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Gamma-Radiation Effect on Mechanical Properties of Pp/Epdm Polymeric Blends

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Abstract
Thermoplastic elastomers (TPEs) are provided with many properties of elastomeric or rubbery materials, but can be processed with the thermoplastic processing methods; they are one of the fastest growing polymers materials which combine the elastic and mechanical properties of crosslinked rubbers with the melt processability of thermoplastics. In addition, TPEs find a lot of applications in automotive, buildings and construction, wires and cables, etc.; besides their ability to reuse and recycle the production scrap and waste. Polypropylene (Pp) is a commodity, with high melting point, high chemical resistance, low density, with a balance between physical and mechanical properties and easy processing at low cost. Nevertheless, Pp shows limitations for some special applications, in terms of impact resistance; in order to minimize this characteristic, an impact modifier, as Ethylene Propylene Diene Terpolymer (Epdm) can be used. Due to different polarity and structure between the thermoplastic and elastomeric phases, most thermoplastic elastomers are incompatible. Poor interfacial adhesion and high interfacial tension between rubber and thermoplastic phases are main reasons for incompatibility of these systems; in case of Pp and Epdm, there is a relatively low interfacial tension (driving force for the transformation of a co-continuous structure into a dispersed one) between Pp and Epdm (approximately 0.3 mN/m), reducing the rate of breakup and facilitating the formation of a continuous structure. Pp/Epdm blends are completely recyclable and reusable, safe to the environment, thus improving the overall profitability of the process. Besides a higher impact resistance shown by Pp/Epdm blends, it is possible that Epdm incorporation in Pp causes a reduction in tensile resistance. In order to eliminate this interference and assuring a more comprehensive application in automotive, buildings and construction, Pp/Epdm blends, rubber contents ranging from zero to 30% (thirty percent), were subjected to gamma-irradiation, within doses from 5 to 20 kGy. Isotactic Pp was considered for this work. Characterization assessments included mechanical, thermal and rheological essays.

1. Introduction

Due to a very attractive combination of physical and mechanical properties, low density and low cost, Polypropylene (PP) is one of the most versatile commodity polymers, with continuously increasing applications. The main disadvantage of this polymer is its poor low impact strength. In order to overcome all these limitations the addition of ethylene propylene diene monomer (EPDM) to PP is depicted as a successful tool to be implemented (R. Zhao and G. Dai, 2002; P. T. T. Minh et al, 1998; P. Ezzati et al, 2008;

A. R. Jalivand et al, 2007).

However, this type of thermoplastic elastomer olefinic (TPO) has an unstable morphology because of coalescence of dispersed rubber particles and low compatibility between rubber phase and thermoplastic matrix. The use of thermoplastic elastomers (TPEs), for example, those ones based in EPDM and PP has been increasingly raised last years. This special class of TPE fits thermoplastic processing characteristics to elastomers conventional physical properties at working temperature (L. Torre and J. M. Kenny, 2000). In addition, TPEs show an important advantage related to the reuse and recycling of production scrap, as well wastes, in favor of environmental protection (N. R. Legge et al, 1987; S. K. De and A. K. Bhowmick, 1990).

The incorporation of an elastomer to PP, besides improving impact resistance can lower tensile strength, in some cases. stabilize the morphology of the blend it was applied gamma-irradiation, low doses (5 to 20 kGy) at a dosage rate of 5 $kGy.h^{-1}$. It is generally accepted that ethylene-propylene elastomers show significant resistance to the action of ionizing radiation (I. Banick and A. K. Bhowmick, 2000; J. Bik et al, 2003; N. Dély et al, 2005). Oxidative degradation can never be entirely eliminated because molecular oxygen is present in the majority of environmental conditions. Technological application of this class of polymers in the radiation processing field must take into account the consequences of oxygenated products accumulated during the start step of irradiation on material stability (T. Zaharescu et al, 2010). In summary, the addition of EPDM to PP, within a range up to 30%, usually leads to a good balance of properties (E. Martuscelli, 1995).

Our study was focused on assessments via ATR-FTIR related to blends structure, in function of their compatibility and doses irradiation. Izod impact strength indicated blends impact performance and melt index assessments predicted the influence of radiation on flow properties. Crystallinity and degradation temperature were obtained from thermal analyses, via DSC and TGA, respectively. Finally, by using texturometer and dynamometer it was possible to determine tensile strength and elongation at break, in order to characterize mechanical behavior for all blends herein studied.

2. Experimental Section

2.1. Materials

Polypropylene (Pp): PP-440K, Quattor/Braskem, 3.5 g/10 Minutes, M. F. I.

Ethylene Propylene Diene Monomer (Epdm): Keltan 5470, Lanxess, 55 Mooney Viscosity at 125°C, 70% Ethylene Content, 25% Propylene Content, 4.6% ENB Content, 39% Crystallinity.

2.2. Samples Preparation

Blends composition are presented in Table I. At first, samples were compounded on a 3.1 L/D, 19/33 compression ratio twin-screw extruder (HAAKE Rheomex 332p), temperatures 175-200°C, 60 rpm. Admixtures prepared were air irradiated in a Co60 source, at room temperature, 5 kGyh-1 radiation rate, at 5, 10, 15 and 20 kGy

Table 1. *Samples composition, parts by weight.*

Components					
PP	100	90	80	70	-----
EPDM	-----	10	20	30	100

2.3. Characterization

2.3.1. Mechanical Essays

TA-Hdi (Stable Micro Systems Texture Analyser) texturometer, 5 kg load cell, 0.5 m/s deformation rate, 2 mm/seg speed and an EMIC model DL 300 universal essay machine, 20 kN load cell, in accordance with ASTM D 638-08. Tensile tests were performed under ambient conditions.

2.3.2. Thermalbehavior

DSC Mettler Toledo apparatus, according to ASTM D3418-08 – Standard Test method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry, by using 5 – 9 mg of sample, within a 25 to 300°C program, at a 10°C/min, in a nitrogen flow of 50 ml/min.

2.3.3. Termogravimetric Analyses (TGA)

It provides complimentary and supplementary characterization information to DSC, by measuring the amount and rate (velocity) of change in the mass of a sample as a function of temperature or time in a controlled atmosphere. Measurements are used primarily to determine the thermal and/or oxidative stabilities of materials as well as their compositional properties. The technique can analyze materials that exhibit either mass loss or gain due to decomposition, oxidation or loss of volatiles (such as moisture). TGA in pellets samples were performed using a DSC Mettler Toledo apparatus, according to ASTM E1641-07 – Standard Test method for Decomposition Kinetics by Thermogravimetry, by using 5 – 9 mg of sample, within a 25 to 600°C program, at a 10°C/min, in a nitrogen flow of 50 ml/min.

2.3.4. Melt Flow Index

Ceast apparatus, *modular line,* 230°C, 2.16 kg load, 240 seconds pre-heating time, according to ASTM D1238-13.

2.3.5. Izod Impact Strength

International Equipment, 21-68 joules capacity, 150 degree release angle of pendulum, range of 4 scales, 0.02 joule minimum resolution; in accordance with ASTM D-256-10; this technique allows the valuation of rupture strength of plastic materials when subjected to impact by bending or abrupt forces.

2.3.6. Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (ATR-FTIR)

Thermo-Scientific spectrophotometer, Nicolet 6700-FT-IR model. Setup collection sample was adjusted for 64 scans, within a 3,500 to 500 cm^{-1} range.

3. Results and Discussion

3.1. Mechanical Essays

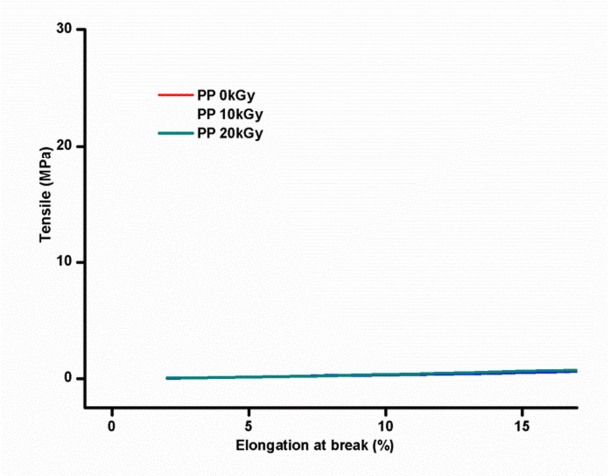

Fig. 1. Mechanical behavior of PP, gamma-irradiated at 0, 10 and 20 kGy.

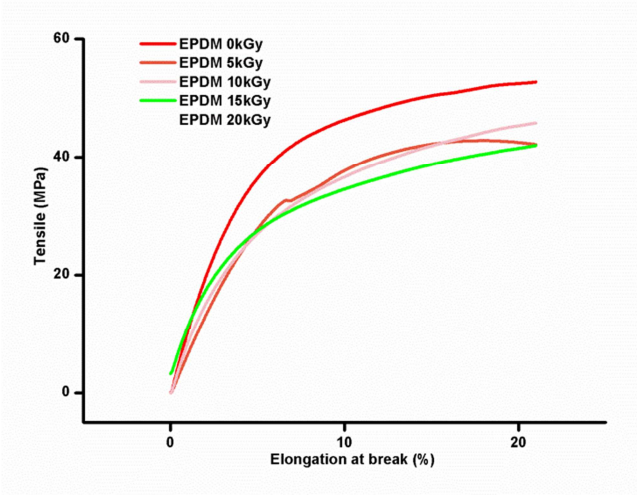

Fig. 2. Mechanical behavior of EPDM gamma-irradiated at: 0, 5, 10, 15 and 20 kGy.

The effect of gamma-irradiation on samples was investigated up to their failure. Tensile strength indicated by the force exerted on samples under tension, were determined from the typical stress–strain curve. When subjected to air-gamma irradiation, PP suffers a decreasing in tensile strength, imparted by scission from its quaternary carbon, that prevails on crosslinking, as shown in Fig. 1. Epdm, according to Fig. 2, shows a reduction in tensile, in function of gamma-irradiation; nevertheless, even at 20 kGy, it exhibits a value (appr. 40 MPa) higher than that one presented by Pp at 0 kGy: appr. 27 MPa. Elongation at break remained constant in all samples, irradiated and non-irradiated ones. Figs. 3, 4 and 5 shows for all Pp/Epdm blends assessed very close tensile results, without depicting a clear trend. However, it can be affirmed that the addition of Epdm to Pp contributed for a raise in tensile, for all blends, irradiated and non-irradiated ones.

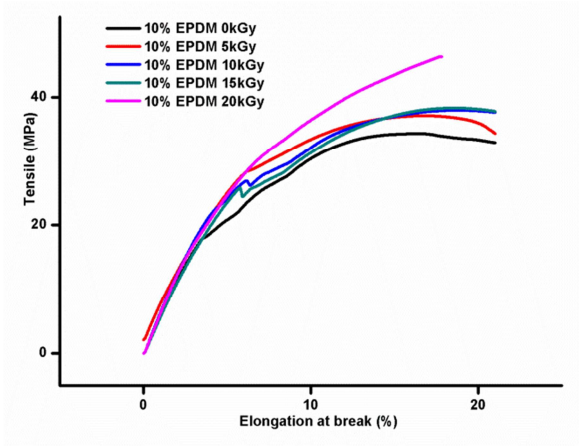

Fig. 3. Mechanical behavior of 90% PP/EPDM 10% blends gamma-irradiated at: 0, 5, 10, 15 and 20 kGy.

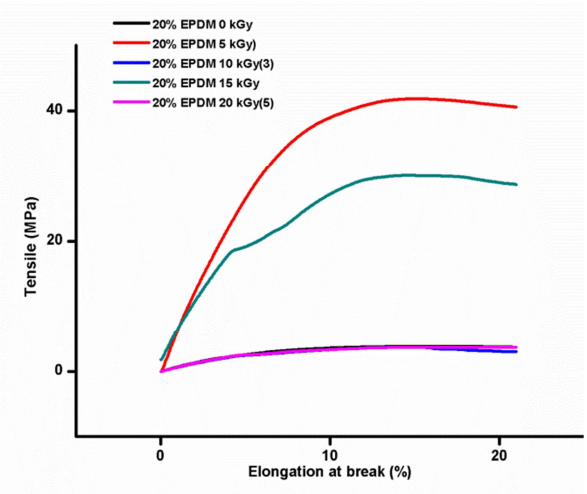

Fig. 4. Mechanical behavior of 80% PP/EPDM 20% blends gamma-irradiated at: 0, 5, 10, 15 and 20 kGy.

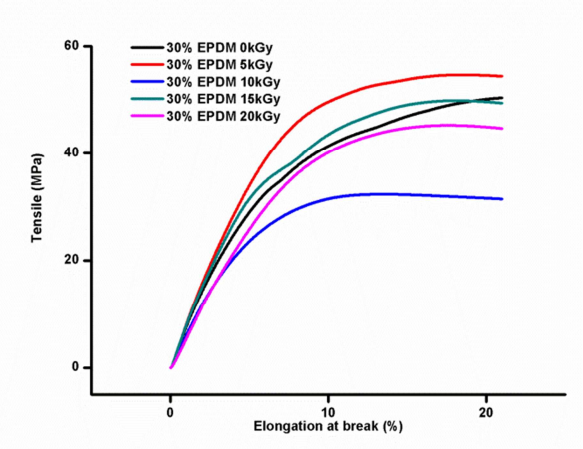

Fig. 5. Mechanical behavior of 70% PP/EPDM 30% blends gamma-irradiated at: 0, 5, 10, 15 and 20 kGy.

3.2. Thermal Behavior (DSC)

In DSC experiment, all samples were heated beyond the melting temperature and cooled back to room temperature, before the second scanning. A higher melting temperature is associated to a higher sample thermal stability. A decrease in heat of fusion suggests that the crystallinity and perfection of the crystal structure are reduced; all non-irradiatedEpdm/Pp blends presented lower heat of fusion and consequently, lower crystallinity. A change in the crystalline structure may result from polymer-polymer interactions in the amorphous phase; therefore, disorder in the crystals is created, reducing the enthalpy of the phase change and consequently, the crystallinity (O. W. Guirguis and M. H. Moselhey, 2012). Figs. 6 to 10 showed for all evaluated samples lower melting and crystallization temperatures in function of doses; all of them depicted lower melting and crystallization temperatures in function of imparted doses. In Table II is presented a complete thermal behavior for all samples tested.

Table 2. *DSC thermal behavior of samples, irradiated at 0, 5, 10, 15 and 20 kGy.*

	ΔH_1 (1st h) [mJ]	T_{m1} (1st h) [°C]	X_{c1} [%]	T_C (1stc) [mJ]	ΔH_2 (2nd h) [mJ]	T_{m2} (2nd h) [°C]	X_{C2} [%]
PP: 0	304.0	180.9	37.3	121.0	352.3	172.9	40.1
5	69.0	181.2	35.1	121.8	381.6	171.9	40.8
10	73.5	180.3	37.7	122.7	457.4	171.3	42.7
15	320.5	170.5	42.6	122.4	380.7	170.5	42.6
20	440.0	178.8	44.8	121.2	419.3	169.3	41.9
EPDM: 0	87.3	180.0	39.0	127.1	295.6	176.9	39.0
5	57.3	174.2	-----	123.7	111.5	173.5	-----
10	229.5	178.5	-----	126.8	261.1	177.4	-----
15	59.7	175.7	-----	124.9	45.5	170.2	-----
20	240.8	177.6	-----	126.5	239.5	174.2	-----
10% EPDM in PP: 0	88.7	180.8	35.6	121.1	376.0	173.7	42.7
5	85.0	178.9	38.2	126.7	385.9	173.5	41.2
10	54.6	178.9	35.7	121.2	342.1	171.2	40.5
15	367.8	178.5	40.9	123.5	374.0	170.9	41.7
20	390.0	178.5	36.6	125.3	443.7	171.0	42.4
20% EPDM in PP: 0	98.0	180.0	30.2	127.0	319.2	175.0	37.9
5	71.4	179.2	34.1	126.7	322.2	173.7	38.5
10	351.8	179.3	35.1	125.8	357.1	175.2	35.8
15	318.2	177.8	36.3	126.7	330.7	172.5	38.6
20	338.8	177.2	41.6	126.2	323.5	170.7	40.8
30% EPDM in PP: 0	275.6	180.2	29.3	126.6	327.9	177.7	34.1
5	291.6	179.2	31.7	127.0	341.5	174.0	38.3
10	305.2	179.8	41.1	126.6	370.8	173.3	41.1
15	365.3	177.8	35.0	127.0	392.3	172.5	39.1
20	315.7	178.3	30.2	126.1	407.8	172.6	40.5

Where: ΔH_1 and$\Delta H_2$are melt enthalpies related to first and second heating, respectively; T_{m1} and T_{m2} are melt temperatures related to first and second heating, respectively; T_c = crystallization temperature; X_{c1} and X_{c2}, crystallinity related to 1st and 2nd.

3.3. Termogravimetric Analyses (TGA)

The same sample procedure described above for DSC was followed for TGA investigations. From Figs. 11 to 15 it was possible to study thermal degradation for all samples assessed. Pure Pp non-irradiated and irradiated at 5, 10, 15 and 20 kGy exhibited overlapped curves, indicating a coincident thermal degradation. Pure Epdm showed an increase in thermal stability in function of irradiation doses. Epdm blends 10%, 20% and 30% in Pp showed a trend pointing toward higher thermal stability in function of irradiation. Table III confirms behavior shown by Figs. 6 to 10, indicating for Epdm and Pp blends T_{onset} (initial degradation temperature) values higher than those ones obtained for pure Pp. An average of 6.0% mass loss (residue) was found in tested samples, that corresponds to remaining degraded amount at temperatures higher than 600°C.

Table 3. *TGA behavior of samples, irradiated at 0, 5, 10, 15 and 20 kGy.*

	T_{onset} [°C]	T_{peak} [°C]	Residue [%]
PP: 0	373.5	450.0	6.9
5	373.5	450.0	5.6
10	373.5	450.0	6.0
15	373.5	450.0	6.1
20	373.5	450.0	5.5
EPDM: 0	391.7	450.0	5.6
5	425.7	452.0	6.3
10	392.7	454.0	5.1
15	434.0	456.0	4.3
20	423.5	460.0	5.5
10% EPDM in PP: 0	389.4	420.0	6.6

	T_{onset} [°C]	T_{peak} [°C]	Residue [%]
5	357.3	410.0	2.8
10	360.3	405.0	6.8
15	363.6	408.0	5.9
20	386.0	418.0	5.0
20% EPDM in PP: 0	399.6	450.0	7.0
5	397.6	450.0	5.9
10	405.1	450.0	5.6
15	397.5	450.0	6.3
20	403.5	450.0	6.3
30% EPDM in PP: 0	391.6	450.0	5.3
5	397.8	450.0	5.7
10	400.1	450.0	6.5
15	398.3	450.0	5.8
20	399.8	450.0	5.4

T_{onset} = initial termal degradation temperature in each stage; T_{peak} = thermal degradation peak temperature in each stage; Residue = remaining degraded sample amount, at temperature higher than 600°C.

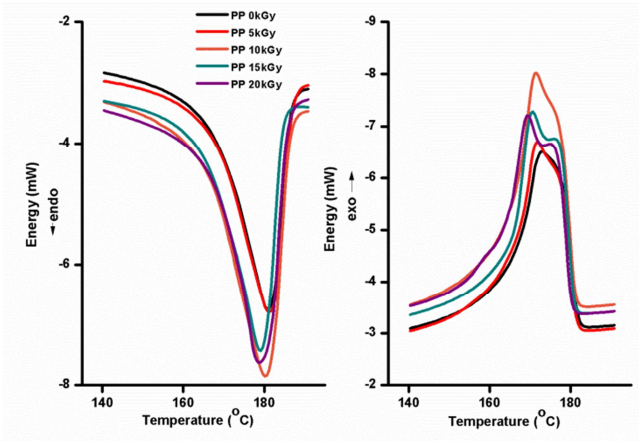

Fig. 6. *DSC thermal behavior for PP irradiated at 0, 5, 10, 15 and 20 kGy.*

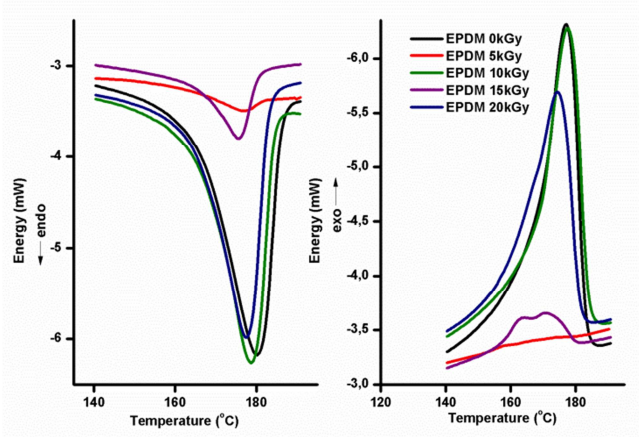

Fig. 7. *DSC thermal behavior for EPDM irradiated at 0, 5, 10, 15 and 20 kGy.*

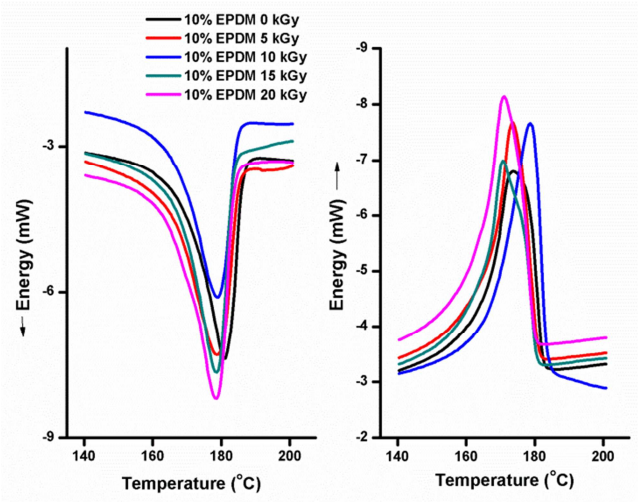

Fig. 8. *DSC thermal behavior for 10% EPDM in PP blends, irradiated at 0, 5, 10, 15 and 20 kGy.*

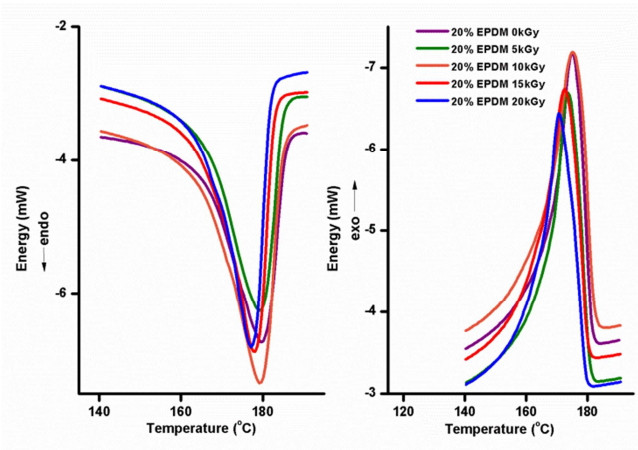

Fig. 9. *DSC thermal behavior for 20% EPDM in PP blends, irradiated at 0, 5, 10, 15 and 20 kGy.*

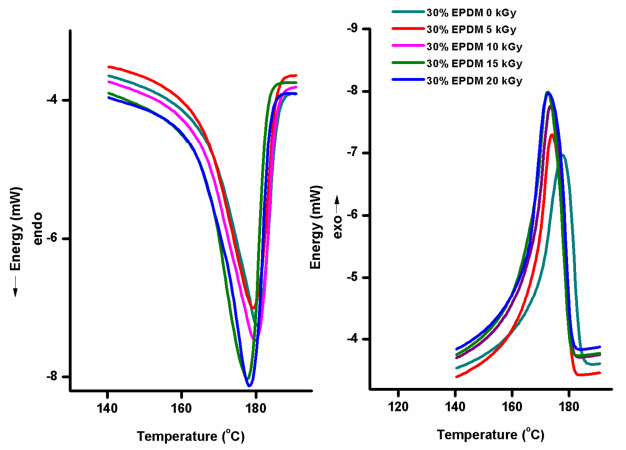

Fig. 10. *DSC thermal behavior for 30% EPDM in PP blends, irradiated at 0, 5, 10, 15 and 20 kGy.*

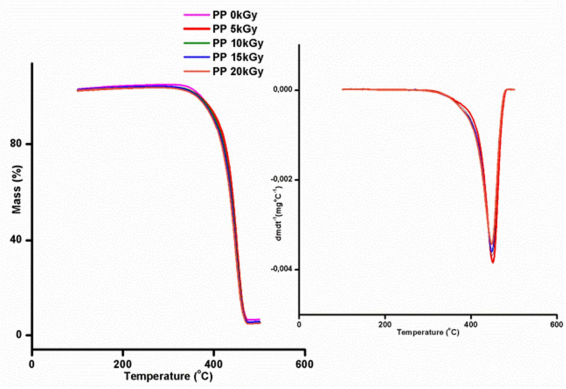

Fig. 11. *TGA curves for PP irradiated at 0, 5, 10, 15 and 20 kGy.*

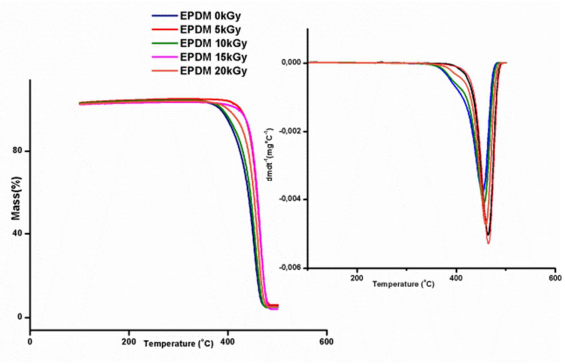

Fig. 12. *TGA curves for EPDM irradiated at 0, 5, 10, 15 and 20 k*

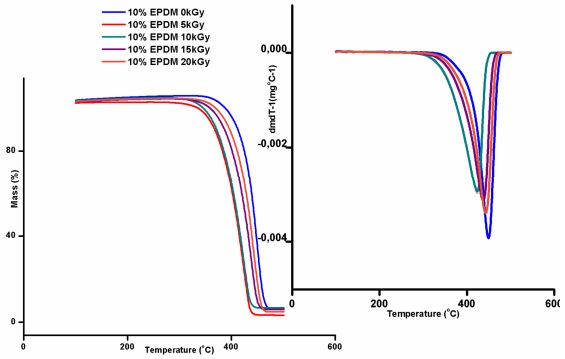

Fig. 13. *TGA curves for 10% EPDM in PP samples irradiated at 0, 5, 10, 15 and 20 kGy.*

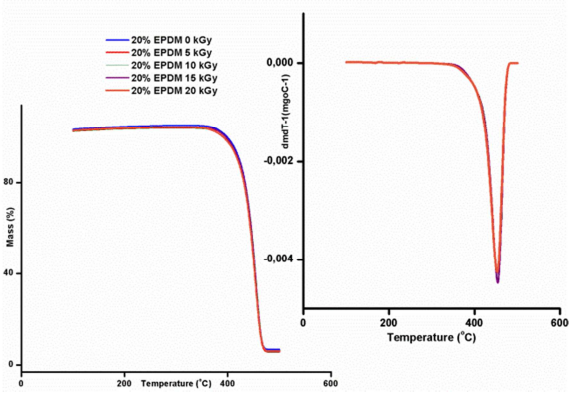

Fig. 14. *TGA curves for 20% EPDM in PP samples irradiated at 0, 5, 10, 15 and 20 kGy.*

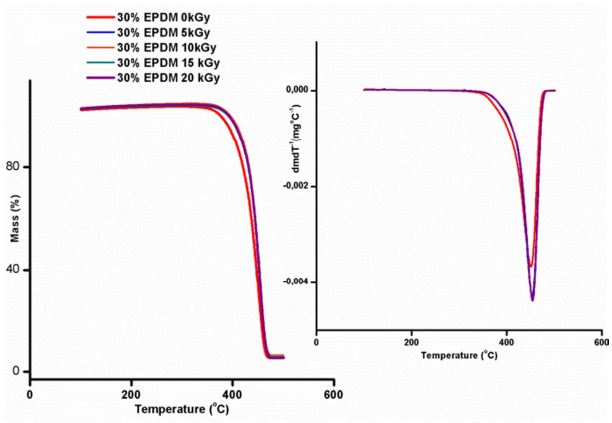

Fig. 15. *TGA curves for 30% EPDM in PP samples irradiated at 0, 5, 10, 15 and 20 kGy.*

3.4. Melt Flow Index

Melt Index (MI), Melt Flow Index (MFI), or Melt Flow Rate (MFR) refers to the grams per 10 minutes pushed out of a die of prescribed dimensions according to ASTM D 1238-13, under the action of a specified load; herein, the experiment was carried out at 230°C, being Pp used as reference. The MFR, then, is an indicator of average molecular weight and is inversely related to it. A resin with an MFR of 50 g/10min indicates a lower molecular weight than one with an MFR of 10 g/10 min. While a higher MFR material may be easier to process, physical properties related to molecular weight, such as impact resistance, are often lower.

Table IV shows MFR evaluations for non-irradiated samples; a higher addition of Epdm to Pp reduced MFR and consequently will contribute for higher molecular weight compounds. All irradiated samples, when tested in the plastometer, showed a very low viscosity, making impossible their assessment; this was due to degradation experimented by Pp air gamma irradiated and Pp/Epdm blends.

Table 4. *Melt Flow Index of non-irradiated samples.*

	PP	10% EPDM In PP	20% EPDM In PP	30% EPDM In PP
M. F. R. at 230°C ($g.10min^{-1}$)	3.6	3.4	2.7	1.9

3.5. Izod Impact Strength

The impact resistance of a polymer is a function of the base resin plus the presence of any impact modifiers (such as elastomers). A low Izod value does give a warning that care should be exercised in design to avoid sharp corners or similar points of stress concentration. It does not by itself, however, necessarily indicate high notch sensitivity. As shown in Fig. 16, impact values obtained for pure Epdm are superior to those ones found for pure Pp. All blends Epdm/Pp showed still higher impact values, either at zero kGy or 20 kGy, pointing toward a mechanical property optimization induced by gamma irradiation.

3.6. Attenuated Total Reflection Fourier Transform Infrared Spectroscopy (ATR-FTIR)

Figs. 17 to 21 showed just peaks inherent to Pp and Epdm, at frequencies (cm^{-1}): 3,000 – 2,850 (C – H stretch, alcanes); 1,375 (CH_3, angular deformation); 1,460 (CH_2, angular deformation); 1,320 – 1,000 (C – O, stretch); 950 –910 (OH bend). There were not recorded hydroxyl, carbonyl, ether, epoxide and double bond formation, according to possible reaction types on Epdm structure via irradiation, as shown in Figures 22 to 27 (F. Hacioglu, 2010), due to low doses involved: 5, 10, 15 and 20 kGy.

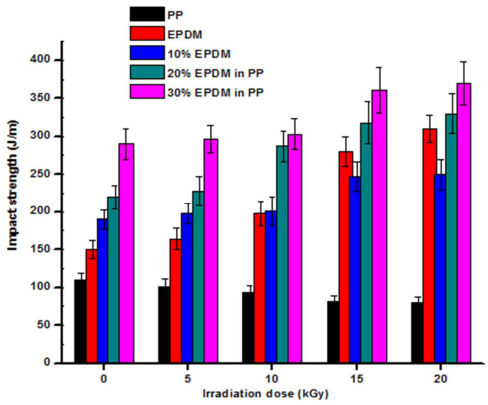

Fig. 16. Notched Izod impact values for pure PP and EPDM and their blends.

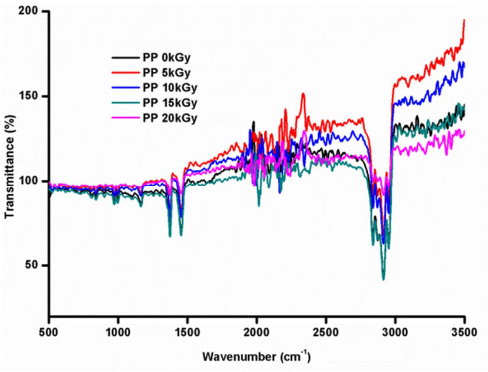

Fig. 17. FTIR spectra for PP irradiated at: 0, 5, 10, 15 and 20 kGy.

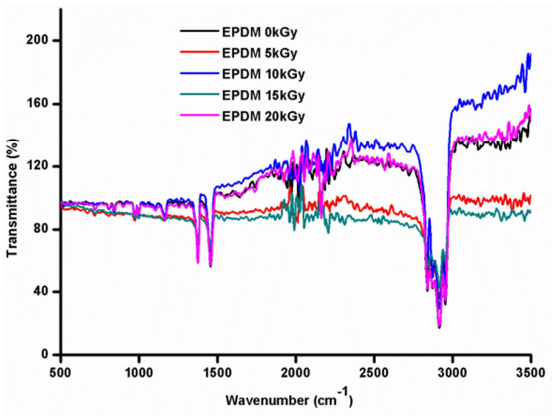

Fig. 18. FTIR spectra for EPDM irradiated at: 0, 5, 10, 15 and 20 kGy.

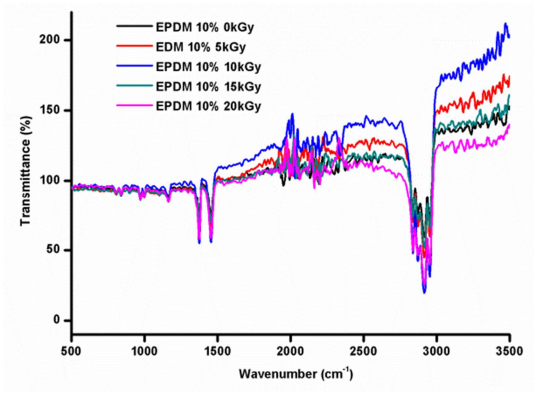

Fig. 19. FTIR spectra for 10% EPDMin PP irradiated at: 0, 5, 10, 15 and 20 kGy.

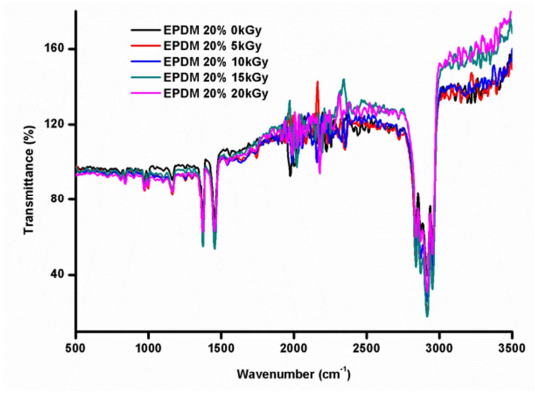

Fig. 20. FTIR spectra for 20% EPDMin PP irradiated at: 0, 5, 10, 15 and 20 kGy.

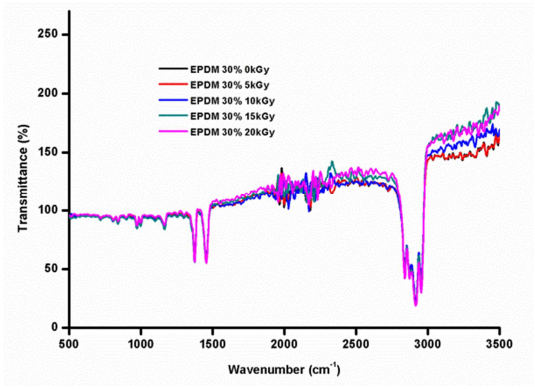

Fig. 21. FTIR spectra for 30% EPDMin PP irradiated at: 0, 5, 10, 15 and 20 kGy.

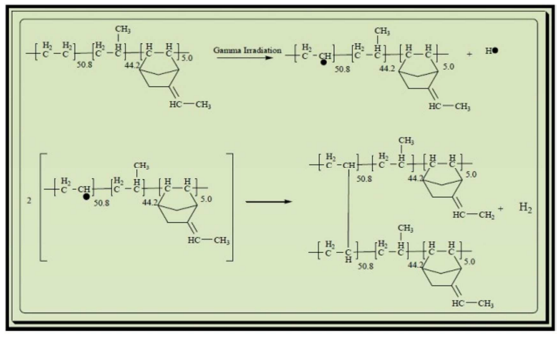

Fig. 22. Crosslinking reactions of EPDM (from ethylene parts) via irradiation.

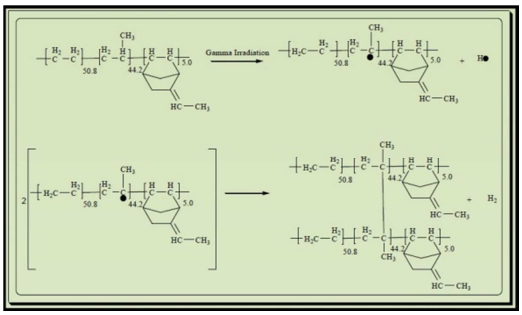

Fig. 23. *Crosslinking reactions of EPDM (from propylene parts) via irradiation.*

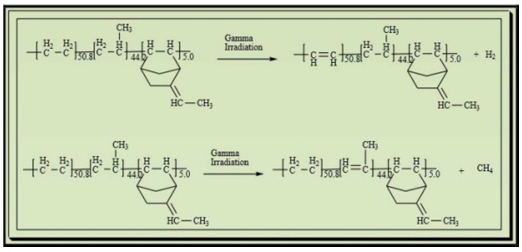

Fig. 24. *Double bond formation of EPDM via irradiation.*

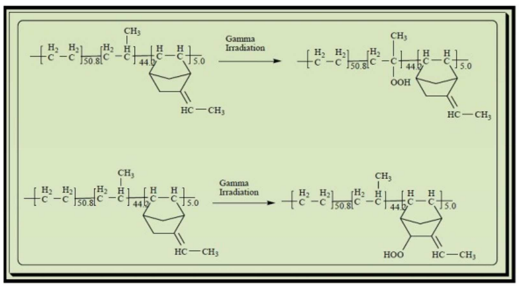

Fig. 25. *Oxidation of EPDM via irradiation.*

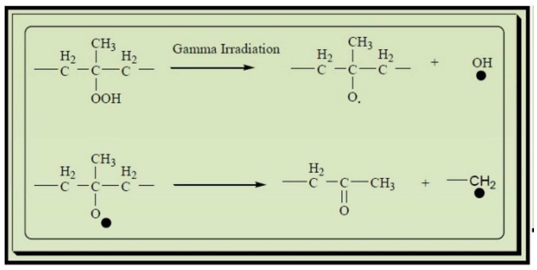

Fig. 26. *Carbonyl formation of EPDM (from propylene unit) via irradiation.*

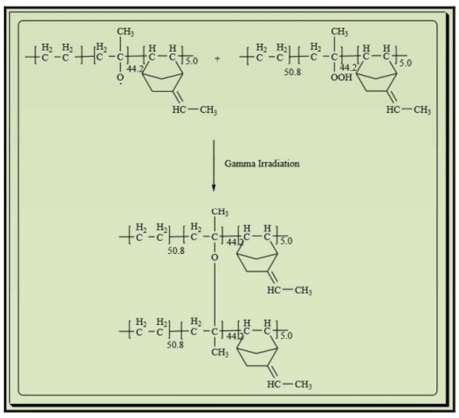

Fig. 27. *Ether formation of EPDM (from propylene unit) via irradiation.*

4. Conclusions

Epdm addition to Pp at 10, 20 and 30% level contributed for optimizing tensile strength and impact resistance. Melting and crystallization temperatures kept low values for all samples tested, independent of imparted doses. A higher thermal stability was experienced by Pp/Epdm blends. Epdm contributed for melt index decreasing in Pp/Epdm blends, pointing toward a higher molecular weight distribution, favoring mechanicals properties. Low doses used: 5, 10, 15 and 20 kGy were not enough to provoke structure changes, according to FTIR assessments.

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