
EFFECT OF GAMMA RADIATION ON THE STRUCTURE AND THERMAL PROPERTIES OF POLYANILINE-COBALT/POLYCARBONATE NANOCOMPOSITE

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ABSTRACT

The induced alterations in the structure and thermal properties of polyaniline-cobalt/polycarbonate, PANI-Co/PC, nanocomposite (NCP) owing to γ irradiation have been investigated. The chemical composition and morphology of the prepared NCP samples were studied using scanning electron microscopy and energy dispersive spectrometry technique. Additionally, X-ray diffraction (XRD), FTIR spectroscopy, thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were performed to investigate the resulted effect of γ radiation. The γ radiation with doses 50-350 kGy leads to the prevalence of intermolecular crosslinking which enhances the disordered phase and increases the bonds between the hydrogen of the PANI amine groups and carbonyl groups of PC. This is reflected in a rise in the degradation temperature values from 400 to 475°C indicating an improvement in the thermostability of the NCP samples. Additionally, the melting temperature increased from 350 to 361°C indicating a more compact structure of the NCP samples.

Keywords: γ radiation, Nanocomposite, XRD, FTIR spectroscopy, TGA, DTA

1. INTRODUCTION

Polymer nanocomposites (NCPs) that include inorganic nanoparticles (NPs) in its matrix attain desirable combined characteristics [1, 2]. The physical properties of these NCPs are altered by varying the percentage of the organic and inorganic constitutes [3]. This modifies the properties of the resulting NCP and allows it to be suitable candidate for different applications [4]. Polyaniline (PANI) is a talented polymer due to its remarkable properties and stability [5]. It is useful in electrical and microelectronic fields [6].

Cobalt nanocrystal got more consideration than Fe and Ni owing to its distinguished characteristics and oxidation-resistance. Cobalt NPs are used in several applications such as catalyst for the Fischer-Tropsch reaction, as coatings and for electroplating [7]. Further, cobalt has two crystal phases: hexagonal close-packed and face-centered cubic. Lately, a new metastable ϵ -phase cobalt that is less dense than the other two phases was discovered by Ding and Banwendi [8]. This structure is cubic (space group P4 1 32) with a unit cell parameter $a = 6.097$. These allow cobalt-based

NPs to be extremely attractive and being efficient NPs [9]. Thus, NCPs based on cobalt have got more attention owing to the significant change in thermal [10], mechanical [11], electrical [12] and magnetic [13] properties relative to the pure organic polymers. On the other hand, Polycarbonates (PC) have significant characteristics such as good toughness, inflexibility and being thermally stable [14]. PC is an appropriate matter for hosting metal NPs owing to its unique properties. The inclusion of metal NPS into the PC matrix has a benefit because the merged matter joins properties from both the organic and inorganic materials [15-19].

The mass fraction of the amorphous portion in the semicrystalline polymers represents an essential task in characterizing the material. Also, the study of thermal characterization of polymers has become extensive. Some polymers have various attractive characteristics that permit it to be one of the most significant polymers. But in spite of its huge technological and financial importance, it still has a lot of problems associated with variation in its physical properties. One of these problems is its poor thermostability owing to the structure

defects created throughout the polymerization route. Therefore, the stabilization of these polymers using irradiation is required [20]. The γ irradiation creates free radicals via degradation, and then forms covalent bonds via crosslinking. This influences the polymer macromolecular construction and its morphology without changing its composition [21]. The bonds that are broken during degradation can be recognized by FTIR analysis of the blank and exposed samples. The current study aims to enhance the structural and thermal properties of PANI-Co/PC NCP using γ radiation, so that the resultant NCP can be used in different applications.

2. METHODOLOGY

2.1 Materials

Aniline monomer, dodecyl benzene sulfonic acid (DBSA, surfactant), Cobalt chloride were obtained from Sigma-Aldrich Company, USA. PC was obtained from Sigma-Aldrich GmbH, Cairo, Egypt.

2.2 Preparation of PANI

A stable polyaniline-dodecylbenzenesulfonic acid (PANI-DBSA) colloidal dispersion was prepared as previously published [22] as follows: aniline monomer and dodecyl benzene sulfonic acid (DBSA) of ratio (3:1) in 75 ml water/isopropanol (IPA) mixture (3:1) have been homogenized for 10 min to form the miniemulsion. Then, 25 ml of ammonium peroxydisulfate (APS) solution (1 wt.%) was added drop-wisely to the former miniemulsion. Polymerization was performed, at room temperature, with strong stirring at 10,000 rpm for 15 min. The color was changed from white anilinium-DBSA complex in water to blue, then turned into dark green. Finally, a green and very stable PANI-DBSA dispersion was obtained. The colloid dispersal is centrifuged, washed by water/methanol mix for several times and then dried in an electric oven for 4 hrs at 70 °C and kept for further use.

2.3 Preparation of PANI-Co NCP

The polymerization reaction has been done; where aniline monomer and DBSA of ratio (3:1) was homogenized by high shearing affect homogenizer at 10,000 rpm in existence of DBSA to form the miniemulsion. A 20 ml of 0.25% cobalt chloride solution has been added

to the formed miniemulsion with continuous strong stirring at 10,000 rpm till a green colloidal dispersion of PANI/Co NCP is obtained. The produced green colloidal dispersion was then centrifuged, washed several time by water/methanol mix, filtered and dried at 70 °C for 4 h [22].

2.4 Preparation of PANI-Co/PC NCP

Three grams of PC powder were dropwisely added to 50 ml of DMF with a continuous vigorous stirring till all PC powder liquefies. Then 0.125 g of the prepared PANI/Co was added to the preceding solution under stirring at room temperature until a homogeneous colored solution is obtained. The obtained solution was then casted onto clean Petri dish and left to dry at 50°C for 24 hours.

2.5 Irradiation facilities

The PANI-Co/PC NCP samples were exposed to γ radiation with doses 50-350 kGy with ^{60}Co source (Canada A.E.A Ltd.) with energy range of 1.173-1.332 MeV at 2.4 Gy/min (A,E.A., NCRRT, Egypt).

2.6 Instrumentation

The chemical composition of the PANI-Co/PC NCP samples was examined by energy dispersive spectrometry (EDS) using scanning electron microscope (JEOL, JSM-5910LV)

The X-ray diffraction (XRD) was conducted using Shimadzu 6000. The X ray diffractometer was operational with Cu- k_{α} ray of $\lambda = 1.5406 \text{ \AA}$ and scan speed of $2^{\circ}/\text{min}$.

FTIR Spectroscopy was performed with a spectrophotometer (Shimadzu, 8201 PC, with an accuracy $\pm 4\text{cm}^{-1}$).

The thermal behavior of the PANI-Co/PC NCP was illustrated applying DTA and TGA Shimadzu-50. For DTA measurements we used a reference material ($\alpha\text{-Al}_2\text{O}_3$ powder). The measurements were performed at $10^{\circ}\text{C}/\text{min}$ with nitrogen gas flow rate $20 \text{ cm}^3/\text{min}$.

3. RESULTS AND DISCUSSION

3.1 SEM Analysis for PANI-Co and PANI-Co/PC NCPs

Fig. 1a shows the SEM micrograph of the synthesized PANI-Co NCP. The Co NPs are formed in the form of clusters or colonies. The

formed NPs are of size in the 44-63 nm. It can be concluded that the formed NPs are in the range of nanosize and represented as bright spots or zones which are quite embedded into the darker zones of polyaniline matrix.

Fig. 1b shows SEM micrographs of the PANI-Co/PC NCP. It is shown that the PANI/Co is well dispersed and compatible with

the PC matrix. The bright spots are for the CO NPs.

Fig. 1c shows the energy dispersive spectrometry image revealing the compositional analysis for PANI-Co NCP. The elements are with percentages near nominal values (Table 1).

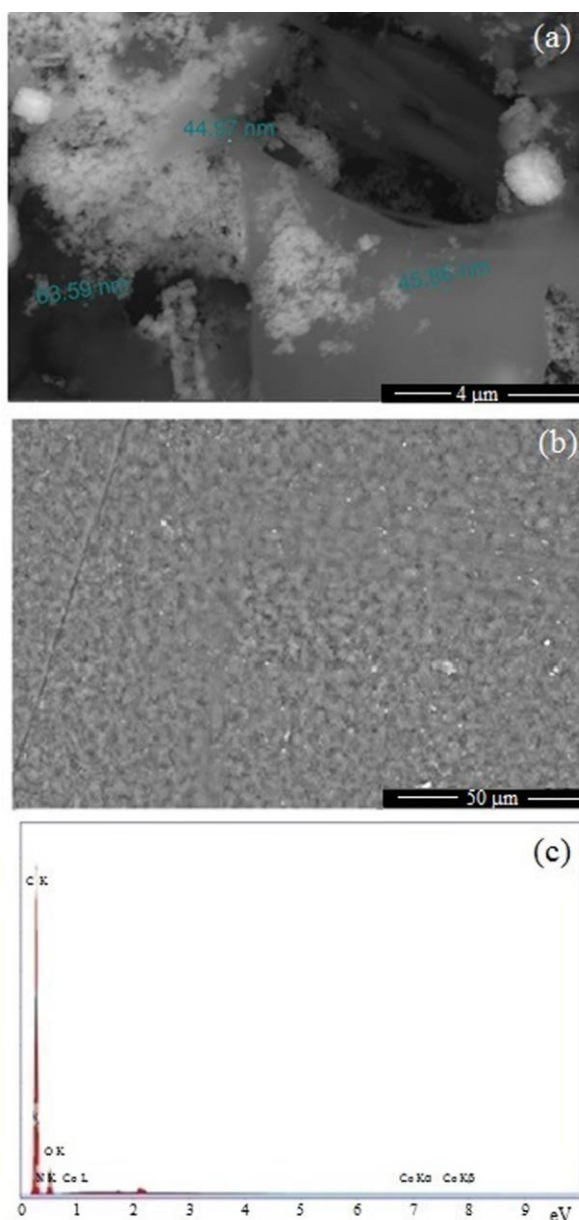


Fig. 1 SEM micrograph of the prepared (a) PANI-Co, (b) PANI-Co/PC NCPs and (c) Energy dispersive spectrometry image of the blank PANI-Co/PC NCP.

Table 1. The chemical composition of the Pani-Co/PC NCP samples

Element	Weight %	Atomic %	Net Int.	Error %
C K	76.14	80.79	1018.38	3.79
N K	3.19	2.90	4.20	31.26
O K	20.41	16.26	100.54	12.09
CoK	0.16	0.03	1.96	64.13

3.2 X ray diffraction

Actually, polymers are semicrystalline containing a mixture of ordered and amorphous phases. The disordered portion in the semicrystalline polymers is responsible for evaluating the performance of the NCP. XRD was conducted on the blank and exposed NCP samples. Fig. 2 shows the XRD patterns of the pristine and exposed PANI-Co/PC NCP samples measured in the 2θ range $10\text{--}60^\circ$. The XRD data showed a wide peak centered at 18° , representing the partial crystalline structure of NCP. This wide peak is due to the amorphous PANI as the amorphous PANI has two wide peaks at $2\theta = 20.4^\circ$ and 24.8° . These peaks are owing to the periodicity parallel and perpendicular to PANI chains, correspondingly [23]. No peaks appeared for Co indicating the incorporation of Co in the NCP matrix. There is slight change in the position of the wide peak after the NCP samples were exposed to γ ; indicating that the lattice parameters were broken [24].

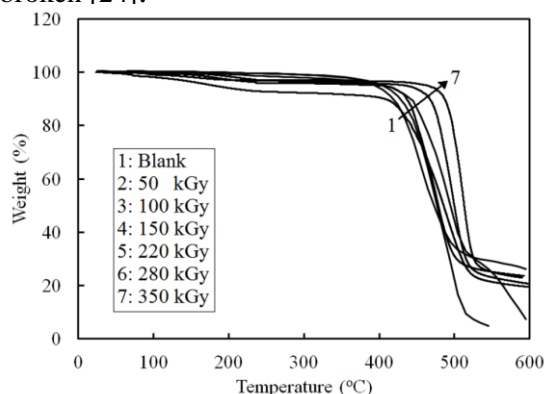


Fig. 2 XRD curves of the exposed PANI-Co/PC NCP samples and the blank.

Lorentz fitting was applied on the diffraction pattern of the peak (centered at 18°) for all NCP samples. Therefore the area under the wide peak, which is the integral intensity I , was computed; then plotted in Fig. 3a versus γ dose. The intensity decreased in magnitude on raising the γ doses to 350 kGy, indicating higher amorphous nature. This can be owing to the action of γ ray that enhances the bonding between the H of PANI amine groups and the carbonyl groups of PC through crosslinking. This reduces the ordered portion and turns it into non-arranged ones. These results can be interpreted based on the fact that the amine and

imine groups involved in the PANI macromolecules contribute to intermolecular reactions that create several hydrogen bonds among adjacent chains. The amine groups of PANI form hydrogen bonds with carbonyl groups of PC which increases through the crosslinking created by γ irradiation. Additional factor that enhances the crosslinking is the presence Co NPs that encourage the charge transfer process due to the shift of charge density between molecular orbital's (MO) of Co NPs and those of PANI which are ligand in character [25]. In our previous work, we got similar trend for the exposed PC/SnS₂ NCP [26]. The exposure of PC-SnS₂ NCP led to the domination of crosslinking that improved the disorder character in the NCP, thus enhanced its resilience and compactness.

The values of crystallite size L were computed applying Scherrer equation:

$$L = (0.89\lambda) / (\Delta W \cos\theta) \quad (1)$$

where ΔW is the peak's halo's width at half maximum intensity. λ is wavelength of the X-rays used (1.54 \AA), and θ is the angle. The crystallite size decreases on raising the γ dose up to 350 kGy (Fig. 3b). The decrease in the L values can be owing to the creation of free radicals, chain scission, and crosslinking of the polymeric NCP chains upon irradiation [24].

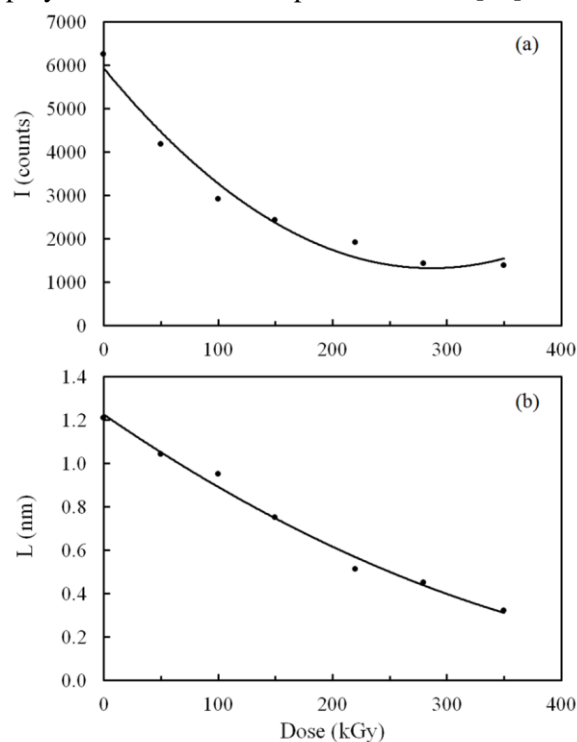


Fig. 3 Dependence of the (a) integral intensity I and (b) crystallite size L on the γ dose.

3.3 FTIR Spectra Analysis

FTIR spectroscopy has been carried out (in the absorbance manner) to explore the structural modifications of the PANI-Co/PC NCP due to γ irradiation. Fig. 4 shows the FTIR absorbance bands for the blank and some exposed NCP samples. The modifications have been predicted considering the variations in the intensity of the peak and any available wave number shift accompanied with the function groups identifying the NCP samples. The modes of chemical bonds in FTIR spectra are characterized according to Noda et al. [27]. The NCP samples exhibit characteristic vibrations at 830 cm^{-1} (Para out-of-plane aromatic CH wag, two adjacent Hs), 1013 cm^{-1} (Para in-plane aromatic CH bend), 1140 cm^{-1} (C-O-C), 1165 cm^{-1} (Carbonate C-O stretch), 1405 cm^{-1} (Para aromatic ring semicircle stretch), 1481 cm^{-1} (stretching mode of vibration for benzenoid unit of PANI) [9], 1605 cm^{-1} (Para aromatic ring quadrant stretch), 1775 cm^{-1} (C=O stretch), 2875 cm^{-1} (CH_3 symmetric stretch), 2980 cm^{-1} (CH_3 asymmetric stretch), 3056 cm^{-1} (aromatic CH stretches) and 3533 cm^{-1} (OH group). It is noticed that some absorption bands in the NCP samples were shifted towards higher wavenumbers than the pure PC polymer. For example, the peaks that should appear at 1160, 1600 and 2970 cm^{-1} actually appeared at 1165, 1605 and 2980 cm^{-1} ,

respectively. This can be owing to the incorporation of the PANI-Co into the PC matrix. All the peaks exhibited variation in intensity with slight shift in wavenumber with the γ dose. The two benzene rings of PC could be recognized in the NCP samples since the appearance of the bands correlated to C-H aromatic stretching (3056 cm^{-1}) and CH_3 symmetric stretch (2875 cm^{-1}) that increased with raising dose to 350 kGy [21]. Additionally, the weak band around 3533 cm^{-1} in the blank NCP demonstrated the limitation of the terminal OH group indicating higher molecular weight [28]. On γ irradiation up to 350 kGy, it decreases meaning the predominance of crosslinking. This can be explained based on that the preparation of PC requires transesterification of diphenyl carbonate with bisphenol A with removal of phenol. Thus the primary composition of OH group will decrease with increasing the chain length [28]. The (C=O) group at 1778 cm^{-1} that appeared in the spectrum of the PC doped with PANI-Co is similar to that in the case of blank PC [29]; possibly owing to the overlapping between bands of PC and PANI-Co. The relatively slight shifts of the bands mentioned above means an exact reaction of PANI-Co with PC concerning the bonding between hydrogen of C=O of PC and amine groups of PANI in the NCP [29]. Moreover, this reaction can also affect the morphology of the NCP.

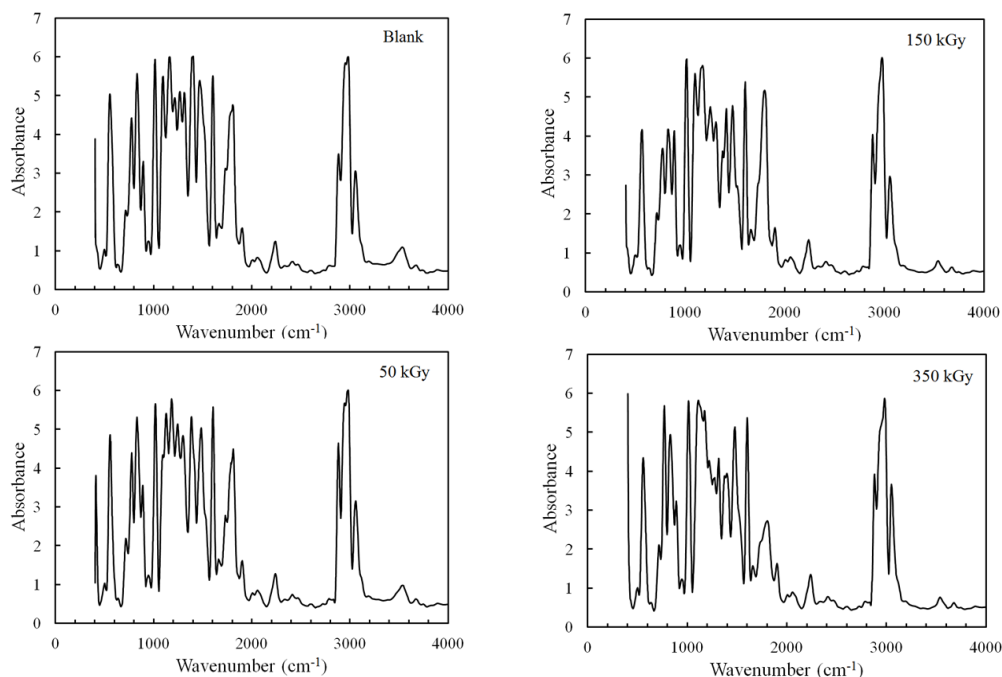


Fig. 4 FTIR absorbance spectra of some exposed PANI-Co/PC NCP samples and the blank.

3.4 Thermogravimetric analysis (TGA)

TGA was conducted on the PANI-Co/PC NCP to determine the modification in thermostability induced in the NCP samples due to γ dose. TGA was carried out up to 600 °C, at 10°C/min. The TGA thermograms of the exposed NCP samples as well as the blank are shown in Fig. 5. It is seen that the NCP samples decompose in one main weight loss stage. TGA curves indicated that the degradation temperature T_o , means the temperature at which the decomposition starts, changes significantly with the γ dose. Fig. 6a shows the dependence of T_o on the γ dose. The onset temperature of decomposition shows a decrease around the 50 kGy exposed sample owing to initial chain scission; then increases on raising the dose to 350 kGy owing to crosslinking. This can be explained based on the fact that the PC in the PANI-Co/PC is thermally stable and degrades at temperatures higher than 300 °C [30]. Additionally, PANI is also thermally stable and its backbone does not degrade even up to 400 °C [30]. Therefore, thermostability of NCP can be affected by the thermostability of both PC and PANI. The γ radiation increases the compact structure of the NCP samples via crosslinking. In addition, the existence of Co NPs affects strongly the thermostability of the NCP.

Activation energy of thermal decomposition (E_a)

E_a is computed to obtain information concerning thermostability of the NCP samples. The technique projected by Horowitz and Metzger [31] has been used to compute E_a for the PVA-PEG/Co₃O₄ NCP. Fig. 6b shows that E_a increases with increasing the γ dose up to 350 kGy. The increase in E_a can be due to the homogenous incorporation of PANI-Co in the PC matrix through crosslinking [21].

3.5 Differential thermal analysis (DTA)

DTA technique was measured up to 400 °C at 10 °C /min. The thermo-grams of the blank PANI-Co/PC and exposed samples showed an endothermic peak owing to the melting temperature (T_m). Fig. 7a shows the DTA thermogram, illustrating T_m . It is seen that the samples were characterized by a range of poorly specific melting temperatures. This is

owing to the degree of freedom of the polymeric chains and the variety in chain length.

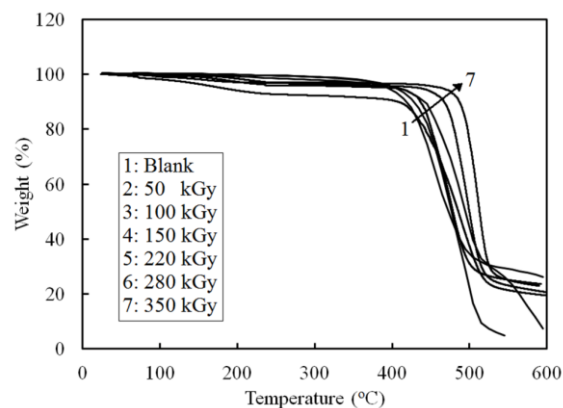


Fig. 5 TGA thermograms of the exposed PANI-Co/PC NCP samples and the blank.

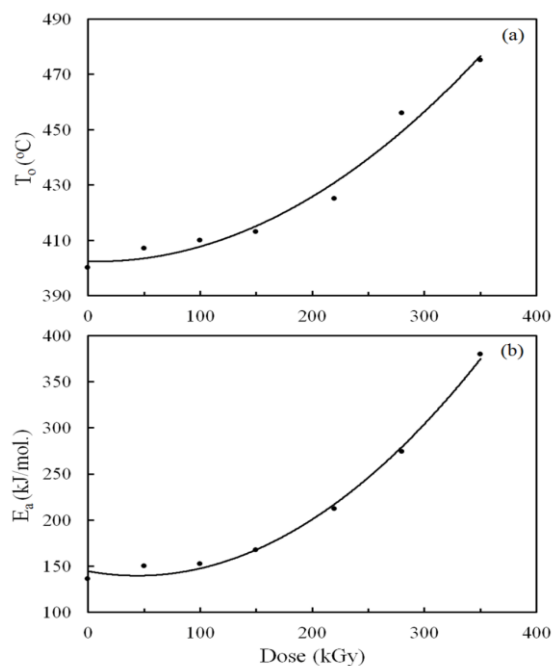


Fig. 6. Dependence of the (a) onset temperature of decomposition T_o and (b) activation energy of thermal decomposition E_a on the γ dose.

The values of T_m were calculated and represented in Fig. 7b versus γ -dose. T_m increased on raising the γ dose to 350 kGy owing to the crosslinking that reduces the ordered phase thus increasing the disorder character. Alternatively, the shorter chains in the NCP have better mobility; consequently allow molecules to be reoriented forming ordered segment. This is similar to the cage effect that includes the free radical re-

combination prior to taking part in reactions that encourage crosslinking [32, 33]. Through crosslinking, the chains can be joined end to end; the increase of the polymeric chain length add also to the shift of T_m toward higher temperatures [23].

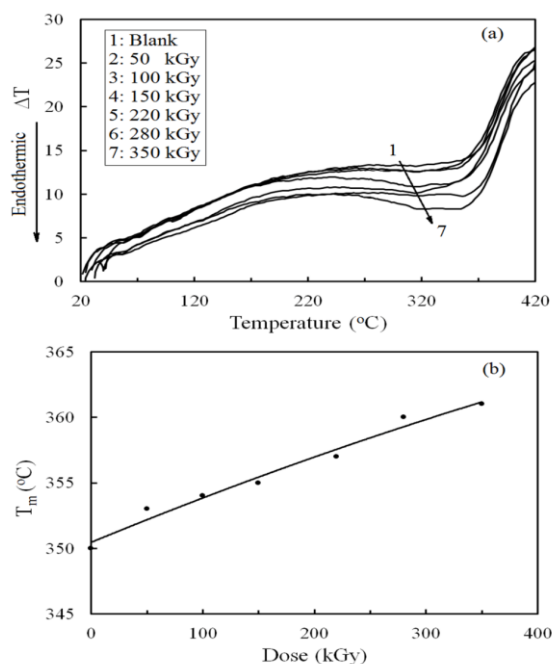


Fig. 7 (a) DTA thermograms of the exposed PANI-Co/PC NCP samples and the blank, (b) Variation of the melting temperature T_m with the γ dose.

4. CONCLUSION

The γ irradiation of PANI-Co/PC NCP causes the domination of crosslinking, modifying its structural and thermal properties. The γ doses up to 350 kGy led to increasing the disorder character in the NCP, thus enhancing its resilience and compactness. This could be drawn from the decrease in both integral intensity and crystallite size. Additionally, the γ radiation encourages the creation of hydrogen bonding between the amine groups of PANI and the C=O of PC through crosslinking. This enhances the thermostability of the NCP samples, with an increase in activation energy of thermal decomposition and melting temperature.

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تأثير أشعة جاما علي الخواص التركيبية و الحرارية للمركب النانوي بولي انيلين - كوبلت/ بوليكرتونات

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3- قسم الفيزياء - كلية العلوم (نبات) جامعة الأزهر - القاهرة

الملخص العربي

تم دراسة التغيرات في الخواص التركيبية والحرارية الناتجة عن التشعيع الجامي للمركب النانوي البولي انيلين- كوبلت/ بوليكرتونات. و تم دراسة التركيب الكيميائي للعينات المحضرة باستخدام الماسح الإلكتروني ومطياف تشتت الطاقة. بالإضافة إلي دراسة حيود الأشعة السينية (XRD) ومطياف الأشعة تحت الحمراء (FTIR) والتحليل الوزني الحراري (TGA) والتحليل الحراري التفاضلي (DTA) وذلك لدراسة تأثير اشعة جاما علي العينات. وقد وجد ان اشعة جاما في مدى الجرعات من 50 إلى 350 كيلو جراي يؤدي إلي زيادة التشابك (crosslinking) والذي يزيد الروابط بين الهيدروجين في البولي انيلين ومجموعات الكربون في البوليكربونات . وقد انعكس هذا علي زيادة درجة حرارة التحلل من 400 إلى 475 م مما يدل علي تحسين الثبات الحراري للعينات المحضرة. وكذلك زيادة درجة حرارة الإنصهار من 350 إلى 361 درجة مئوية مما يشير إلي هيكل تركيبى اكثر صلابة وإحكام.