Effect of Gamma Irradiation on the Thermal and Dyeing Properties of Blends Based on Waste Poly(ethylene terephthalate) Blends

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ABSTRACT: The effect of gamma irradiation on the properties of virgin, waste poly(ethylene terephthalate) (PET), and their blends was characterized by IR spectroscopy, thermogravimetric analysis and differential scanning calorimetry. The dye affinity for disperse dyes of the different PET polymers, before and after gamma irradiation was also studied. The results showed that the thermal stability of virgin, waste PET, and their blends was improved after gamma irradiation. The dye affinity for the disperse dye in terms of color intensity was improved after the exposure to gamma irradiation. The dye ability with disperse dye, in terms of color intensity (ΔE^*), for virgin/waste PET (80/20%) blend, as an example, was improved by $\sim$ 53 and 98% after the exposure to doses 30 and 50 kGy of gamma irradiation, respectively. In conclusion, the modification of waste PET or its blends with virgin PET by gamma irradiation may provide a practical method for the recycling to obtain useful products. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 117: 3482–3490, 2010

Key words: poly(ethylene terephthalate); gamma radiation; recycling; thermal stability; dyeing properties

INTRODUCTION

Plastic products based on polymeric material are used in all aspects of life and manufactured for different purposes covering different items from bottles for mineral water and soft drinks to more sophisticated parts in copying machines. Because of the nonbiodegradability of most synthetic polymers, wastes have to be disposed off in an environmentally practical method.

Management of solid waste is an important problem, which is becoming progressively worse as a byproduct of continuing economic growth and development. Development of technologies for reducing polymeric waste, which are acceptable from the environmental standpoint, and which are cost-effective, has proven to be a difficult challenge because of complexities inherent in the reuse of polymers. The recycling of waste with virgin poly(ethylene terephthalate) (PET) is one of those acceptable methods. The processing of virgin and recycled PET in a twin-screw extruder was studied.[1] Lower melt viscosity and molecular weight, along with higher carboxylic and end group concentration, were observed for recycled PET depending on PET purity. The miscibility, crystallization, and melting behavior in blends of virgin and recycled PET with poly(ether imide) using differential scanning calorimetry (DSC) was studied.[2] The blends exhibit single, composition-dependent T_g's over the entire composition range and a negative value of the Flory interaction parameter, indicating miscibility of the amorphous polymers. The recycling of poly(ethy1ene terephthalate) from the recovery of a blow-molded bottle was investigated.[3] The crystalline behavior in terms of glass transition temperature (T_g), crystallization temperature (T_c), melting temperature (T_m), and its dynamic crystallization kinetics were investigated. These crystalline behaviors explain the better mechanical properties of the R-PET. Thermal cycles of the processes of the R-PET and its blending specimens with engineering PET (E-PET) show the importance of the thermal treatment of the plastic PET in the improvements of mechanical strength and increased crystallinity.

Because of the ability of ionizing radiation to alter the structure and properties of polymeric materials, and the fact that it is applicable to all types of polymers, irradiation holds promise for affecting the polymer waste problem. High-energy radiation can induce both chain scission and/or

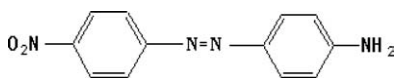*Correspondence to:* A. W. M. El-Naggar (ab_nagga@ yahoo.com).

Figure 1 Chemical structure of Samaron disperse dye.

crosslinking.[4,5] Waste PET bottles were converted to either acrylate or methacrylate-terminated PET oligomers by a two-stage process, first glycolysis with diethylene glycol (DEG) followed by reaction with either acryloyl chloride or methacryloyl chloride.[6] The oligomers are viscous oils that could be blended with commercial radiation-curable oligomers, cast into films and cured by UV irradiation. The films had Young's moduli in the range of 1–3 GPa and tensile strengths of 4–40 MPa. Low-molecular-weight constituents of PET, irradiated with ^{60}Co gamma rays at 25 and 50 kGy, were analyzed by HPLC–MS with atmospheric-pressure chemical ionization.[7] Consistent with earlier results, the concentrations of the major compounds that are present in the nonirradiated PET do not change perceptibly. However, it was found a small but significant increase in terephthalic acid ethylester, from less than 1 mg/kg in the nonirradiated control to about 2 mg/kg after 50 kGy, which has not been described before. These results raise the possibility of converting waste PET into high value-added radiation-curable oligomers for coatings applications. The present work is undertaken to study the effect of gamma irradiation on the structural, thermal, and dyeing properties of virgin and waste PET polymers as well as their blends as a method of recycling waste PET.

MATERIALS AND METHODS

Materials

Virgin PET used throughout this work was purchased from Aldrich Chemical Company (USA) in the form of pellets with a molecular weight (M_w) of 58,000 and inherent viscosity of 0.59. Waste PET used in this work was based on clear bottles used for mineral drinking water manufactured in 2009. Most of drinking PET bottles having M_n of 25 kg/mL, M_w of 60 kg/mol, and density of 1.15 g/cm^3. The bottles were first washed, dried, and chopped in particles of $\sim$ 3 mm main size. The waste PET particles were washed again with acetone and dried in vacuum oven. Samaron$^{®}$ (disperse dye) was kindly supplied by Hoechst, Germany. It is usually used for dyeing polyester fibers and applied at elevated temperatures with the aid of carriers because it is insoluble in water. Generally, the basic structure of disperse dyes and in particular the Samaron is based on azo compounds[8] as shown in Figure 1.

Preparation of virgin/waste PET blends

A Brabender plasticorder extruder (PL 2100 Mixer) operating at a constant rotating speed of 60 rpm and at 260°C was used for the preparation of virgin and waste PET polymers and their blends in the form of sheets. The sheets from the plasticorder extruder were then molded compressed under hot press to form square films (5 cm × 5 cm) and thickness of $\sim$ 0.2 mm. The blends were prepared to contain 20, 50, and 80 wt % of waste PET.

Gamma irradiation

All the polymers and blends were exposed to gamma irradiation prior to melt extrusion. Irradiation to the required doses was carried out in the Co-60 gamma cell (made in India), National Center for Radiation Research and Technology, Cairo, Egypt.

FTIR spectroscopic analysis

The IR spectra of the different polymers were performed over the range 500–4000 cm^{-1}. The IR spectra were acquired by transmission FTIR with a Mattson 5000 FT-IR spectrometer. The spectra were taken with resolution of 4 cm^{-1} and were averaged over 16 scans.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) study was carried out on Shimadzu-30 (TGA-30) at a heating rate of 10°C/min under nitrogen atmosphere from room temperature to 600°C. The initial TGA data were used to determine the different kinetic parameters of thermal decomposition reaction. The derivative of the TGA thermograms (DTGA) or the rate of reaction (dw/dt) was determined by taking the weight loss every 2 min. The rate of reaction dw/dt was plotted against temperature from which the different kinetic parameters in terms of T_{onset}, T_{endset}, and T_{peck} temperatures can be determined.

Differential scanning calorimetry

DSC was carried out on Shimadzu DSC-50 calorimetry at a heating rate of 10°C/min under a flowing nitrogen gas at a rate of 20 mL/min. The X_c was determined according the commonly known relationship:

$$X_c(\%) = (\Delta H_m / \Delta H_o) \times 100$$

where ΔH_m is the melting enthalpy for the polymer, calculated from the area under the peak of the DSC heating curve and ΔH_o is the melting enthalpy for
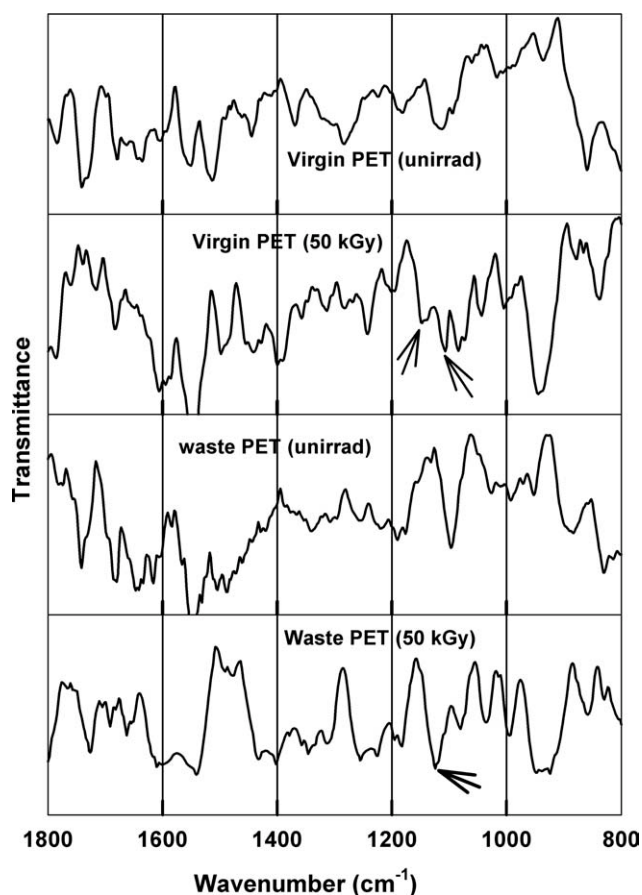
Figure 2 IR spectra for virgin and waste PET polymers before and after gamma irradiation to a dose of 50 kGy within the wavenumber range 1800–800 cm^{-1}.

an entirely crystalline polymer. The reported ΔH_o for PET is 140.1 J/g.[9]

Dyeing with disperse dyes

The disperse dye concentration (5 wt % based on material weight) was stirred with 10–20 times of its weight of water at 50–60°C and stand for 10 min. The PET samples were introduced in the disperse dye bath, the temperature was raised to boiling, and the dyeing is continued at that temperature for 1 h.

Color intensity measurements

The color intensity of dyed samples was measured by a microcolor meter equipped with a data station made by Brano Lange, GmbH, Konigsweg 10, D-1000 Berlin (Germany). The L*, a*, b* system used in this method is based on the Commission International de Éclair (CIE), color triangle, (X, Y, and Z). The L*, a*, b* color components used in measurements represent the dark-white axis, the green-red

axis, and the blue-yellow axis, respectively. The color intensity (ΔE^*) of the dyed samples was determined according to Judd-Hunter standardization, 1976 as follows:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

RESULTS AND DISCUSSION

IR spectroscopic analysis

In this work, the chemical and physical properties of virgin and waste PET as well as their blends at different ratios was modified by the exposure to gamma irradiation before melt extrusion. Ionizing radiation can induce in polyesters excitations or radical formations that can evolve in charge transfer, chain break, disproponation, β-scission, and other ordinary ion and radical reactions. Increasing formation of radicals with the dose (time) can allow crosslink. Oxygen stored in the polymer is a well-known radical scavenger; it quickly reacts with formed radicals producing peroxides that can evolve into stable
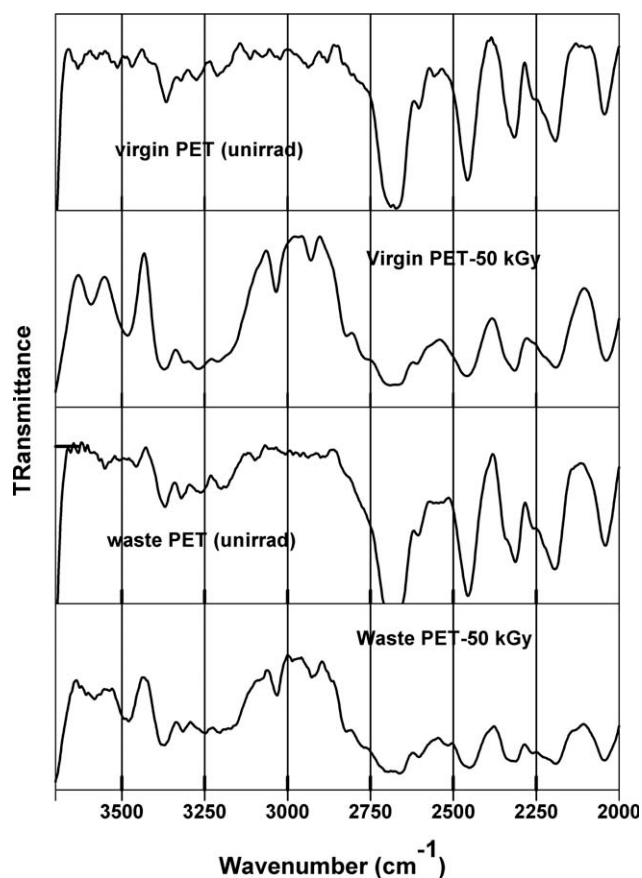

Figure 3 IR spectra for virgin and waste PET polymers before and after gamma irradiation to a dose of 50 kGy within the wavenumber range 3750–2000 cm^{-1}.
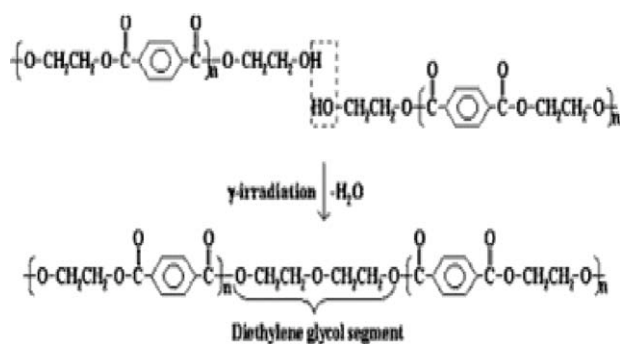
Figure 4 Proposed mechanism for the formation of DEG in PET polymer chains at the low doses, 5 and 10 kGy of gamma irradiation. (This scheme was reprinted from Ref. 16).

products like alcohols, aldehydes, ketones, carboxyl acids, and esters.[10,11]

IR spectroscopic analysis was used to illustrate the changes in the structure because of gamma irradiation. Figures 2 and 3 show the IR spectra of the virgin and waste PET polymers over the entire range of wavelengths (in two figures). The IR spectra of unirradiated virgin PET showed an absorption band at 2950 cm^{-1}, which is due to the C—H stretching as almost all organic compounds. In addition, an absorption band can be seen at 1730 cm^{-1}, which is due to the C=O stretching of the ester groups. The absorption band at 2850 cm^{-1} and the series of
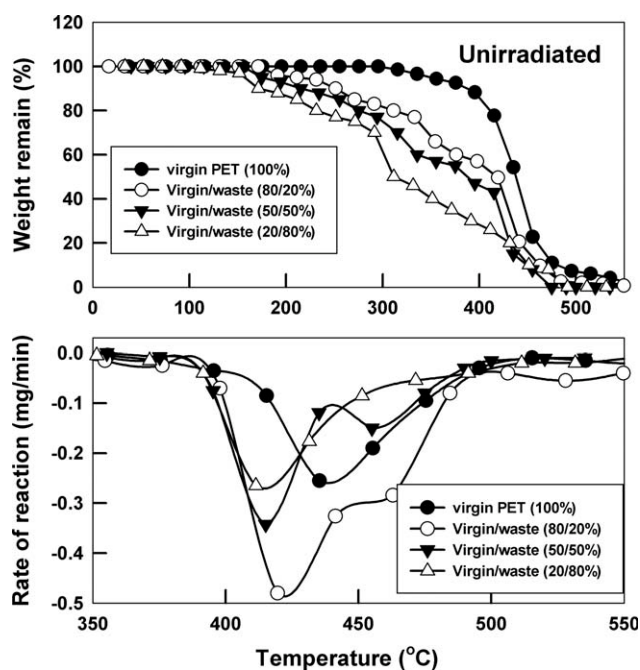

Figure 5 TGA thermograms and the corresponding rate of thermal decomposition reaction curves for unirradiated virgin PET and virgin/waste PET blends of different ratios.
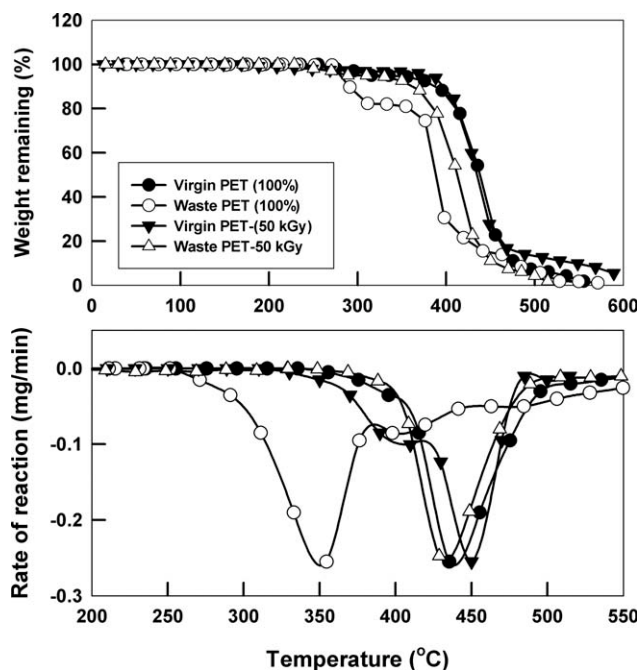

Figure 6 TGA thermograms and the corresponding rate of thermal decomposition reaction curves for virgin and waste PET polymers before and after exposed to a dose of 50 kGy of gamma irradiation.

bands at 1620–1420 cm^{-1} indicate the presence of aromatic C—H and the benzene rings, respectively. The intensity of C=O stretching is seen to decrease after gamma irradiation of waste and appeared as broad peak in the case of virgin PET indicating that the crosslinking event is the major process in accordance with a study on the photodegradation and photocrosslinking of thin films of benzophenone/polystyrene blends.[12]

It was reported that small amounts of DEG are present in all commercial PET as an unavoidable byproduct of synthesis.[13] The DEG contents before and after gamma irradiation of PET polymers within the dose range 5–200 kGy was investigated by NMR.[14] The most obvious features of the spectrum of unirradiated PET film are the singlet at δ8.1 arising from the four equivalent methylene protons of the phenyl repeat unit and the singlet at δ4.8 arising from the four equivalent methylene protons of ethylene glycol unit in the spectrum. Small triplets at δ4.1 and δ4.6 indicate the presence of DEG unit in polymer chain. The proposed mechanism of DEG formation was based on the increased DEG values at low dose and the reported results.[15] The ethylene glycol end groups in each polymer chains reacted with each other and DEG was formed at the low dose, 5 and 10 kGy (as shown in Fig. 4). However, it was observed that the DEG content was decreased from 2.21 mol % at an irradiation dose of 5 kGy to 2.14 mol % at 60 kGy. It was assumed that increased
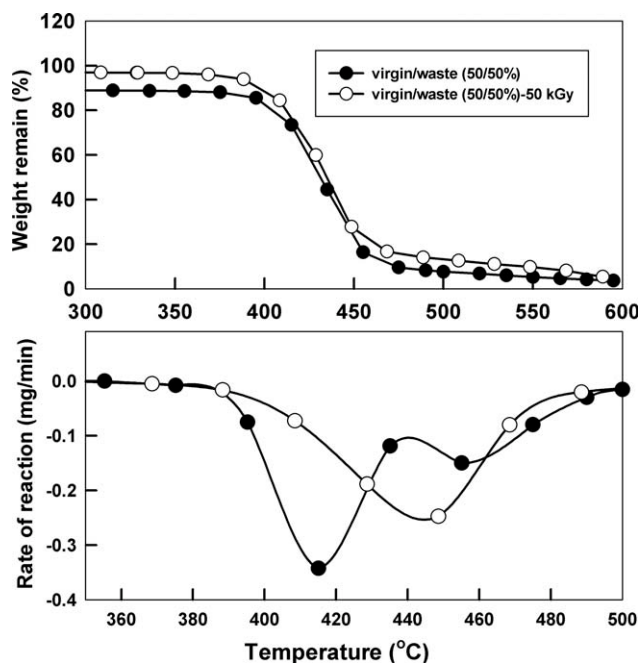
Figure 7 TGA thermograms and the corresponding rate of thermal decomposition reaction curves for virgin/waste PET (50/50%) blends before and after exposed to a dose of 50 kGy of gamma irradiation.

molecular weights at low dose could be caused by the formation of DEG in the polymer chains. In addition, it was found that the molecular weights M_n, M_w, M_z, M_w/M_n, intrinsic viscosity and carboxyl end groups at low dose showed no or only small changes. In the present work, on the other hand, the formation of DEG because of gamma irradiation of virgin or waste PET at a dose of 50 kGy can be confirmed by appearance of an absorption-stretching band of the ether linkage —O— in the IR spectra at 1050–1260 cm^{-1} as shown in Figure 2.

Thermogravimetric analysis

TGA was used to investigate the effect of waste PET addition and gamma irradiation on the thermal sta-

bility of virgin/waste PET blends. Figure 5 shows the initial TGA thermograms for unirradiated virgin PET and its blends with different ratios of waste PET. Based on the percentage weight remaining, it can be seen that the thermal stability of virgin PET decreases with increasing the heating temperature and ratio of waste PET. This may be explained based on waste PET is subjected to cycles of thermal melting and extrusion during the industrial processes. Figures 6 and 7 show the initial TGA thermograms of virgin and waste PET polymers before and after they had been exposed to a dose 50 kGy of gamma irradiation and TGA thermograms of virgin/waste PET (50/50%) blends before and after they had been exposed to a dose 50 kGy of gamma irradiation (as an example), respectively. It can be seen that the major thermal decomposition of unirradiated or gamma irradiated PET polymers occurs within the temperature range 300–400°C. In this regard, the unirradiated virgin PET displayed higher thermal stability, with lower weight loss, than unirradiated waste PET. However, gamma irradiated virgin and waste PET polymers showed higher thermal stability than the unirradiated ones. Based on Figure 7, the difference in weight remaining (%) is not clear; however, gamma irradiated blends displayed slight higher thermal stability than the unirradiated ones.

To have a better understanding of the thermal stability, the derivatives of the above TGA thermograms (rate of thermal decomposition reaction, dw/dt) were plotted vs. temperature as shown in Figures 5–7. The different kinetic parameters were presented in Table I. It can be seen that the rate of reaction curves displayed similar trends; however, the different kinetic parameters (T_{onset}, T_{endset}, and T_{peak} temperatures) differ from one material to another. It can be seen that the main thermal decomposition reaction for unirradiated virgin and waste PET occurs within temperature range of $\sim$ 100°C (difference between T_{onset} and T_{endset}). However, the T_{onset}, for unirradiated virgin PET is much higher than that for unirradiated waste PET. The difference

TABLE I
Kinetic Parameters of the Thermal Decomposition Reaction of Virgin and Waste PET Polymers and Their Blends Before and After Gamma Irradiation to the 50 kGy Dose

| PET polymers and blends	T_{onset} (°C)	T_{endset} (°C)	T_{peak} (°C)	Activation energy, E^* (kJ/mol k)		
				Left step	Right step	E^* average
Unirradiated virgin PET	405.2	502.2	438.1	−155.98	158.14	157.06
Unirradiated waste PET	290.8	383.5	349.2	−101.05	104.25	102.65
Gamma irradiated virgin PET	354.5	486.4	449.5	−156.76	269.38	213.07
Gamma irradiated waste PET	401.4	487.3	432.6	−112.30	133.23	122.765
Unirradiated virgin/waste PET (80/20%)	392.6	491.0	421.7	−152.63	110.92	131.775
Unirradiated virgin/waste PET (50/50%)	383.4	442.9	415.3	−118.73	109.54	114.135
Unirradiated virgin/waste PET (20/80%)	386.7	456.6	414.3	−72.39	90.10	81.245
Gamma irradiated virgin/waste (50/50%)	383.6	485.2	444.4	−121.24	141.71	131.475
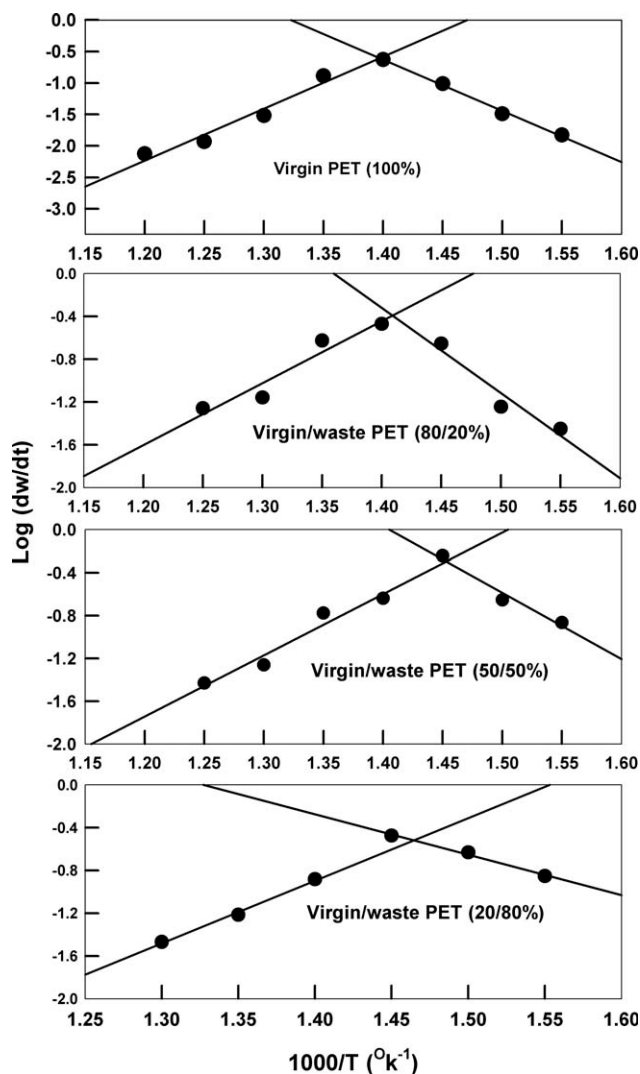
Figure 8 Determination of activation energy plots for unirradiated virgin PET and its blends with different ratios of waste PET.

in T_{onset} and T_{endset} was found to increase after gamma irradiation. On the other hand, it can be seen that the T_{onset} decreases with increasing the ratio of waste PET in the unirradiated blends.

According to T_{peak}, the thermal stability of the different materials may be arranged as follows: Gamma irradiated virgin PET (450.0°C) > gamma irradiated virgin/waste PET, 50/50% (444.3°C) > unirradiated virgin PET (438.1°C) > gamma irradiated waste PET (432.6°C) > unirradiated virgin/waste PET, 80/20% (421.7°C) > unirradiated virgin/waste PET, 50/50% (415.3°C) > unirradiated virgin/waste PET, 20/80% (411.4°C) > unirradiated waste PET (349.2°C). According to T_{onset}, the thermal stability of the different materials may be arranged as follows: Unirradiated virgin PET (405.2°C) > Gamma irradiated waste PET (401.4°C) > Unirradiated virgin/waste PET, 80/20% (392.6°C) > Unirradiated virgin/waste PET, 20/80% (386.7°C) > Gamma irradiated virgin/
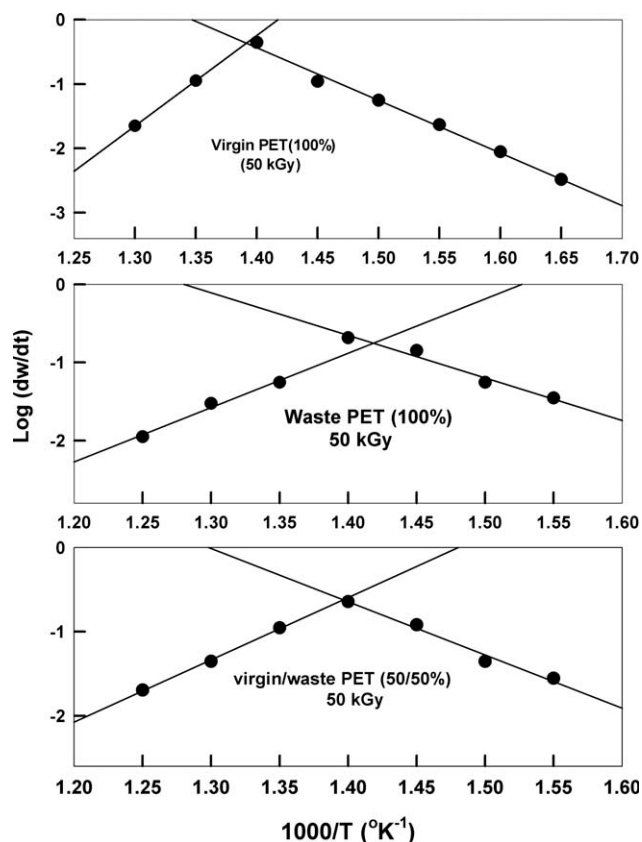

Figure 9 Determination of activation energy plots for virgin, waste PET polymers and virgin/waste PET (50/50%) after gamma irradiation to a dose of 50 kGy.
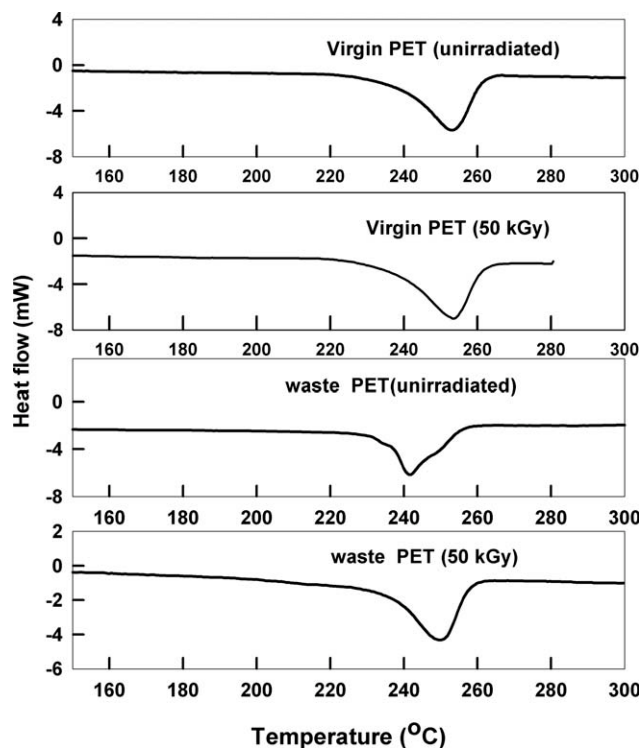

Figure 10 DSC thermograms for virgin and waste PET polymers before and after gamma irradiation with a dose of 50 kGy.
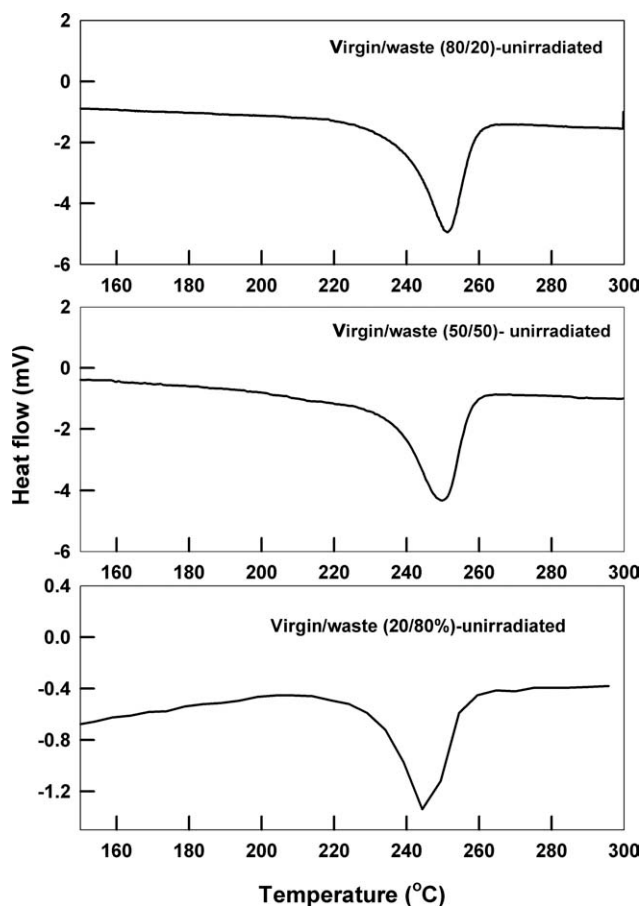
Figure 11 DSC thermograms for unirradiated virgin/waste PET blends of different ratios.

waste PET, 50/50% (383.6°C) ≥ Unirradiated virgin/waste PET, 50/50% (383.4°C) > Gamma irradiated virgin PET (354.5°C) > Unirradiated waste PET (290.8°C).

The thermal stability was further confirmed by determining the kinetic parameters of the thermal decomposition reaction. A method based on the rate of reaction proposed by Anderson and Freeman was utilized, in which the quantities $\Delta \log \dot{w}$ and $\Delta \log dw/dt$ corresponding to a constant small difference of $\Delta(1/T)$ over the entire course of the initial TGA curve were first determined.[17] The Anderson–Freeman equation, which relates these quantities is:

$$\Delta \, \mathrm{Log}(dw/dt) = n\Delta \log \dot{w} - (E^{*}/2.303 \, R)\Delta 1/T$$

where dw/dt is the rate of reaction (mg/min), $\dot{w}$ is the reactant mass (mg), R is the gas constant (J/mol K), E^{*} is the activation energy (J/mol) and "n" is the order of the reaction. When $\Delta \log (dw/dt)$ is plotted against $\Delta \log \dot{w}$, it gives a straight line of slope "n" and the intercept gives the activation energy E^{*}.

When $\Delta \log (dw/dt)$ was plotted against $\Delta \log \dot{w}$ for the different PET polymers and blends ether before

or after gamma irradiation over the temperature range 300–500°C, the data points did not fall on a straight line. Therefore, on the basis of the Anderson–Freeman equation, the thermal decomposition reaction within this range of temperatures does not depend on the residual mass but it depends on temperature and follows zero-order reaction. In thin case, log (dw/dt) is plotted against $1/T$ and the slope is equal to $E^{*}/2.303R$, from which the activation energy can be calculated. Figures 8 and 9 show the determination of activation energy plots for different pure PET polymers and blends before and after gamma irradiation. The activation energy for all the polymers and blends of PET is summarized in Table 1. Based on the results in Table 1 and Figures 8 and 9, it can be seen that the activation energy of the right side of the derivatives of thermal decomposition reaction (DTGA) is always greater than those of the left side. The activation energy values indicate clearly that gamma irradiation improves the thermal stability.

The improvement in thermal stability can be explained based on the various reactions took place
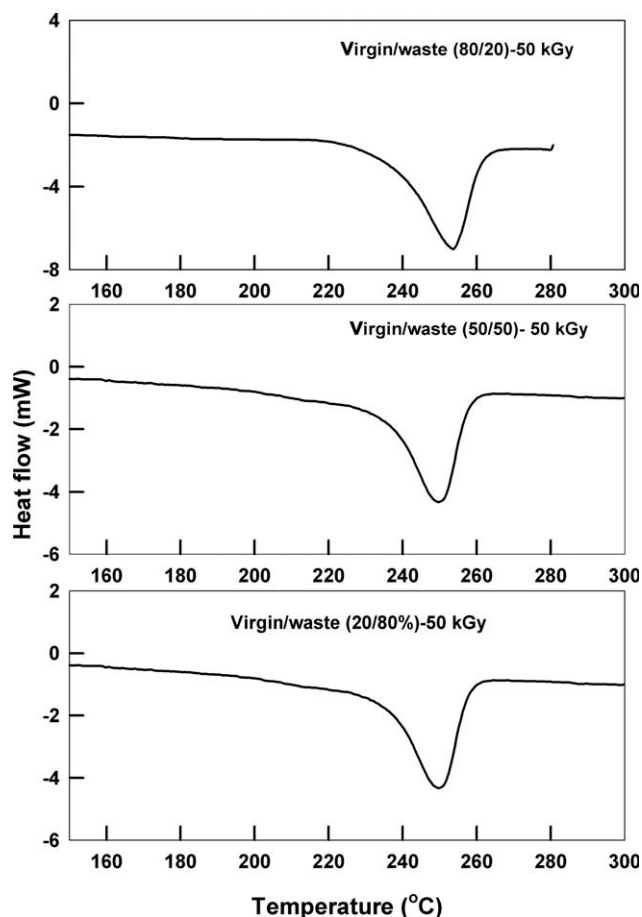

Figure 12 DSC thermograms for gamma irradiated virgin/waste PET blends of different at a dose of 50 kGy.

TABLE II
Melting Temperature, Enthalpy of Crystallization, and Crystalline Fraction for Virgin Waste PET, and Their Blends at Different Ratios Before and After Gamma Irradiation to a Dose of 50 kGy

PET polymers and blends	T_m (°C)	ΔH_c (J/g)	X_c (%)
Virgin PET (100%)–unirradiated	252.0	107.3	76.6
Virgin PET (100%)–50 kGy	253.8	103.6.	73.2
Waste PET (100%)–unirradiated	241.9	109.0	77.8
Waste PET (100%)–50 kGy	249.9	105.5	75.3
virgin/waste PET (80/20)–unirradiated	251.4	108.3	77.3
virgin/waste PET (80/20)–50 kGy	253.4	106.2	75.8
virgin/waste PET (50/50)–unirradiated	249.4	109.7	78.3
virgin/waste PET (50/50)–50 kGy	251.3	104.4	74.5
virgin/waste PET (20/80)–unirradiated	244.3	112.5	79.3
virgin/waste PET (20/80)–50 kGy	249.4	102.6	73.2

in PET during gamma irradiation, such as crosslinking. It has been reported that aliphatic polyesters have the tendency to crosslink upon gamma irradiation, whereas PET crosslinks with low efficiency. In this regard, the reported crosslinking efficiencies for PET $G(X)$ are between 0.035–0.14 and the chain scission $G(S)$ between 0.7–0.17.[18] Thus, it is expected that gamma irradiation of PET would results in oxidative degradation to some extents than crosslinking leading to the formation of carbonyl and carboxyl groups. The dissociation energies of the covalent bonds C—H, C—C, C=O, C—O, and O—H were reported to be 414, 347, 741, 351, and 464 kJ/mol, respectively.[19] Therefore, the additional formed carbonyl and carboxyl groups will eventually possess higher thermal stability.

Differential scanning calorimetry

DSC technique was used to investigate the effect of waste PET addition and gamma irradiation on the melting and crystallinity of virgin/waste blends of different ratios. Figure 10 shows the DSC curves for virgin and waste PET polymers before and after gamma irradiation at a dose of 50 kGy. It can be seen that the DSC scan of unirradiated virgin PET showed a wide endothermic peak with a maximum at 252.0°C, which due the crystalline melting temperature (T_m). This T_m was slightly increased to 253.8°C after gamma irradiation. For unirradiated waste PET, the DSC curve showed a wide endothermic peak with a maximum at 241.9°C, whereas the T_m was increased to 249.9°C after gamma irradiation.

Figures 11 and 12 show the DSC curves for virgin/waste based PET blends of different ratios before gamma irradiation at a dose of 50 kGy. It is clear that the DSC curves and the change in the T_m for the blends are similar to those observed for the

individual polymers. However, the blending of waste PET with virgin PET causes a slight increase in the T_m and a decrease in the crystallinity (X_c) as shown in Table II. These findings are in accordance with previous work on the effect of electron beam irradiation on the crosslinking of polymer blends based on LDPE and PET.[19,20]

Dyeing properties of PET polymers and blends

The importance of disperse dyes has increased largely with the appearance of the hydrophobic synthetic fibers, in which they are used in dyeing polyesters. The absorption of such dyestuffs depends in the first place on the use of carries at elevated temperatures.

Figure 13 shows the color intensity of films of virgin/waste PET blends of different ratios exposed to two doses of gamma irradiation and dyed with the disperse dye Samaron. It can be seen that the increase of waste PET ratio in the unirradiated blends results in a decrease in the color intensity. However, at any blend ratio, the increase of irradiation dose was accompanied with an increase of color intensity. In this regard, the increase of irradiation dose from 30 to 50 kGy results in an increase of color intensity by $\sim$ 5, 29, and 42% for the blends containing 20, 50, and 80% of waste PET, respectively. The mechanism by which the dyeing with the disperse dyes is not strictly explained; however, there is an acceptable mechanism, in which hydrogen bonding takes place between the primary amino or amine on the dye and the carbonyl groups of PET.[8] The exposure of PET to gamma irradiation
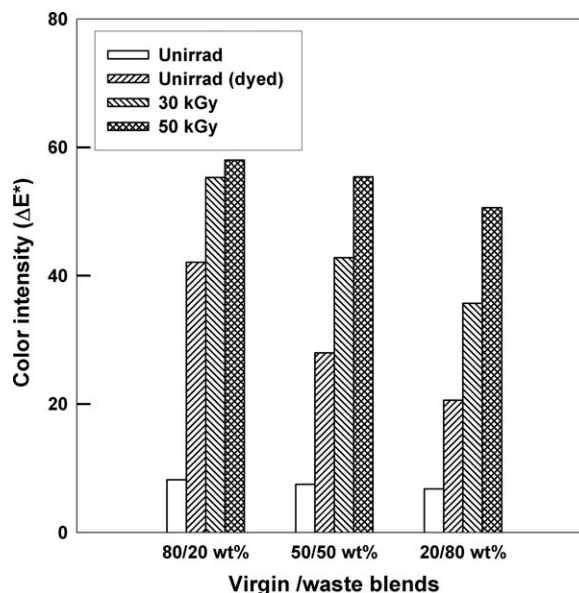

Figure 13 Color intensity (ΔE^*) of films of virgin/waste PET blends exposed to different doses of gamma irradiation after dyeing with disperse dye.

will eventually leads to the formation of peroxy and free radicals.[21,22] These radicals would decompose to form oxygenated groups capable to combine with the amine or amino groups.

CONCLUSIONS

Ionizing radiation can induce in polymers excitations or radical formations that can result in chain scission or crosslinking. These reactions are important in modifying the chemical and physical properties of polymers. As a method of recycling, in the present work, the thermal and dyeing properties of virgin/ waste PET polymer blends were improved by exposing to a dose of 50 kGy of gamma irradiation prior to extrusion. The results indicate improvements in these properties after gamma irradiation, which may be considered a method of recycling instead the conventional chemical methods, such as depolymerization to small fragments.

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