



Influence of Electron Accelerator Irradiation on Epoxy Nanocomposite Materials for Spacecraft Structure

A. Anwar^a, S. Lotfy^{b*}, M.R. Balboul^c and M. Shamekh^d

^a Space Technology Center, Cairo, Egypt

^b Polymer Chemistry Department, National Center for Radiation Research and Technology, Egyptian Atomic Energy Authority, Cairo, Egypt

^c Solid State Physics and Accelerators Department, National Center for Radiation Research and Technology (NCRRT), Atomic Energy Authority (AEA), Cairo, Egypt

^d Materials Science and Technology Department, Military Technical College (MTC), Cairo, Egypt

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Different types of surfactants namely, nonionic, anionic and cationic represented by Polyoxyethylene sorbitan monooleate (Tween 80), sodium dodecyl sulfate (SDS), and cetyltrimethylammonium bromide (CTAB) respectively were used to select a proper type of surfactant enhancing dispersion quality of multiwall carbon nanotubes (MWCNTs) in the epoxy resin. In this study, the effect of electron beams which are one of the most severe space environment threats. The candidate material proposed in this study will be used as a structural space material. The candidate material is characterized by further transformation infrared spectroscopy (FTIR) so as to identify the mechanical properties, surface tension, and electrical dispersion measurement. The mechanical results revealed that the strength increases by 10% while adding the CTAB surfactant, however, it decreases by 27% and 32% by adding Tween-80 and SDS respectively. The anionic surfactant SDS, despite keeping the stiffness, the reference sample is of lower strength and elongation. The CTAB improves the mechanical properties by improving the strength and stiffness while elongation is significantly decreased by adding any of the surfactants. The surface tension of Tween 80 and anionic surfactant SDS, is $\sigma = 24.4$ mN/m while the surface tension in case of the CTAB is $\sigma = 25.4$ mN/m. The surface tension and electrical dispersion measurement results reveal that the nonionic surfactant Tween 80 led to a uniform dispersion of MWCNTs in the epoxy than other surfactants. The effect of 100-kGy irradiation via electron beam on structure and its electrical properties of the epoxy composites was studied. Improving the dispersion quality of the MWCNTs in epoxy nanocomposite materials leads to utilizing these materials in spacecraft structure.

Keywords: Spacecraft structure, epoxy, nanocomposites and electron accelerator irradiation

Introduction

The polymeric materials are currently counted as one of the most important materials used in manufacturing the spacecraft structure[1]. So as to fulfill the required specifications of the spacecraft structures, they must be made of lightweight

materials to endure the static, dynamic and thermal stresses that affect the spacecraft during launching, deployment, and operation of these structures [2]. The structural space materials are exposed to harsh environment threats; electron beam is one of these environment species. The accommodation of the

spacecraft equipment is provided by the structure. In addition, the structural materials protect the spacecraft entire system. According to the spacecraft altitude, corresponding to the mission, the electrons and protons beam are considered an important threat that affect the spacecraft mission. The sequences of the developments of the spacecraft structures since the first satellite "Sputnk-1" in 1957 are the metallic-structure, the composite of fiberglass composite sandwich panels. Then boron fiber-epoxy resin composites were used after those carbon fibers to replace the boron in fibers-epoxy-graphite composites[2]. There is a need to develop the materials and techniques spacecraft structures.

Advanced composites used epoxy resins which are well-known thermosetting matrices, displaying excellent characteristics for a wide range of applications. Epoxy resin is a radiation-resistant polymer [3]. Epoxy has good mechanical properties, low molecular weight, low cost, ease of manufacturing, low shrinkage (1–5 %) during cure, good physical bonding to other substances, and good chemical resistance[4--6]. The epoxy resin used as a surrounding substance in architecturally strong composite materials in combination with carbon fibers, glass fibers, aramid fiber (Kevlar fibers) etc., are used as fibrous reinforcements for applications such as adhesives[7] in nuclear reactors, high-energy accelerators, automotive aerospace, and military[8,9]. The most widespread form of polymerization from academic and commercial perspectives is the free-radical polymerization technique which is broadly applicable to a wide range of monomers[10]. The effect of the electron accelerator and proton beam irradiation on both the mechanical and chemical properties of polyamide[11] and epoxy composites[12,13] were studied. Ahmad Anwar et al, in 2016, studied one of the space hazards affecting on polymeric materials by exposure of the polyimide to ionized and particulate radiation at a dose up to 1000kGy by gamma source and evaluated its performance[14]. One of the most direct and easy tools of generating radicals is ionizing radiation which initiates polymerization, copolymerization, grafting, and crosslinking of vinyl and vinylidene type of monomers[10]. High gamma doses affected the mechanical properties of the studied material. The results obtained by the authors matched and confirmed by the quantum modeling[14]. The degradation performances of

epoxy-based shape memory polymer have been studied by Hou et al.[15] under 1 MeV electron irradiation, who found that the shape recovery rate decreased rapidly from unirradiated 98.6% to 85.9% with increasing fluence up to $200 \times 10^{14} \text{ cm}^{-2}$.

Multiwall carbon nanotubes (MWCNTs) have optical, electrical, and mechanical properties[16,17]. MWCNTs dispersion is important for their nanocomposites application[18,19]. The mechanical and chemical are two widely methods for MWCNTs dispersion used. Lu et al[20] reported that the mechanical method of MWCNTs dispersion by ultrasonication and mixing via high-shear are time-consuming and less efficient. In the case of the chemical method, the covalent methods depend on improve solubility in solvents via functionalization with the chemical, while the noncovalent method affects by π - π stacking interaction to facilitate the adsorption of the chemical moieties onto the nanotube surface.

Several studies have contributed to the leading role of surfactants in the MWCNTs dispersion through the epoxy matrix [21-25]. Dispersion of MWCNTs in solvents includes chemical treatment to enable unbundling during the occurring of interfaces between the MWCNTs surface and supporting solvent[19]. The classification of surfactants known to be depending on the charge of the head groups, thus, nonionic[26-28] anionic[29,30] and cationic[31,32] The surfaces adsorption to the surfactants generally depend on its chemical properties, surfactant molecules structure and solvent.

In this work, the authors try to use three different types of surfactant as a dispersion tools for MWCNTs in epoxy nano-composites materials for spacecraft structure irradiated by electron accelerator.

Materials and Methods

Materials

The MWCNTs (length 4–10 μm , outer mean diameter 8- 10 nm, inner diameter 4nm, mean number of walls up to 15) were purchased from Egyptian petroleum research institute (EPRI), prepared by chemical vapor deposition method (CVD). The purity of the neat MWCNT is greater than 90%. Biresin® CR82 is composed of two components biresin CR-82 part A, and CH-80-6 part B (hardener), epoxy resin system were purchased from Sika Advanced Resins (SIKA

Deutschland GmbH), Germany. The same epoxy resin system was used in many research published papers [33-35]. The neutral surfactant; poly oxy ethylene sorbitan mono oleate (Tween 80) were purchased from MP Biomedicals, Inc. France. The anionic surfactant; sodium dodecyl sulfate (SDS) >99% were purchased from El Nasr Pharmaceutical Chemicals Co (ADWIC), Egypt. The cationic surfactant; cetyltrimethylammonium bromide (CTAB) 99% were provided by sigma Aldrich Chemical Co., USA. All chemicals used without additional purification.

The chemical molecular structure of three surfactants (a) Neutral surfactant; Tween 80; (b) Anionic surfactant; SDS and (c) Cationic surfactant; CTAB are presented in Figure (2).

Preparation of the epoxy-MWCNTs nanocomposites

Three types of surfactants; nonionic (neutral) surfactant; Tween 80[28], anionic surfactant; SDS[25] and cationic surfactant; CTAB[36] were applied to enhance dispersion of MWCNTs in the epoxy resin. A desired amount of surfactant which is relatively high concentration[37]; 15 fold of critical micelle concentrations (CMC) for each surfactant dissolved in organic solvent (acetone), as the concentration of surfactants above CMC, causes micelles to form so all additional surfactants added to the system to form micelles (Table 1).

The procedure implemented for the treatment is based on previously reported method[22]. Multi-walled carbon nanotubes 0.5 % dispersed in acetone containing the surfactant and the mixture was subjected to the horn of 6 mm of an ultrasonic homogenizer; model TU-65-Y with ultrasonic power of 650W. The horn sonicator operates at 40% power amplitude, a 5-second pulse on, and a 1-second pulse off. The ultrasonication time of 60 minutes was used to obtain proper dispersion of MWCNTs in organic solvent. The mixtures was left to be cold at room temperature before adding the hardener CH-80-6 part B for curing the epoxy resin system. Next, the curing of all samples were carried out via mixing curing agent CH-80-6 part B (the hardener) for curing the epoxy resin system into MWCNTs-epoxy nanocomposite in an aluminum mold and silicones pans which represented in Figure (1). Then, the organic solvent and the bubbles were removed under vacuum (< 200 mbar) at 50 °C for 24 hours.

Surface tension

Force Tensiometer K20 Version 1.25. For measurements of the surface tension, the ring method was utilized[38] with these parameters; stage speed 25 %, reading limit 10, standard deviation 0.01 mN/m standard deviation values 5, acceleration of gravity 9.807 m/s², correction methods based on tables which have been determined empirically by the authors Harkins and Jordan (H&J)[39].



Figure (1): The aluminum mold and silicone pans used for molding the samples



Figure (2): Chemical molecular structure of three surfactants. (a) Neutral surfactant; Tween 80; (b) Anionic surfactant; SDS and (c) Cationic surfactant; CTAB

The surface tension σ according to the following equation:

$$\sigma = \frac{F}{L \cdot \cos\theta}$$

Where F is force maximum F_{\max} , L is the wetted length of the ring, which is the sum of the inner and outer circumference.

Electron beam accelerator irradiation

The irradiation of samples was carried out at a constant dose of 100 kGy in the presence of air using an ICT electron beam accelerator at the National Center for Radiation Research and Technology, Cairo, Egypt. The irradiation was performed at a beam current of 16 mA, an

accelerator energy of 2.7 MeV, and a conveyor speed of 1.08 m/min.

Table (1): The amount of surfactants used as 15 fold of critical micelle concentrations

CTAB		Tween 80		SDS	
CMC	Molecular Weight	CMC	Molecular Weight	CMC	Molecular Weight
1.0 mM	364.5 g/mol	13-15 mg/L	-----	8.2 mM	288.38 g/mol
Amount of CTAB in 100 ml for 15CMC \approx 0.55 g		Amount of Tween 80 in 100 ml for 15CMC = 0.021 g		Amount of SDS in 100 ml for 15CMC \approx 0.23 g	

Mechanical tensile test

This test was conducted in the Military Technical College (MTC) according to the ASTM D638-14[40] on five specimens for each sample to secure 95% level of confidence. Each sample was described in the section 2.2 as they have the same constituents' percentage, but different types of surfactants. To evaluate the tensile properties of the tested samples, a Galdabini universal tensile testing machine (Quasar 100) was used; the test was conducted at room temperature with a load cell 100 kN and crosshead rate 5 mm/min.

Electrical dispersion evaluation of MWCNT/epoxy nanocomposites

Electrical test instrument (Keithley 2635A SourceMeter® instruments USA) was used for resistance measurement on investigate specimens with dimensions of $60 \times 40 \times 2$ mm. The electrical resistance of multiple points on the specimen surface was measured using copper electrodes. Four test points were carefully selected to measure the electrical resistance of each sample as shown in Figure (3). It has been assumed that a uniform dispersion of MWCNT nanocomposites in epoxy would be associated with very close identical resistance values. Measurements were made before and after exposure to electron beam irradiation at a dose of 100 kGy. The pure epoxy sample (without adding MWCNT) was excluded from the electrical resistance measurement due to high insulation values.



Figure (3): Four test points for dispersion evaluating system epoxy nanocomposites materials

Results and Discussion

Surface tension

Disaggregation of MWCNTs in epoxy matrix is a critical challenge, since it tends to be self-associate into micro-scale aggregates[41]. To improve mechanical and electric performance of MWCNTs epoxy composites, the surfactant was used as a dispersion tool. Three different types of surfactant namely, nonionic; Tween 80, anionic; SDS, and cationic CTAB were investigated to enhance dispersion of MWCNTs in the epoxy resin. The surface tension of water was measured using the ring method[38] to confirm the validation of the surfactant effect. The water surface tension yielded an average value as $\sigma = 70.7$ mN/m which agrees with the previously provided results[42]. The results reveal that there is a significant decrease in the surface tension of water by the addition of surfactants as shown in Table(2).

Table (2): Surface tension changes by the change of the surfactant type

	Density (g/ml)	Surface tension (mN/m)	St.-Dev. (mN/m)
Epoxy	1.1	26.7	0.07
Acetone	0.79	22.8	0.01
Water	0.998	70.7	0.03
Water SDS	0.998	30.4	0.09
Water CTAB	0.998	33.7	0.03
Water Tween 80	0.998	36.4	0.09
Epoxy-Acetone	0.79	24.2	0.03
Epoxy-MWCNTs-Acetone	0.79	25.1	0.02
Epoxy-MWCNTs-Acetone SDS	0.79	24.4	0.06
Epoxy-MWCNTs-Acetone CTAB	0.79	25.4	0.05
Epoxy-MWCNTs-Acetone Tween 80	0.79	24.4	0.04

In case of epoxy resin composites, the changes in the surface tension were represented in slight

decreases from $\sigma = 26.7$ mN/m of epoxy resin to $\sigma = 24.2$ mN/m after dilution the epoxy resin with acetone. The changes in the surface tension of the epoxy-MWCNTs acetone composites was found to be in the order of $\sigma = 24.4$ mN/m in case of the nonionic surfactant; Tween 80 and anionic surfactant; SDS, $\sigma = 25.1$ mN/m in case of the surfactant free and $\sigma = 25.4$ mN/m in case of the cationic surfactant CTAB. According to surface tension experimental results, the dispersing power of the surfactants follows the following trend:

Tween 80 = SDS > surfactant free > CTAB

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Dispersing power

The obtained results reveal the superiority of the nonionic and anionic over the cationic surfactants and the surfactant free epoxy in dispersing the MWCNTs in epoxy matrix[43]. Surfactant molecules direction as that hydrophobic tails face the MWCNTs, while hydrophilic heads face the epoxy, causing a lowering of the epoxy-MWCNTs interfacial tension. The adsorption of surfactant onto MWCNTs is because of interactions of the hydrophobic surfactant tails[16,44]. The MWCNT surface tension lowered because of the physical adsorption of surfactants on the MWCNTs surface, which prohibits the formation of aggregates. The enhanced interfacial interactions gave a rise to improved dispersion of MWCNTs in the epoxy matrix. Similar results were found in earlier publications where dispersion of the MWNTs in acetone was increased by adding a nonionic or anionic surfactants to produce epoxy-MWCNTs composites[45-46] Micelles formation disables the van der Waals forces to prevent MWCNTs to form aggregation[47].

FTIR analysis of the epoxy blank, epoxy-MWCNTs, epoxy-MWCNTs SDS, epoxy-MWCNTs CTAB, and epoxy-MWCNTs Tween 80 before and after exposure to electron beam irradiation with a dose of 100 kGy were carried out and presented in Fig. 3(A), (B). All the spectra were measured at range 400 - 4000 cm^{-1} wavenumber. The FTIR spectra show peaks at 823 cm^{-1} of an epoxy group[48,49] while the peaks -CH₂ at 1470 cm^{-1} , C-H bending at 1400 cm^{-1} and C-H stretching band at 3000 cm^{-1} of MWCNT-epoxy composites which conforming to aromatic C-H, C-C and C=C groups of MWCNTs fillers[47]. Bands found at the range of 1450, 1510 cm^{-1} indicated the presence of S=O stretching

vibration of SO₄ from the sulfate head group of SDS molecule[30]. Peaks related to C-H stretching vibration of methyl and methylene groups (2933 and 2850 cm^{-1} , respectively) of CTAB are distinguished[50]. The spectrum of (epoxy-MWCNTs Tween-80) composites the presence of the peaks at 946 cm^{-1} , 1100 cm^{-1} , 2855 cm^{-1} and 2900 cm^{-1} of the surfactant involving -H₂C-O-CH₂-, -CO-O-CH₂-, -CH₂-CH₃ indicates that the functional groups of Tween-80 have been anchored onto the composites. Also bands around 3400 cm^{-1} show the existence of -OH group which was due to the Tween-80[27]. The products of the radio-oxidation reaction represented in new peaks at 1383 cm^{-1} and 1349 cm^{-1} were observed after exposure to electron beam irradiation[51]. The stretching bands at 1710-1770 cm^{-1} of the carbonyl group are attributed to oxidative species. The epoxy materials generally have a low oxygen diffusion coefficient under high-energy ionizing radiations known as the superficial oxidation phenomenon [52].

Mechanical properties measurements

Figure (5) shows that the surfactants affect the mechanical properties not only of the epoxy neat, but also of the epoxy nanocomposite (epoxy-MWCNTs). From these curves, the elongation presented in the X-axis that occurred due to the loading could be described. The ultimate strength is offered in the Y-axis at which the specimen was fractured and the modulus of elasticity (stiffness) was measured by the inclination of the curve on the x-axis. First, it is noticed that the addition of MWCNT's without surfactants decreases the ultimate strength by about 13% and the stiffness by 20%. Also, the elongation decreased by approximately 10%. In this research, the sample contains epoxy-MWCNTs will be considered as the reference sample. Second, addition of the surfactant with its variable types leads to a change in the mechanical properties and it will be described as follows:

- a) The ultimate strength: it is appeared to increase by about 10% while adding the CTAB surfactant, however, it decreases by about 27% and 32% by adding tween-80 and SDS respectively.
- b) The stiffness, increases by adding CTAB and tween-80 by about 64% and 21% respectively while adding SDS surfactant keeps it with insignificant change.

c) The elongation is clearly decreased by adding any of the selected surfactants.

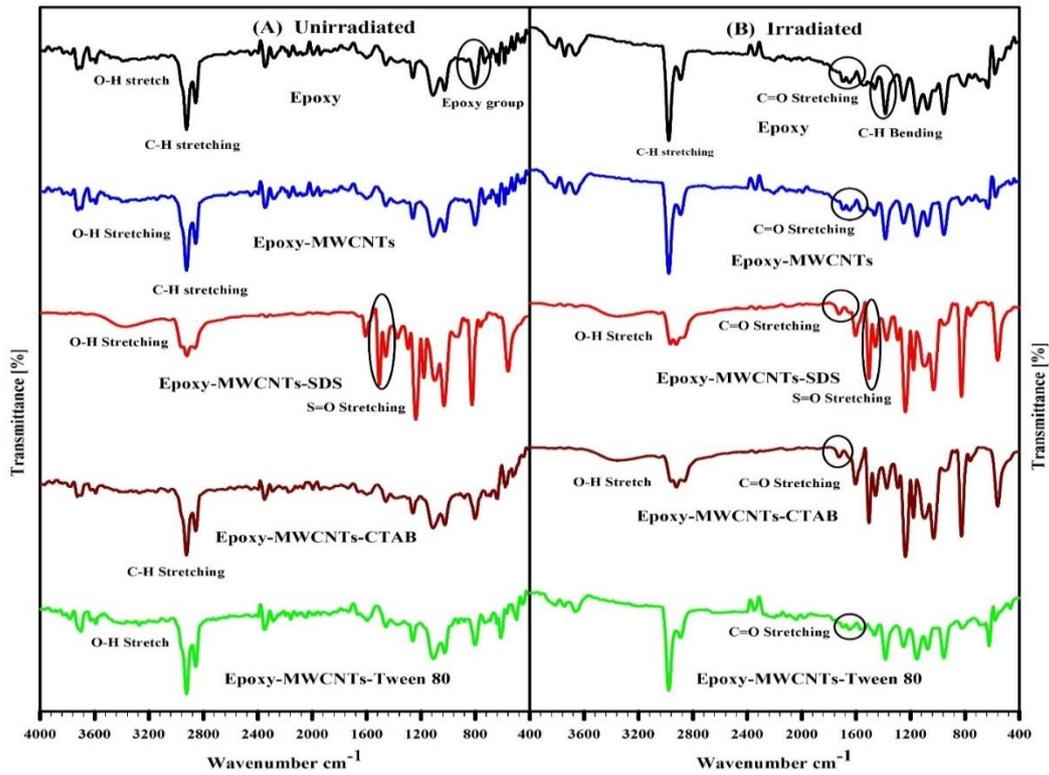


Figure (4): FTIR spectra of (a) unirradiated and (b) 100 kGy electron beam irradiated; epoxy blank, epoxy-MWCNTs, epoxy-MWCNTs SDS, epoxy-MWCNTs CTAB and epoxy-MWCNTs Tween 80

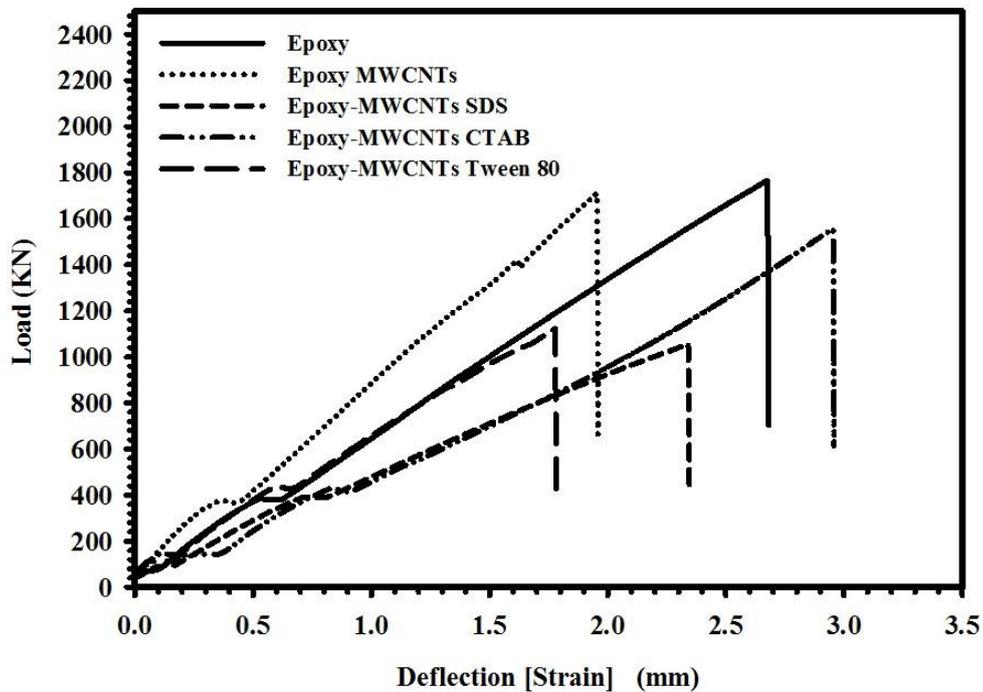


Figure (5): Mechanical properties of epoxy and epoxy-MWCNTs with three types of surfactants

Electrical dispersion measurement

Figure (6) shows the electrical dispersion evaluation results of MWCNT/epoxy nanocomposites for three types of surfactants in addition to blank (without surfactant) for non-irradiated samples. Each electrical test was performed by measuring the resistance at four different points to demonstrate the effectiveness of the MWCNT dispersion distribution.

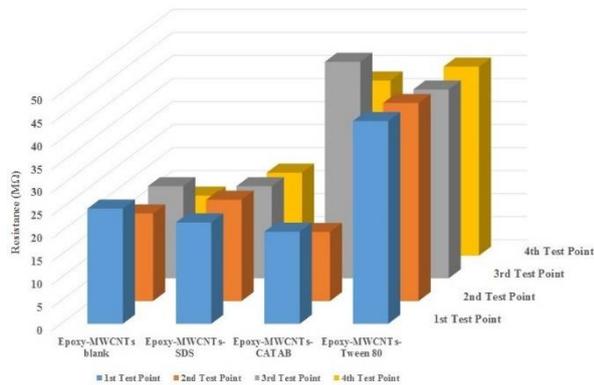


Figure (6): Electrical resistance graph for non-irradiated MWCNT/epoxy blank and three types of surfactants SDS, CTAB and Tween 80

It is clearly from this Figure that we have fluctuation in the resistance for Epoxy-MWCNTs blank in addition to Epoxy-MWCNTs CTAB. On the other hand, the results of both Epoxy-MWCNTs SDS and Epoxy-MWCNTs Tween 80 show very close resistance values for different test areas. This is interpreted as evidence that SDS and Tween 80 surfactant led to a more uniform dispersion of MWCNT in the epoxy as compared to other surfactants. This finding is in a good agreement with the results obtained by surface tension measurements. It is worth to mention that for blank and CTAB surfactant there is a specific area with a very low resistance value which can be explained by high agglomeration of MWCNTs in this area; this is not recommended. In conclusion, in terms of measuring the resistance, the addition of MWCNTs nanoparticles to the epoxy resins reduces the resistance, the more uniform dispersion is the more closely resistive values for the entire sample.

Figure (7) shows the electrical dispersion evaluation results of electron beam irradiated samples after exposure to dose of 100 kGy. The electrical resistance values of all irradiated samples

are increased by different magnitude after e-beam irradiation. The typical processes that occur during e-beam irradiation of pure epoxy polymer are cross-linking and chain scission reactions in dose range up to 100 kGy[53]. On this basis, the significant increase in electrical resistance values after e-beam irradiation can be attributed to the difficulty of electrons mobility between MWCNTs nanoparticles resulting from cross-linking; MWCNTs separation. On the other hand, after e-beam irradiation it could be observed that the best relative type of surfactant is only SDS, which has kept some form of regular dispersion of MWCNT in epoxy.

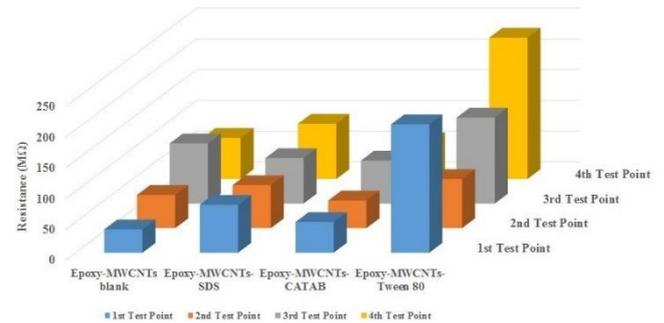


Figure (7): Electrical resistance graph for 100 kGy electron beam irradiated MWCNT/epoxy blank and three types of surfactants SDS, CTAB and Tween 80

Conclusions

Enhancing the dispersion of MWCNTs in the epoxy resin is a critical challenge. Three types of surfactants namely, nonionic surfactant; Tween 80, anionic surfactant; SDS, and cationic surfactant; CTAB were applied in concentration of 15 fold of critical micelle concentrations (CMC). Multi-walled carbon nanotubes 0.5 % dispersed in acetone containing the surfactant then ultrasonication occurred for 60 minutes obtain proper dispersion of MWCNTs in organic solvent. Measurements of the surface tension were conducted using the ring method. The trend of dispersing power of the surfactants is Tween 80 = SDS > surfactant free > CTAB. From the mechanical properties point of view, adding SDS, despite keeping the stiffness, the reference sample, is of lower strength and elongation. In addition, Tween-80 improves the stiffness on the account of the strength. The CTAB surfactant enhance mechanical properties by improving the strength and stiffness. The Tween-80 is not bad if the other properties agreed with improving dispersion quality. The electrical resistance results reveal that SDS and Tween 80 surfactant led to a more

uniform dispersion of MWCNT in the epoxy matrix before irradiation. Whereas, after 100 kGy of electron beam irradiation only SDS surfactant maintained some form of regular dispersion of MWCNT in the epoxy matrix. Therefore, it is highly recommended to use SDS surfactant as it gives an accepted distribution of MWCNT in the epoxy before and after 100 kGy e-beam irradiation.

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