymer and CNT particles could be another reason for the mechanical performance improvement as illustrated in Table. 1. The significant increase of tensile strength has been observed mainly between MWCNTs ratio 3 wt. (%) - 5 wt. (%). According to recent research work, MWCNTs reinforcement mostly provides also strength refinement, furthermore, it can also transfer their strength to composite materials by taking over the load applied to the matrix material. It has been confirmed that MWCNTs prohibit the propagation of cracks in the matrix during erosion situations through their foaming motion. This case is also confirmed by the literature. However, the minimum strengthening amount can vary due to acquire strength improvement according to the reinforcement material. But the MWCNTs used in the existing study generally give better results at 5% wt. [60-61].

Table 1. Mechanical Properties of Nano Composite.

	Elastic modulus E (Gpa)	Ultimate Tensile strength (Mpa)	Max force (N)	Ductile %EL	Toughness×10^6 J/m^{-3}	Hardness (Mpa)
(HDPE-0wt.%)	0.96±0.04	27.2	126.3±7.6	7.40	160	56.2±1.1
(HDPE-1wt.%)	0.72±0.03	24.2	112.7±3.5	11.48	610	62.08±1.8
(HDPE-2wt.%)	0.75±0.02	25.5	118±0	8.70	400	64.5±1.5
(HDPE-3wt.%)	0.74±0.01	26.1	121.7±2.5	9.54	210	66.2±1.07
(HDPE-5wt.%)	1.02±0.02	29.0	134.3±1.5	11.29	250	77.4±1.5

As shown in Fig. 4 (b) and (c) that the value of Vickers hardness increases with increasing the addition of MWCNTs. MWCNTs hardness increased up to 37% compared to pure HDPE. Furthermore, it shows also the increase of the viscoelastic moduli and Young’s modulus. This could be attributed to the narrowing of the interspaces between molecules and reduced mobility. One of the best ways to reinforce mechanisms in polymer matrices is to transfer shear stresses in the nanoparticle-matrix interface and to nucleate a 3D nanostructured network. It reduces the mobility of the polymer bonds, thus leading to changes in a glass transition and elastic modulus of the nanocomposite, and yielding focus at very low strains[62-63].

Table 2. Mechanical Properties of Hybrid Al_2O_3/HDPE/MWCNT Nano Composite.

	Elastic modulus E (GPa)	Ultimate Tensile strength (MPa)	Max force (N)	Ductile %EL	Toughness MJ/m^{-3}	Hardness ($HV_{0.03}$)
(0wt%CNT, 3wt%AL_2O_3)	0.97±0.013	24.6	125.7±1.53	23.2	569	64.58±1.2
(0.6wt%CNT, 2.4wt%AL_2O_3)	1.00±0.073	28.01	130.7±9.61	25.5	705	64.58±1.2
(1.2wt%CNT, 1.8wt%AL_2O_3)	0.97±0.008	27.3	127±1.00	19.79	399	70.20±1.3
(1.8wt%CNT, 1.2wt%AL_2O_3)	0.95±0.053	26.5	123.3±6.81	27.7	604	56.62±1.5
(2.4wt%CNT, 0.6wt%AL_2O_3)	0.97±0.002	27.1	126.8±0.29	17.9	369	62.25±1.6

According to the results also it has been found that addition of alumina linearly increases the hardness and elastic modulus as shown in Table 2. Hardness and Elastic modulus have been figured out against MWCNTs and alumina content. The maximum level of hardness can be seen at (HCNTAL3) was 70.20±1.3 MPa with an increase in hardness by 6% comparing to 3wt%MWCNT and Elastic modulus values and 1±0.073 GPa at (HCNTAL2). Toughness and ultimate tensile for composites containing (HCNTAL2) increased by 75% and 7.6% compared to (3wt%CNT). Moreover, ductility also increased by adding aluminum oxide into the polymer matrix as illustrated in Table. 2. For composites containing (HCNTAL4) and (HCNTAL2) the ductility has been increased with adding aluminum oxide to the matrix showing an improvement ~145%, 200 % in comparison with 3wt% MWCNTs and 5wt%MWCNT polymer matrix respectively. It has been found that adding aluminum oxide to MWCNTs content caused marked improvements in mechanical properties of the resultant 3%wt MWCNTs / HDPE composites and equal to 5wt%MWCNTs/ HDPE. The possible idea behind that is the homogenous dispersion of nanotubes into the polymer matrix as confirmed using TEM images.

3.3 TGA analysis of HDPE/MWCNT composites.

TGA curves shows that onset temperature dwindling with the addition of chemically treated MWCNT due to amorphous carbon present in the CNTs, Mass loss degradation temperature of the composite increases as the MWCNTs ratio increases according to the TGA data as shown in Fig. 5 (a).

The lowest mass loss rate value is obtained for 3% and 5% MWCNTs. This exploits that mass loss melting speed rate decreases with increasing MWCNTs weight ratio and melting point temperature increases as shown in Fig.5 (b).

3.4. Moisture absorption

One of the most important parameters which considered a key factor that has been identified for polymer nanocomposite was water absorption because it usually affects their operating time[64-65]. The idea that cellulosic fibers easily absorb water is one of the reasons for fiber surface treatments. The particles treated by graphite fibers and its derivatives may absorb less amount of moisture, and thus support adhesion to the polymer matrix, which results in better execution in a humid environment. The high-water absorption of the polymer nanocomposite may cause difficulties during processing. This can be due to partial curing of the thermosetting matrices, the presence of gaps or cracks, or even poor matrix–fiber adhesion [66]. It was observed that HDPE/MWCNT nanocomposites express less water absorption compared to that of pure HDPE alone as shown in Fig. 6. The inclusion of MWCNTs facilitated the crystallization of HDPE and therefore led to higher crystallinity in HDPE Nanocomposites. This made it more difficult for water to diffuse into the HDPE matrix.

3.5 Cytotoxic activity of AL_2O_3/HDPE/MWCNT composites

The in vitro cytotoxicity tests of AL_2O_3/HDPE/MWCNT composites over human normal epithelial cell line 1- BJ1 (normal Skin fibroblast).The sample prepared by using the composite with concentrations between (100 to 0.78 μg/ml) and 100-75 PPM MTT with a different activity. It can be considered that MTT assay probing system for the study of the toxicity particularly because of normal cells very sensitive against this material. According to the results, the majority of cells remains safe and viable under all concentration which is in strong agreement with the literature as shown in Fig.7.

Conclusions

In summery HDPE Polymeric nanocomposite has been reinforced with MWCNTs and alumina by using a wet chemical approach. Structure and morphology of the material have been characterized using SEM, TEM, XRD, and TGA. It has also has been reported that there is a weak interface between 1, 2%wt CNT particles with the polymeric HDPE material, however good physical bonding of MWCNTs has been reached when the ratio becomes 5%. Improvement in the mechanical properties of the material after incorporation of the CNT particularly with ration 5%. TGA characterization of the prepared composite exploits that mass loss melting speed rate decreases with increasing MWCNTs weight ratio and melting point temperature increases. Also, water absorption has been studied, all samples which sometimes limit the surface treatments and it shows good performance with the lowest absorptance for 5% compared to other ratios. As this work is going to be applied for medical applications, it is important to determine the effect of the composite over samples. Cytotoxicity or cell cycle study of the normal cells has been done for this purpose and it shows low toxicity against human normal cells. According to these results, we can conclude that HDPE/MWCNTs/AL_2O_3 composite could be a good and promising candidate material for the total joint replacement.

Declarations

 Availability of data and materials 'Not applicable'

 Competing interests

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

 Funding

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

 Authors' contributions 'Not applicable'

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Figures

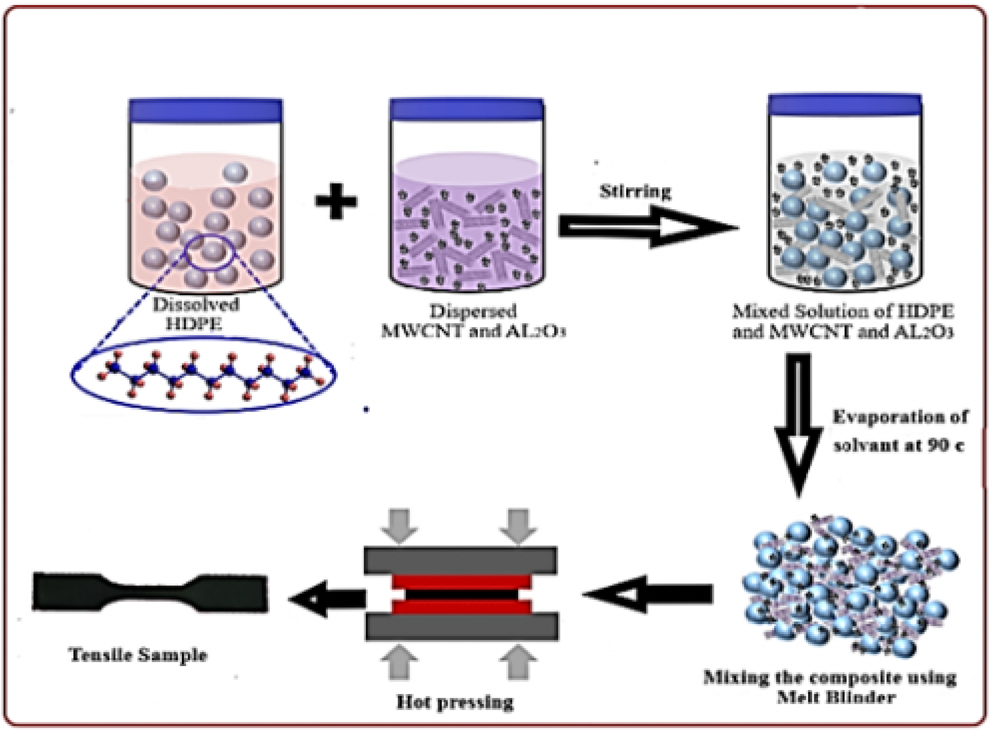

Figure 1

Schematic diagram of HDPE/MWCNT/ AL2O3 manufacturing process

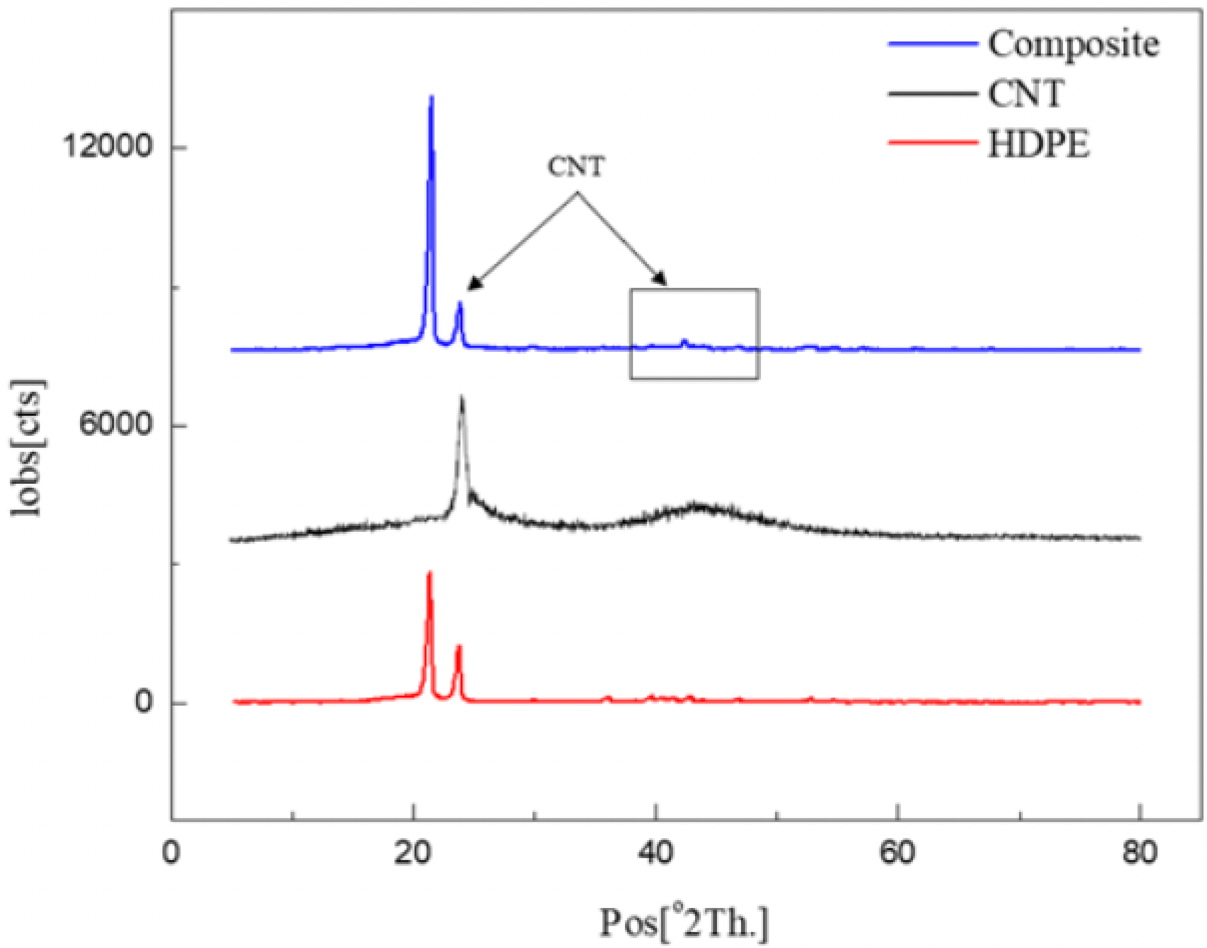

Figure 2

XRD patterns of HDPE, MWCNT, and HDPE/MWCNT hybrid composite

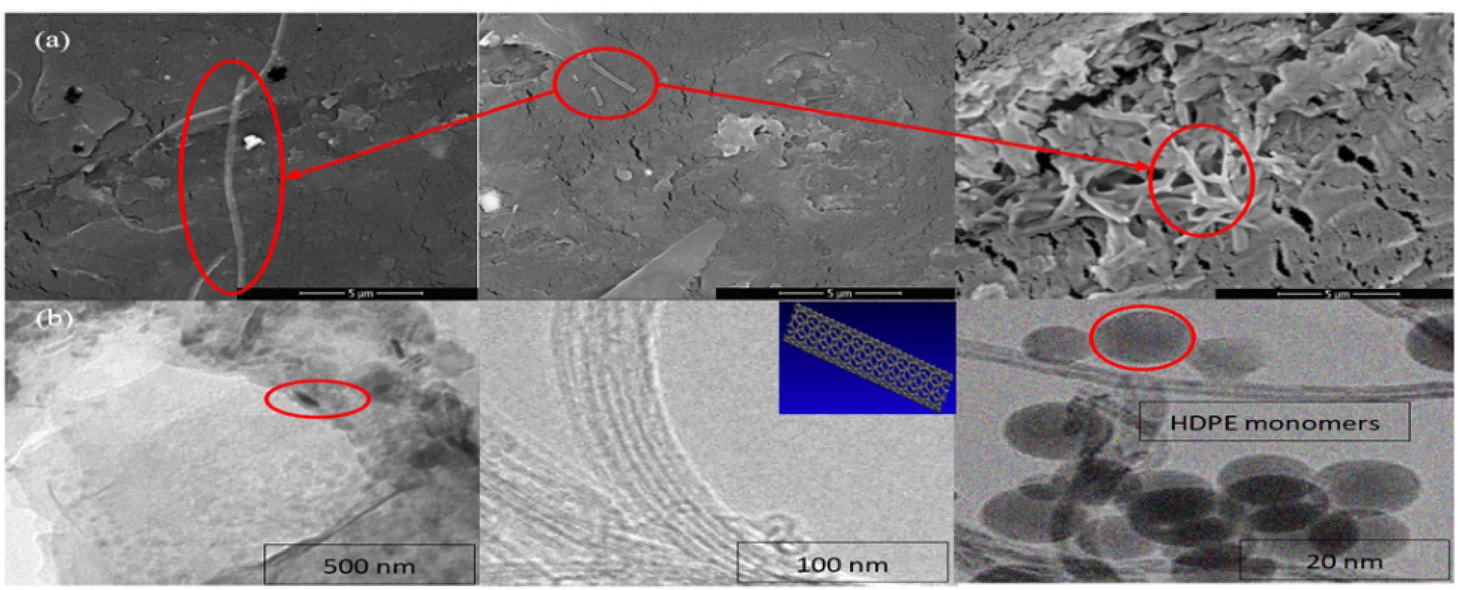

Figure 3

(a) SEM images of HDPE/MWCNT hybrid composite with 5% MWCNT ratio nanocomposite and (b) HETEM of the same composite.

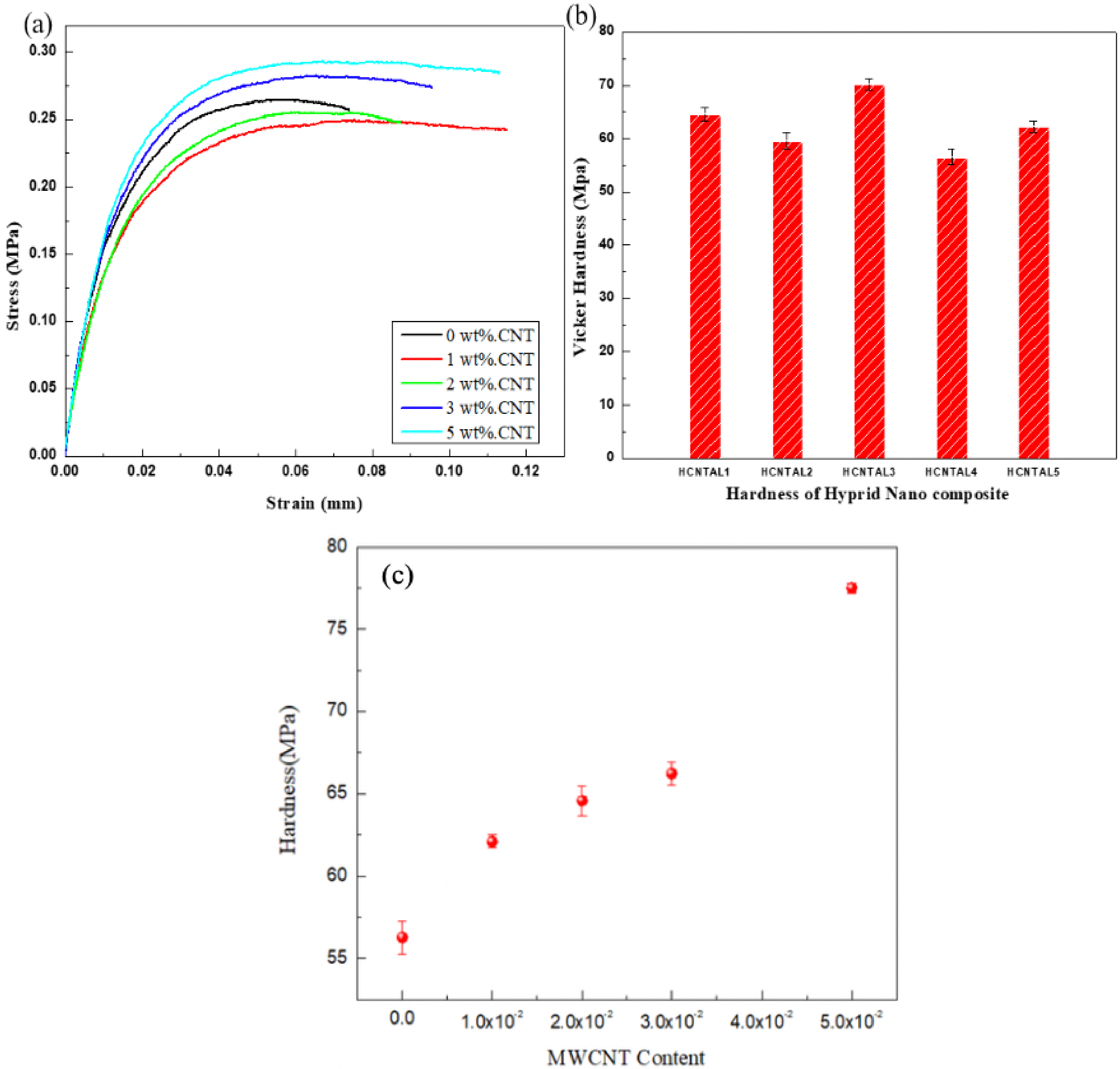

Figure 4

(a) Stress-strain curve for HDPE/MWCNT Nanocomposite, (b) Hardness of hybrid Nanocomposite and (C) Linear equation of Micro Vickers Hardness.

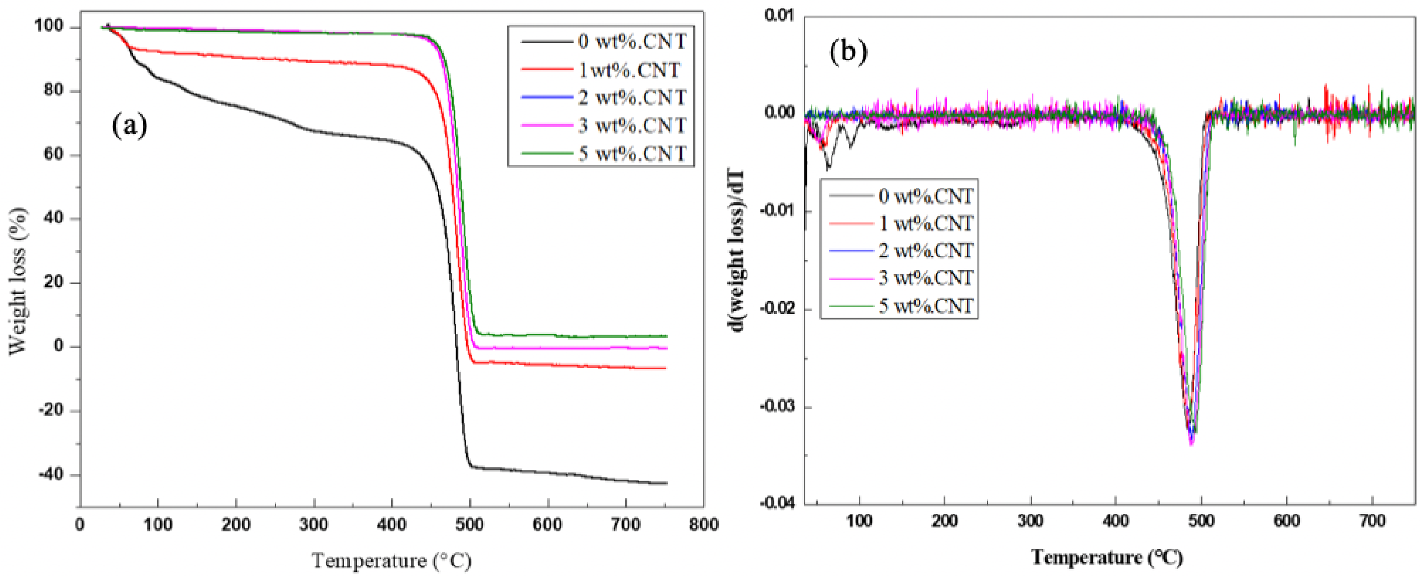

Figure 5

(a) TGA curves of HDPE/MWCNT composites and (b) DrTGA Curve Of CNT-HDPE Nanocomposite.

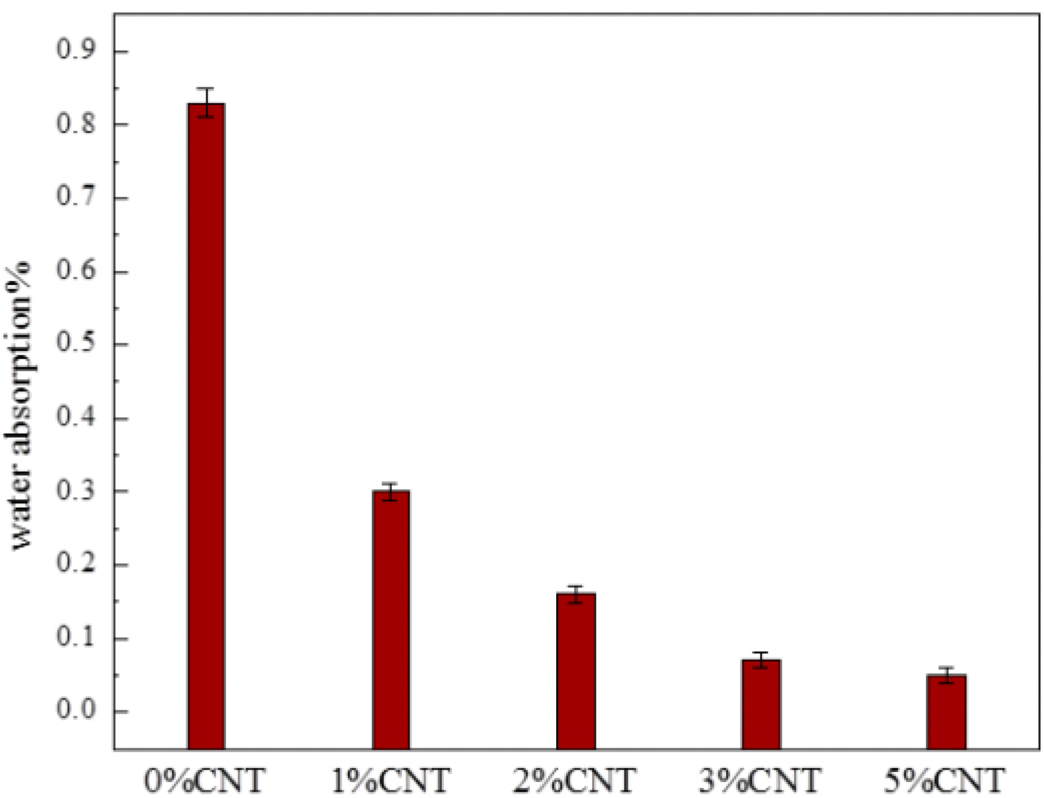

Figure 6

Results of water absorption

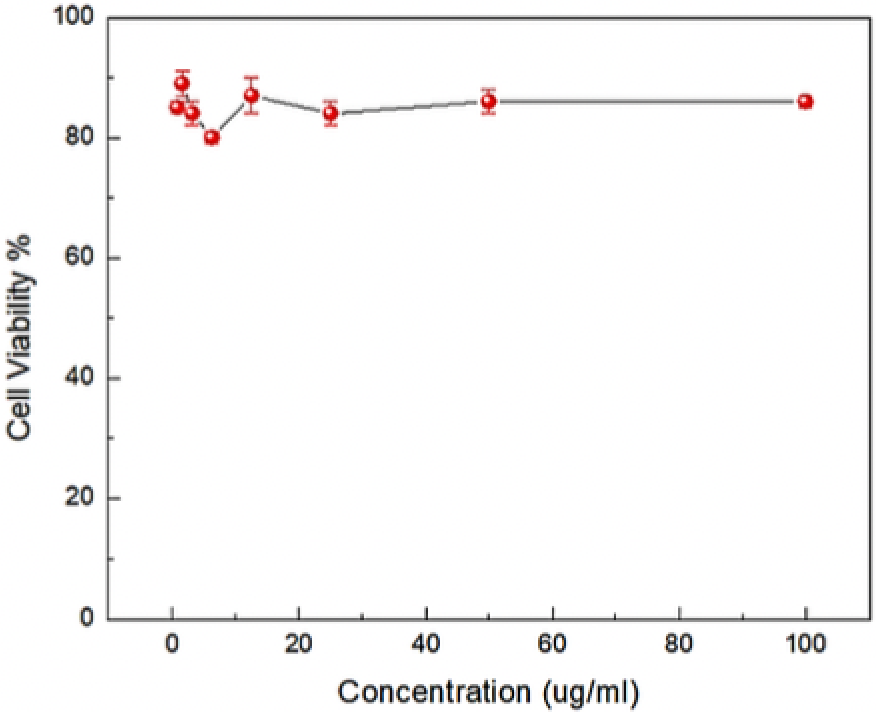

Figure 7

Cell viability of AL2O3/HDPE/MWCNT over human cell lines.