Effect of Surface modified f-CNTs on the Electrical and Mechanical Properties in Polymer blend (PC/PMMA/PS) to form Polymer Nano composites (f-PCNCS)

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Abstract—Nano composites PCNs and f-PCNs were prepared by a mixture of PC/PMMA/PS polymer blend, MWCNTs and functionalized f-MWCNTs by simple oxidation reaction carried in a mixture of acids HNO₃, HCl, H₂SO₄.The functionalized f-MWCNTs were incorporated into polymer blend matrix at varying ratio of 0.1% and 0.3% at 260°C and 270°C to prepare polymer Nano composites (PNCS).The new prepared Nano composites, pure polymer and polymer blend (PC/PMMA/PS) were characterized by FTIR to check the nature of the sample. It was found that remarkable improvement in mechanical and electrical properties was observed in the case of composites made by functionalized CNTs and polymer blend. As compared to pristine CNTs toughness the functionalized CNTs increased the toughness tremendously from 128 to 215 about 60% and elastic modulus increased from 6.6 to 50 about 14%.The Nano composite PCNs also show low resistivity and semi conducting behavior at 270°C.The properties of these biocompatible blends can be used in biomedical applications involving the biological systems which need smaller charge in medicinal apparatus, bio electrodes coatings, etc.

Keywords: Multiwall carbon nanotubes, polycarbonate, poly methyl methacrylate, polystyrene, nano-composite, strength, electrical properties.

1 INTRODUCTION

Due to the outstanding properties of carbon nanotubes like electrical, mechanical, optical, chemical and thermal properties are being considered as promising candidate for the development of CNTs based nano composites. Because CNTs have many potential applications in high strength and conductance, sensors, radiation sources and field emission displays energy storage and energy conversion devices, probes and nano-sized semiconductor devices [1-3]. Thermal stability and flam retardancy were greatly improved by the incorporation of CNTs in the polymer based nano composites. By the incorporation of CNTs in to the poly composites not only dramatically increased the modulus and strength but they also elevate the glass transition temperature, melting temperature, decomposition temperature and transport number.

CNTs based nano composites have good conducting properties therefore they are used as metal for conducting purposes. Besides these CNTs improved other properties of nano composites like optical properties, photovoltaic and hydro-

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phobic properties [3, 4]. To explore novel properties for future application CNTs are being modified with polymers [2]. CNTs exist in two forms i.e. single walled carbon nanotubes (SWCNTs) and multiwall carbon nanotubes (MWCNTs). MWCNTs have certain advantages over SWCNTs; for example they are not dependent like SWCNTs and are good conductors like copper, and its aspect ratio [5]. High quality polymer-CNTs composites required homogenous dispersion that is difficult to achieve. There are various techniques used for composite preparation like melt mixing, sonication where recently functionalization with chemicals and solubilization of CNTs is an effective mean for homogenous dispersion [5, 6].

A lot of researcher studied MCNTs, but recently considerable attention was given to functionalized or modified MWCNT with polymer in polymer composites preparation. MWCNTs chemically modified by an ionic liquid have been used as additives of ionic liquid lubricants in steel-steel contact [7].

The preparation of CNTs polymer composites have been reported by various researchers in literature. Han et al. prepared polycarbonate/MWCNTs by ultra-sonication and studied it properties [8]. Gultner prepared amino functionalized MWCNTs composite with polycarbonate (PC), poly(styreneco-acrylonitrile)(SAN) and reactive component (RC) the blend were tuned for localization [9] Lahelin *et al.* prepared polystyrene (PS)/CNTs, Poly (methyl methacrylate) /CNT composites and were characterized after optimized conditions by using SEM and TEM. The composites were also analyzed for thermal, mechanical, electrical and conductive properties [10]. Shah and Rizvi prepared PS/MWCNTs Nano composites and characterized the resulting Nano composites by thermal, elec-

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trical and thermo gravimetric/differential thermal analysis (TG/DTA techniques [11]. Carrion et al. prepared PC/PS/PMMA/SWCNTs Nano composites and the dry tribological performance of the new samples were determined using SEM and TEM microscopy, TG analysis, x-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The best tribological performance was determined for PS/CNT and PMMA/CNT, with a wear rate reduction with respect to the neat polymers of 74% and 63%, and of a 65% and 60% with respect to PS/CNT and PMMA/CNT, respectively [7]. The current studies focus on the comparison of pristine polymers (PC, PMMA and PS), polymer blends (PC/PMMA/PS), and Nano composites of PCNs and f-CNs using FTIR characterization and mechanical properties.

2 **EXPERIMENTAL**

The material used were MWCNTs purchased from Nano port Co. Ltd, China, having diameter 20-30 nm, length 50-70 μ m. PC supplied by CHI Mel Company, with density 1.22 m³ and melting point 147°C.PMMA used was purchased from Panasonic Manufacturing Malaysia Berhhad (density: 1.18 m³, melting point: 160°C).PS having density 1.12 m³, melting point 240°C from Diamond Company. All the other chemicals including Sulphuric acid (H₂SO₄) 98%, Nitric acid (HNO₃) 65%, hydrochloric acid (HCl), distilled water and deionized water were of analytical grade.

MWCNTs (6.0 g) was taken and placed in muffle furnace at 450°C for 4 h followed by treatment with HCl to remove amorphous carbon and other metal traces. MWCNTs was mixed with the mixture of 65% HNO₃ and H₂SO₄ 95% in 1:3 ratios and kept the solution in 40 KHz ultrasonicator at 90°C for 4 h followed by magnetic stirring at 50°C for 3 h using reflex condensation. The solution was diluted 7 times for pH 5.5 maintenance. The carbon nanotubes was filtered using 0.2 μ m size polypropylene membrane followed by drying in oven at 120°C for 7 h.

The PC/PMMA/PS blends were prepared using 85% PC, 10% PMMA and 5% PS at 260°C and 270 °C for different cycle time (i.e., 5 min, 10 min and 15 min) were prepared using HAAKE minilab. The blend of 0.1% pristine was cross linked 0.3% functionalized **MWCNTs** with to form PC/PMMA/PS/MWCNTs Nano composites. After blending, the polymeric materials were transferred to the pellitization pelletized the sample in the form of wires and finally convert these wires into pellets. After pillarization the pellets were then pressed in the form of thin film sheets of about 0.5 mm thickness and 5-6 cm in length at 210 °C for 20 min and 200 bar pressure by using hot press. These thin films are carefully cut precisely into desired length and shape with the help of scissor in order to prepare the samples for electrical and mechanical analysis. The composition of blend at different temperature and in different time interval is given in Table 1.

Fourier Transform Infra-red Spectrophotometry (FT-IR, Nicolet, 6700) from Thermo Electron Corporation, USA was used for structural analysis of various samples, like functionalization of multi-walled CNTs (F-MWCNTs) and attachment of functional groups to MWCNTs and polymeric materials.

The mechanical properties of all samples were analyzed via

TABLE 1 SCHEMATIC REPRESENTATION FOR PREPARATION PC/PMMA/PS BLENDS WITH PRISTINE AND FUNCTIONALIZED MWCNTS.

Stage 1				Stage 2													
				Ī	A				В								
X=PC					PC	CNs=	=X				F-PCNs=X						
`	(85%)+PMMA(10%)+PS(0.5%)				%	(99.9%)+MWCNTs (99.7%) +f- (0.1) MWCNTs (0.3%			3%)								
2	260°C 270°C				260°C 270°C 260°C 27			70°	С								
Μ	Minutes Minutes			es	Minutes		М	inut	es	Minutes		Minutes					
0 5	1 0	1 5	0 5	1 0	1 5	0 5	1 0	1 5	0 5	1 0	1 5	0 5	1 0	1 5	0 5	1 0	1 5

SHENZEN Universal Testing Machine for ambient conditions for cross head speed of 5mm/min and 0.5KN load.

The data obtained from pristine polymers, their blends and Nano composites are determined and compared with each other. In order to study the electrical behavior of pure polymer (PC, PMMA and PS), PC/PMMA/PS and PCNs and f-CNs, the samples were subjected for impedance spectroscopic studies in which electrical quantity is measured as function of electrical field frequency. These complex quantities include electrical impedance Z, dielectric permittivity ε_{i} electrical modulus M, admittance Y and loss tangent tan δ . The substrate areas were 300 ± 37 mm². The electrolytes contained in the three elements cell was an aqueous solution of 1MLiClO₄. The active geometric area of the working electrode was $144 \pm 24 \text{ mm}^2$. The counter electrode was platinum and the reference electrode was Ag/AgCl. Impedance measurements were performed with a frequency response detector (Model 1025) driven by a potentiostat /galvanostat apparatus (mod 263A) from EG&G Instruments. The data were acquired using WINDETA software having fully automated analyzer interfaced with a computer. Z View software was used for fitting measured results.

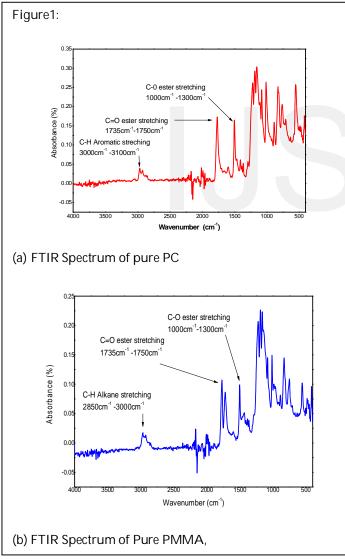
3 RESULTS AND DISCUSSION

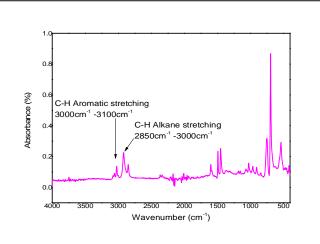
The functionalization of multi-walled carbon nanotubes (MWCNTs) involve surface modification by treating them with a mixture of acids i.e. HNO3, HCI and H2SO4 to produce functionalized/Modified MWCNTs which possess OH and COOH groups on its walls/surface. The interaction between MWCNTs and polymeric matrix is greatly affected by the nature of binding forces which depends upon the dispensability. Different PC/PMMA/PS blends were prepared using 260 °C and 270 °C with cycle times 5 min, 10 min and 15 min. Among the blends prepared homogenized blends were formed at 270 °C with 15 min cycle. Pristine MWCTs and f-MWCNTs were separately added with the blends (PC/PMMA/PS polymer matrix) to produce polymer Nano composites (PNCs) using identical temperature and cycle timing as for polymer blends. Pristine polymers, MWCNTs, polymers blend, f-MWCNTs, blend of pristine MWCNT with polymer blend (PNCS) and

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were subjected to FTIR, for evaluation of the presence of OH and COOH groups and other function group's elucidations. FTIR spectrum of pure polycarbonate (PC) in Fig. 1(a) shows a peak around 3000 cm-1-3100 cm-1 is magnified due to C-H aromatic stretching. However, the peak around 1735 cm-1-1750 cm-1 and 1000 cm-1-1300 cm-1 are due to C=O and C-O ester stretching. FTIR spectrum in Fig. 1(b) represents pure methyl methacrylate (PMMA) showing peaks around 2850 cm-1-3000 cm-1 are due to C-H alkane stretching. The peak at 1735 cm-1-1750 cm-1 and 1000 cm-1-1300 cm-1 are due to ester stretching of C=O and C-O bonds, respectively. Pure Polystyrene (PS) FTIR peak of C-H aromatic stretching appears at 3000 cm-1-3100 cm-1 and the peak of C-H alkane stretching appears at 2850 cm-1 -3000 cm-1 as shown in Fig. 1(c).

FTIR spectrum in Fig. 1(d) of PC/PMMA/PS blends shows peaks of C-H aromatic stretching appears at 3000 cm⁻¹-3100 cm⁻¹. The peak of C=O ester stretching appears at 1735cm⁻¹-1750cm⁻¹ and the peak of C-O ester stretching appears at 1000 cm⁻¹-1300





(c) FTIR Spectrum of Pure PS

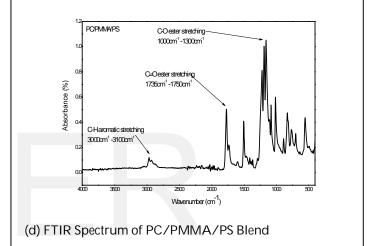
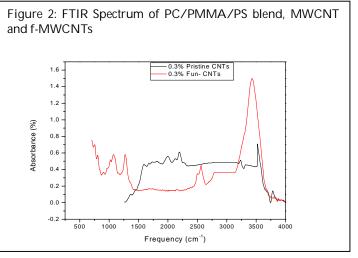


Fig.2 reveals the fact that there is no observable change in the FTIR traces of the composites due to presence of MWCNTs and Carboxylic group of f-MWCNTs. It may be due to very low concentration of the MWCNTs in the system. The other factor may be the fact that both MWCNTs and PC have carboxylic molety which show peaks at the same positions i.e. major peaks at 1768cm⁻¹and 1510cm⁻¹Respectively (12).



International Journal of Scientific & Engineering Research Volume 6, Issue 10, October-2015 ISSN 2229-5518

The improvement in the mechanical properties of polymer matrix by dispersion of MWCNTs was investigated by tensile testing the results are given in table 1.Pure polymer i.e., PC, PMMA, PS were blended together to produce homogenized blend by optimizing the processing condition of temperature, time and torque. The stress strain behavior of blends compared to pristine polymer shows a combination of mechanical properties (Fig.3). Modulus of elasticity for PC, PMMA is very high and very low for PS. Addition of PS decreases the overall modulus of elasticity for blends producing an intermediate (13).

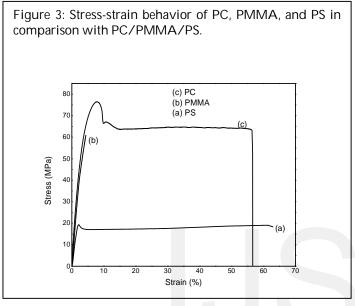
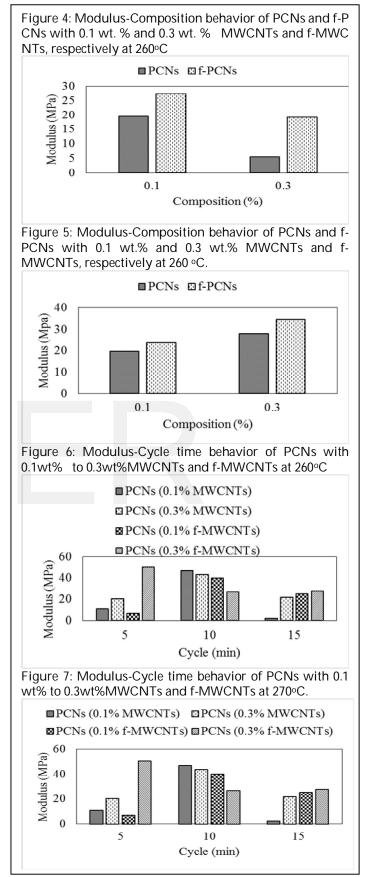


Table 2 Mechanical properties of PC, PMMA, PS and its blend (PC/PMMA/PS)

Composition	Tensile Strength MPa	Modulus of Elastici- ty MPa	Fracture Strength MPa	Elongation at break %	Toughness MJ/m3	
РС	170.46	6021.8	54.64	29.3	3586	
PMMA	107.5	5444.98	39.95	4.51	152.56	
PS	40.3	69.73	19.06	57.91	1113.60	
Blends	53.01	2.3	44.48	9.08	31.8	

Different compositions of pristine MWCNTs without modification for different processing temperature and time were blended with polymer blend. The modulus verses composition and modulus verse cycle time of dispersed MCNTs in blends show irregular trend in mechanical properties because of the improper dispersion of MWCNTs into the polymer matrix. This also resulted in phase separation between polymer matrix and MWCNTs.

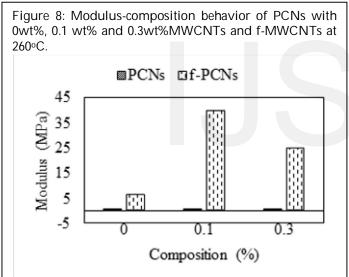


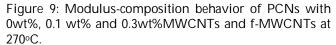
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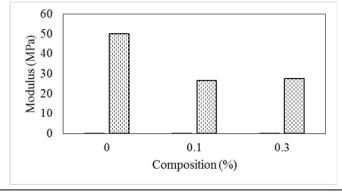
Table 3 Mechanical properties of PCNs at 260°C, 270 °C (Modulus vs. Composition and Modulus vs. Cycle time)

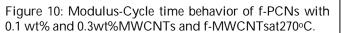
Composition	Cycle time (mins)	Tensile Strength MPa	Modulus of Elas- ticity MPa	Fracture Strength MPa	Elongation at break (%)	Toughness MJ/m3
0.10	5	67.1	34	67.1	4.01	161.9
0.1% MWCNTs at 260oC	10	73.78	8.33	73.78	5.50	274.18
(0.1%)	15	83.66	16.66	83.99	9.47	555.05
	5	75.26	13.15	42.64	20.5	128.93
0.3% MWCNTs at	10	81.50	0.90	40.36	11.93	705.39
260oC	15	94.75	2.38	39.96	26.6	215.9
0.1%	5	82.92	10.83	2.75	13.34	606.27

As MWCNTs showed irregular blend for mechanical properties dispersed in to the polymer matrix. The f-MWCNTs were used for production of PCNs. The modulus verses composition and modulus verses cycle behaviors of PCNs were compared with their polymer blends and other properties.









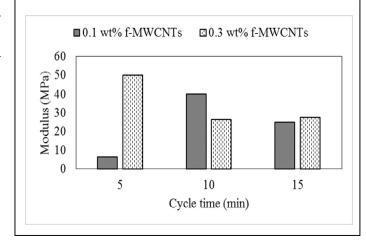


Table 4:

Mechanical properties of pristine CNTs at 260oC (Modulus vs. Composition and Modulus vs. Cycle time)

Compo- sition	Cycle time (Mins)	Tensile Strength Mpa	Modulus Of Elastici- ty MPa	Fracture Strength MPa	Elonga- tion at break (%)	Tough- ness MJ/m ³
	5	67.1	34	67.1	4.01	161.9
Pristine at 260°C (0.1%)	10	73.78	8.33	73.78	5.50	274.18
	15	83.66	16.66	83.99	9.47	555.05
	5	75.26	13.15	42.64	20.5	128.93
Pristine at 260°C (0.3%)	10	81.50	0.90	40.36	11.93	705.39
(0.070)	15	94.75	2.38	39.96	26.6	215.9

Composition of f-MWCNTs and different processing temperature by dispersing f-MWCNTs into the polymer matrix at 260°C for 5, 10 and 15 min cycle was observed that by incorporation of f-MWCNTs the elastic modulus of the f-PCNs was increased as compared to PC/PMMA/PS blend because f-MWCNTs acting as reinforcement material.

The maximum enhancement in elastic modulus was observed in f-MWCNTs were in carp orated at 270°C for 5min cycle time, because at these processing conditions the f-MWCNTs were properly dispersed into the polymer matrix

The conductivity in a sample material with dielectric properties should be measured by using the ac impedance spectroscopy technique. The impedance (Z) is defined as the ratio of the voltage to the current at a given frequency, and it is represented as a complex quantity that consists of a real part (resistance, Z' or R) and an imaginary part (reactance, Z" or XC - XL) with phase angle Θ .

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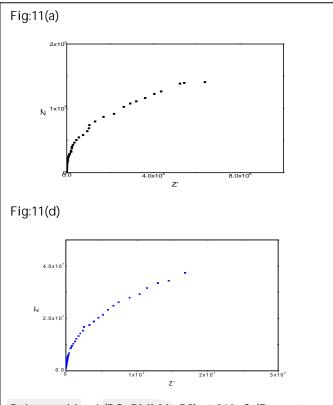
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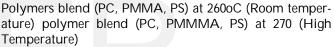
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Table 5
Mechanical properties of f-MWCNTs at 270oC (Modulus
vs. Composition. Modulus vs. Cycle Time)

Composition	Cycle time (min)	Tensile Strength MPa	Modulus of Elas- ticity MPa	Fracture Strength MPa	Elongation at break (%)	Toughness MJ/m3
	5	74.0	6.66	74.0	3.74	5.01
0.1% f- MWCNTs at 270oC	10	28.3	40	28.3	0.98	17.7
	15	10.0	25	10.0	0.97	156.6
	5	75.2	50	42.08	20.69	128.09
0.3% f- MWCNTs at 270oC	10	81.6	26.6	40.97	11.9	705.39
	15	101.0	27.5	39.63	26.6	215.9

With the changing of temperature, impedance plane plots of these blends and PNCS samples showed different modulations. Figure 11 shows a decrease in the diameter of semi-circle of the impedance plane plot curves of polymer blend (PC, PMMA, and PS) with the increases of temperature (room temperature). At room temperature all the data was scattered and the semi-circle is not complete which indicate that the polymers are highly resistive and cannot conduct electricity. Therefore the temperature is divided into two regions to highlight main results of impedance plane plots. In the temperature region at 260°C (room temperature), only one semicircular arc is observed due to highly resistive materials of polymer blend and show low conductivity result as shown in Figure 11(a). At 270°C (room temperature), high frequency data is close to each other and low frequency changes can be clearly seen. At 270°C show at better result as compared to 260°C because the resistance of the material decreases and shows semi conducting behavior because of more exposure of polymers (PC, PMMA, PS) to high temperature as shown in Figure 11(b). The addition of pristine- CNTs Blends and functionalized CNT to polymer blend at different temperature at 260°C and 270°C signifies overall lowering the electrical resistance and increase the Electrical Conductivity as shown in Figure 11(e)- 11(f). The common spectral feature observed that for both polymer blend and a mixture of pristine f-CNTs Blends sample are that with increases of temperature an decrease in the size of the diameter of semi-circle of the impedance plane plot arcs result in shift from the center of arcs which eventually decrease the resistance of the polymer blend . Such behavior exists in semiconductor material (i.e. decrease in resistivity with increasing temperature) and the phenomenon is referred as negative temperature coefficient of resistance (NTCR).







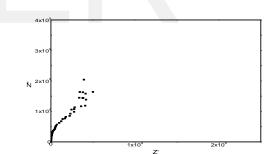
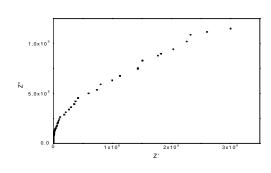
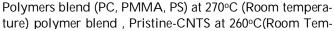
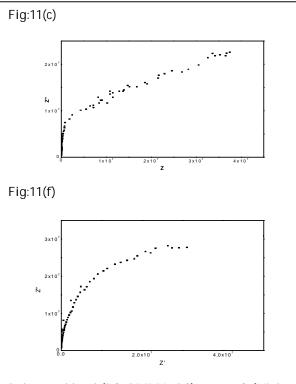


Fig:11(e)

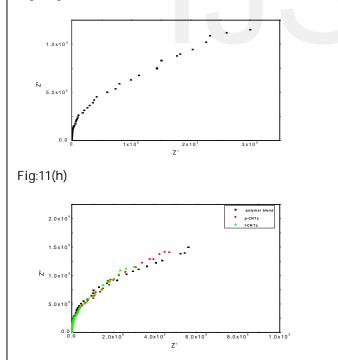


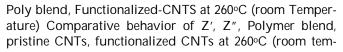


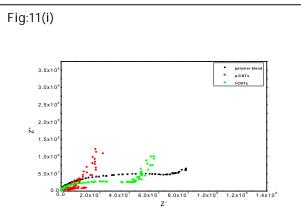


Polymers blend (PC, PMMA, PS) at 260oC (High temperature) poly blend, Functionalized-CNTS at 270oC (High Temperature)

Fig:11(g)







Comparative behavior of Z', Z", Polymer blend, pristine CNTs, functionalized-CNTs at 270 (high temperature)

Comparing the impedance and the subsequent conductivity behavior of Z', Z" of Polymer blend, pristine CNTs, functionalized-CNTs at in different measurement conditions of temperature that at 270 °C (high temperature) and 260 °C (room temperature) as shown in Figures 11(H-I), it was revealed that the conductivity of the polymer blend along with the pristine-CNTs as well as functionalized CNTs at 270 °C (high temperature) is much better as compared to temperature at 260 °C because of low resistivity and more dispersion of functionalizes CNTs as compared to pristine CNTs in the polymer blend. At low frequency changes in impedance can be seen while at higher frequency all curves merges and cannot give semi- circle, so at high frequency changes is not appreciable.

4 CONCLUSION

New Nano composites PCNs and f-PCNs of PC/PMMA/PS with MWCNTs and functionalized MWCNTs by HNO₃ and H_2SO_4 (1:3) mixture, (f-MWCNTs) and the sample were characterized by FTIR, mechanical and electrical techniques and the results of pure PC, PMMA and PS polymer were compared with PC/PMMA/PS blend, and with PCNs, f-PCNs. It was found that the addition of PS decreased the overall modulus of elasticity, PCNs showed irregular trend in mechanical properties because of the improper dispersion of MWCNTs which also resulted in phase separation between polymer matrix and MWCNTs. Elastic modulus of f-PCNs was increase as compared to PC/PMMA/PS blend as f-MWCNTs are acting as reinforcement material.

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