Thermomechanical and Deformation Properties of Electron Beam Modified Polypropylene Copolymer Grafted with Acrylic Monomer

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Abstract: **Mechanical and structural properties of polypropylene (PP) block copolymer irradiated with 5 MeV electron beam irradiation (absorbed doses 25 – 150 kGy) have been investigated.**

Increase of the strength-strain characteristics of PP modified with bisphenol a dimethacrylate monomer (BAD) as the cross-linking agent with concentration of 0.5 – 3 wt.% have been researched.

Properties of materials grafted with BAD have been investigated by the methods of differential scanning calorimetry and infrared spectroscopy.

Thermorelaxation (σ_{TR}, MPa) and residual stresses (σ_{RS}, MPa) caused by isometric heating and subsequent cooling of irradiated and oriented (ε = 100%) samples with 3 wt.% BAD have been measured.

Thermorelaxation (σ_{TR}, MPa) and residual stresses (σ_{RS}, MPa) have indicated a relatively high efficiency of cross-linking.

Increase of mechanical properties (E, σ_b, and $\varepsilon_{b)}$ at absorbed doses 25 - 150 kGy with increase in the content of BAD in comparison to entire PP has been determined.

Keywords: **polypropylene, electron beam irradiation, cross-linking promoter, thermomechanical and structural properties**

I. Introduction

Polypropylene (PP) is widely used in packaging materials, medical and pharmaceutical products, coating materials and other industrial and engineering application areas.

High energy ionization radiation methods (γ-rays, accelerated electrons) have been widely used in polymer industry over the last 70 years. Radiation processing has several advantages over conventional chemical and thermal processing, especially in the process of radiation sterilizing of polypropylene in electron accelerators [1].

There are two competing processes that are accompanied by polymers during the irradiation – the cross-linking that leads to the increase in thermal, mechanical, electrical, chemical properties, and the degradation of polymeric chains, chemical bond cleavage as a result of radiation-induced chain scission. Thermal and radiation-induced oxidation processes during the irradiation and during the post irradiation time can affect processes of material ageing. These structural changes are characteristic of PP especially at high irradiation doses.

Several additives have been investigated by many scientists like antioxidants based on hindered amines, organic-phosphites and phenol type antioxidants like Irganox 1010 [1,2], and several cross-linking promoters (cross-linking agents, sensitizers and antirads) mainly based on monomers with allyl and acrylate functional groups that can engage in radical reactions by grafting and cross-linking to polymer backbone [3,4,5], and improve the resistance to chain scission during and after the exposure of polymer systems to ionizing irradiation. The authors of [6] have found that a well-known cross-linking agent triallyl cyanurate (TAC) monomer could act as an energy transfer agent, resulting in prevention of degradation due to the transfer of energy through the triazine ring that is not possible in the case of aliphatic acrylates like trimethylolpropane triacrylate and others.

Bisphenol a dimethacrylate (BAD) as aromatically stabilized acrylate monomer is an alternative to aliphatic promoters. The functional group of bisphenol synthesized from acetone (the reason of acronym a) has two phenol type aromatic rings stable against the radiation, which is a well-known fact in case of aromatic compounds like aromatic polyesters, polycarbonate and other monomer and polymer compounds. The aromatic unit has a protective effect, the resonant structure of the aromatic ring enabling a considerable amount of energy to be absorbed without any rupture of the bonds [7]. It is predictable that radiation grafting of BAD to PP could act as a protection group against undesired effects of irradiation and perhaps also could improve the mechanical and thermal stability of polypropylene.

The additive of BAD monomer already has been used as a cross-linking agent by irradiation grafting on bisphenol polycarbonate and resulted in better cross-linking determined by increase of the gel fraction in comparison with triallyl cyanurate [8].

The first aim of our research was to investigate changes on stress – strain properties of irradiated polypropylene and the application of BAD additive on improvement of the radiation stability of polypropylene.

It is well known that polyolefin type polymers like polyethylene and its composites are widely used for manufacturing of thermoshrinkable materials that show a "form-memory effect" achieved by the preliminary cross-linking of polyethylene amorphous phase by the effect of ionizing radiation. Upon treating the polyethylene by ionizing radiation, a reticulate molecular structure is formed, whose degree of cross-linking depends on the absorbed radiation dose [9, 10].

Although thermoshrinkable products of radiation cross-linked polyethylene have been used in different technologies for many years, studies on investigation of new type of thermoshrinkable materials still remain urgent. Radiation stabilized (by grafting with radiation promoters that increase

the gel fraction of PP) polypropylene may also be used for these applications.

Thermorelaxation and residual thermoshrinkage stresses caused by heating and subsequent cooling of irradiated and oriented to 100% polypropylene samples with 3 wt.% of the grafted monomers have been investigated for such a purpose.

Calorimetric data like melting and crystallization temperatures and enthalpies as well as changes of structure and degree of crystallinity of investigated materials have also been investigated.

II. MATERIALS AND INVESTIGATION METHODS

Isotactic polypropylene block copolymer of trademark PHC31 (density ρ – 0.905 g/cm^3) supplied by SABIC was used as the matrix material. Bisphenol a dimethacrylate (m. p. 72^{o}C, BAD) supplied by Sigma-Aldrich (Fig. 1) was used as a cross-linking promoter additive.

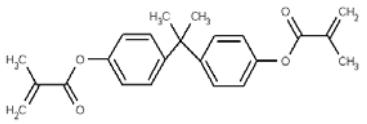

Fig. 1. Structure of bisphenol a dimethacrylate (BAD)

Blending polypropylene pellets with cross-linking promoter was carried out by thermoplastic mixing at the temperature of 190^{o}C for a total duration of 4 min. The concentration of additive was as follows: 0.5, 1 and 3 wt.%. No other additives (antioxidants etc.) were used.

Compression-moulded blends (with average thickness 0.5 mm) were obtained by pressing at 190^{o}C and 1 MPa.

Obtained blends were irradiated in linear particle accelerator with accelerated electrons (energy 5 MeV, dose rate 1.2 MGy/h, the temperature of irradiation – 290 ^{o}C) in air atmosphere. The absorbed irradiation doses D_{abs} were 25, 50, 100 and 150 kGy.

Stress-strain behaviour of composites was determined by Tinius Olsen H1KOS universal testing machine at 20^{o}C at a tension rate of 50 mm/min for stress-strain parameters and at a tension rate of 1 mm/min for determination of elastic modulus according to ISO 527-1 standard. 5 parallel specimens were tested.

Thermomechanical behaviour of the radiation modified blends, after their preliminary orientation by extension up to 100%, was investigated in the temperature range of 20 to 200°C. Specimens in the form of 5 x 5 x 2 mm strips were gradually heated and cooled under isometric conditions. The arising thermal relaxation σ_{TR} and residual thermal shrinkage σ_{SHR} stresses were determined by a strain gage with 0.0l N sensitivity.

Differential scanning calorimetric measurements of melting, cooling and second melting were carried out by Mettler Toledo DSC 1/200W equipment in the temperature range from 25^{o}C to 300^{o}C at the heating rates 10^{o}C/min. The calorimeter was calibrated before use with indium standard. The weight range of the samples was from 10 to 12 mg. The weight of the sample was taken into account so that the DSC curves were rescaled to 1 g of sample.

Fourier transform infrared spectra (FTIR) were measured on Thermo Nicolet 380 FT-IR device with Smart Orbit attenuated total reflectance analyzer from 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1}.

III. RESULTS AND DISCUSSION

Mechanical characteristics of polypropylene and specimens with different content of cross-linking promoter BAD were obtained form the stress-strain curves, which showed the character of semi-crystalline polymers with a formation of neck at the yield point. The effect of both the content of the additives, as well as the amount of absorbed irradiation dose was established from the curves.

Elastic modulus of entire PP increased with increment of absorbed dose. Values of elastic modulus of polypropylene grafted with BAD monomer showed similar manner – mechanical stiffness of PP materials grafted with the promoter increased with increment of the content of BAD in PP matrix as a result of cross-linking and chain scission processes during the irradiation (Fig. 2).

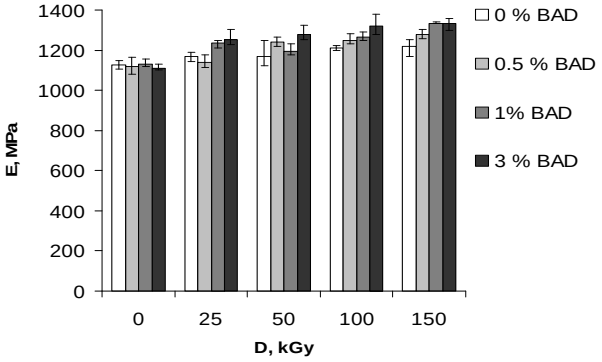

Fig. 2. Effect of the content of BAD on the changes of elastic modulus (E) for blends irradiated with different absorbed doses (D_{abs})

Changes of deformation properties are illustrated in Fig. 3 and Fig. 4.

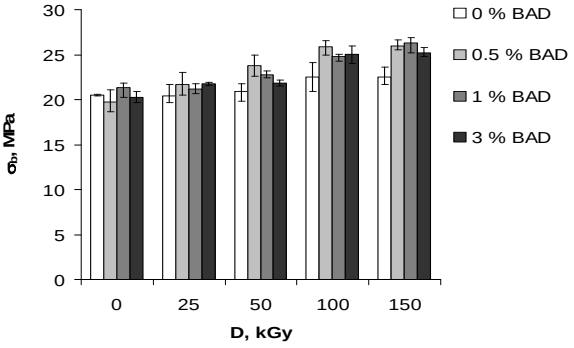

Fig. 3. Effect of the content of BAD on the changes of tensile strength at break (σ_b) for blends irradiated with different absorbed doses (D_{abs})

For entire PP and specimens with the content of BAD 0.5-3 wt.% values of strength at break σ_b increased gradually with the increment of absorbed dose and with the content of the grafting concentration of the BAD monomer indicating increase of mechanical stiffness of material as a result of cross-linking.

Values of elongation at break for entire PP and specimens with BAD gradually decreased with the absorbed dose as a result of cross-linking process. The process of decrease reduces with the increment of the content of BAD as it is seen in Fig. 4.

For example, the values of elongation at break ε_b for PP grafted with 3 wt.% BAD at dose 150 kGy were 39% in comparison with ε_b value 20% of entire PP at 150 kGy.

Mechanical properties of PP grafted with cross-linking promoter based on bisphenol a dimethacrylate showed the increased mechanical stability and a better cross-linking effect even at the content of BAD 0.5 wt.% in comparison with the entire PP monomer. It can be explained by the result of the previously discussed effect of aromatic units in the structure of BAD especially at the doses higher 50 kGy when the cross-linking and chain scission reactions occur due to the absorbance of some part of the energy that could harm the structure due to the scission and breakage of large structure of PP branched with BAD and cross-linked through the bridge groups of BAD moieties.

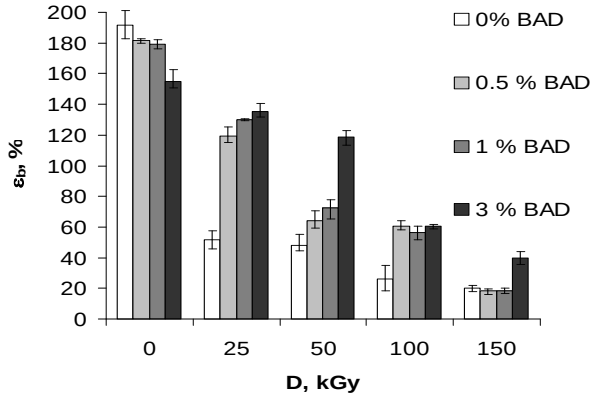

Fig. 4. Effect of the content of BAD on the changes of elongation at break (ε_b) for blends irradiated with different absorbed doses (D_{abs})

Thermomechanical curves obtained from isometricaly oriented (ε = 100%) PP specimens with the content of BAD 3 wt.% are illustrated in Fig. 5.

In the initial process of producing thermoshrinkable materials, it is necessary to find the optimal temperature for the orientation extension process of a radiation cross-linked material. For a high density polyethylene it is carried out at 140 – 150°C – at the temperature above the melting point of the crystalline phase of [9]. Different temperature regimes were investigated above the m. p. temperature of our PP from 150 °C to180°C. It was found that the optimal temperature for the orientation extension was 160 °C.

From all the irradiated specimens (with the content of promoters 0.5 – 3 wt. % and entire PP), it was possible to orientate only the samples with the content of BAD 3 wt.% The other specimens, especially irradiated at absorbed doses >50 kGy and with absence of promoter, began to melt during the orientation process that indirectly implied to low gel fraction and cross-linking efficiency.

Thermomechanical curves of PP with the content of 3 wt.% BAD showed similar characteristics to cross-linked entire polyethylene irradiated up to absorbed doses > 150 kGy in comparison with thermorelaxation streses σ_{TR} [10]. Values of thermorelaxation stresses were in the range of 0.9 – 0.4 MPa and decreased with increment of irradiation dose to 100 kGy. Values of residual thermal shrinkage stresses were in the range of 1 – 0.5 MPa and also decreased with the increment of absorbed dose.

The relatively small values of residual thermal shrinkage stresses σ_{RS} may be attributed to low crystallinity of PP that is seen also from the results of DSC analysis (Table 1). It also coincides with the results of deformation (Fig. 4) properties according to which plasticity of PP increase with increment of the content of BAD monomer grafted to PP.

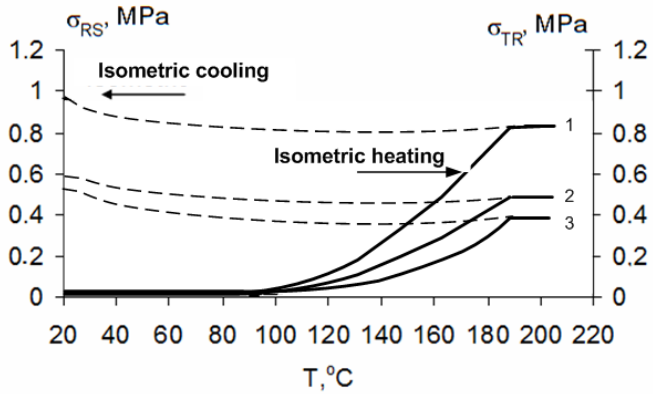

Fig. 5. Thermomechanical curves of oriented PP blends grafted with 3% BAD and irradiated with absorbed doses D_{abs}: 1 – 25 kGy; 2 – 50 kGy; 3 – 100 kGy

For PP samples, the first heat cycle was performed in order to erase the thermal and ageing history of the samples, and the cooling scan (Fig. 7) and the second heating scan (Fig. 6) were analyzed, as further heating scans gave the same DSC curves. All the data are compared in Table 1.

DSC analyses were performed on the specimens of PP, both non-irradiated and irradiated at absorbed doses of 25 and 150 kGy. In the case of the PP samples, the second temperature scan is the result of morphological changes resulting both from irradiation and chemistry induced by the first heat cycle on the polymer.

Some of the useful information derived from DSC heating scans included the melting temperature, which was taken as the maximum of the endothermic peaks, and the heat of fusion, determined by integrating the areas under the endothermic peak. The melting temperatures of PP were similar to that of PP homopolymer indicating a small effect of the constituent of polyethylene on the melting temperature in copolymer.

The temperature ranges of the melting temperatures derived from the second heating DSC scans of the melting peaks extended from 125 to 175°C for the unirradiated entire PP, from 123 to 172°C for the 25 kGy irradiated and from 121 to 168°C for the 150 kGy irradiated entire PP specimen. Broad shoulder of the endothermic peak of PP specimen irradiated at 150 kGy dose was detected. The temperature ranges for PP grafted with 3 wt.% BAD extended from 124 to 174°C for the 25 kGy irradiated and from 118 to 172°C for the 150 kGy irradiated specimen.

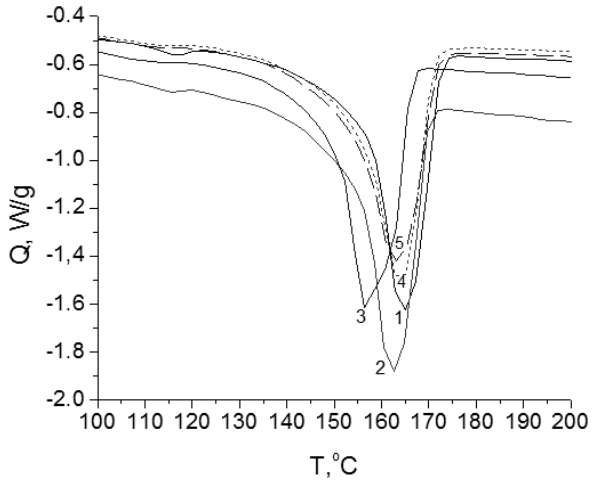

Fig. 6. DSC curves of melting from the 2nd heating scans of entire PP (1 – 0 kGy; 2 – 25 kGy; 3 – 150 kGy) and PP grafted with 3 wt.% BAD (4 – 25 kGy; 5 – 150 kGy)

DSC analysis revealed that maximum melting temperatures of entire PP shifted to lower temperatures with increase of the irradiation dose (Fig. 6, Table 1) as a result of cross-linking and chain scission, the second more predominant at doses > 25 kGy and indicating changes in morphology of PP. Results of maximum melting temperatures of PP grafted with BAD indicated compatibility of the components and effect of additive on cross-linking processes by stabilizing PP that is considered by a very low shift of DSC endothermic peaks to lower temperatures and by decrease of heat fusion enthalpies (Fig 6, Table 1). That may be attributed to protective effect of BAD aromatic ring preventing cleavage of the backbone of PP and acting in cross-linking and chain scission processes.

Ratios of the melting heats of the specimens and the melting heat of the completely crystallized polypropylene ($\Delta H_{f\,100\%PP}$ = 207 kJ/g) were used to calculate the degree of crystallization. The results showed in Table 1 indicate decrease of crystallinity for samples grafted with BAD, which coincide with mechanical data indicating increased cross-linking effectiveness with increase of the content of BAD.

The exothermic peaks of entire PP and grafted with BAD have similar character to melting peaks, indicating the effect of irradiation dose on changes of crystallinity (Fig.7). The results obtained from cooling scans indicate how rapidly the PP crystallizes. Increase of irradiation dose affects the decrease of crystalline rate as the effect of cross-links and the increase of amorphous phase as the cross-linking occurrence.

Cross-linking agent BAD has a small effect on crystallization enthalpies as it is seen in Table 1 and in Fig. 7.

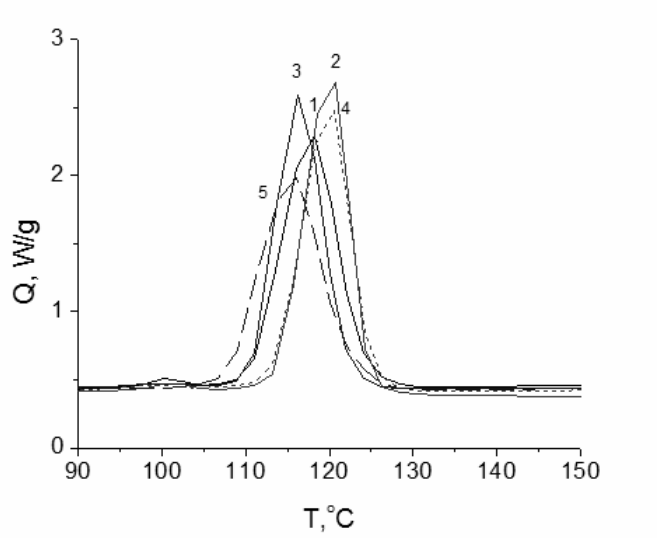

Fig. 7. DSC curves of crystallization of entire PP (1 – 0 kGy; 2 – 25 kGy; 3 – 150 kGy) and PP grafted with 3 wt.% BAD (4 – 25 kGy; 5 – 150 kGy)

The effect of cross-linking reactions affecting formation of spherulites results in increased clarity and stiffness but also imparts some brittleness at dose 150 kGy.

The changes of crystallization are in coincidence with relatively low values of thermoshrinkable stresses in thermomechanical curves (Fig. 5).

TABLE 1

CALORIMETRIC DATA OF ENTIRE AND GRAFTED PP GRAFTED WITH 3 WT.% BAD IRRADIATED WITH DIFFERENT ABSORBED DOSES

Sample	Dose, kGy	Melting (the 1st heating scans)		Melting (the 2nd heating scans)		Crystallization (from cooling scans)		χ_c (%)
		T_m (°C)	ΔH_f (J/g)	T_m (°C)	ΔH_f (J/g)	T_c (°C)	ΔH_c (J/g)	
PP	0	167.58	79.69	165.05	84.9	117.78	88.72	41.0
PP	25	167.98	78.74	162.42	85.55	120.21	89.58	41.3
PP	150	163.98	79.43	156.37	83.95	116.27	86.34	40.5
PP + 3 % BAD	25	168.29	75.07	164.13	81.3	120.01	86.16	38.1
PP + 3 % BAD	150	162.22	76.81	160.1	79.76	115.77	84.21	37.4

The FTIR spectra of entire unirradiated PP and specimens irradiated at absorbed doses 25 and 150 kGy (Fig. 8) consisted of similar absorption bands indicating the isotactic nature of PP – peaks in the range of 2700 – 3000 cm^{-1} attributed to symmetric and asymmetric stretching of CH, CH_2 and CH_3 groups, peaks in the range of 1430 – 1450 cm^{-1} attributed to bending of CH_3 and CH_2, various strong, medium and weak peaks in the range of 807 – 1375 cm^{-1} attributed to bending, rocking, wagging, twisting, stretching and rocking, the absorption bands at 999 – 977, 1167 and 1219 cm^{-1} attributed to the helicoidally conformation of PP indicating the isotacticity of PP.

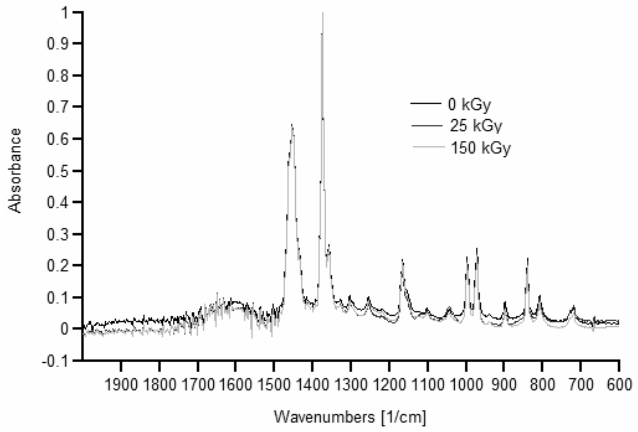

Fig.8. FTIR spectra of entire PP irradiated with different absorbed doses (D_{abs}, kGy)

The increase of absorbance bands with increment of absorbed dose at bands of 1102 and 1044 cm^{-1} may be attributed to some C-O signals of additives. Small increase of medium absorbance peak of 1303 cm^{-1} attributed to wagging and stretching of methylene may be due to addition of more CH_2 groups as the effect of cross-linking. Decrease of the bands with the dose at 840 and 898 cm^{-1} attributed to length of isotactic sequences may also be attributed to increase of cross-links that may complicate the ability to bend PP backbone and affect scission of the C–H and C–C bonds in the polypropylene due to cross-linking and chain scission reactions.

Characteristic absorption band at 728 cm^{-1} attributed to sequence of four contiguous methylene groups and band at 720 cm^{-1} attributed to 5 contiguous methylene groups indicated the character of PP block copolymer with increased absorbance at absorbed dose 25 kGy that indicated the cross-linking in these moieties.

As predicted, no signs of thermal or radiation-induced oxidation like bands of carbonyl groups (C=O) at 1700 – 1800 cm^{-1} and changes of –OH groups at 3000 – 3600 cm^{-1} attributed to stable oxidation products (ketones, acids, esters or lactones) were detected or the signals coincided with the baseline. Small recognizable signals in the range of 1630 – 1660 cm^{-1} attributed to double bounds of vinylidene groups were discovered due to some β-chain scission at absorbed dose of 150 kGy.

Important absorbance bands were observed in spectra of PP modified with 3 wt.% BAD (Fig. 9). Absorbance bands of carbonyl groups attributed to BAD decreased with increase of absorbed dose. Other special signals unrecognized in spectra of entire PP and attributed to changes of BAD were investigated – like signals of C-H out of plane stretching of aromatic moieties and signals of C-O ester groups in the range of 1000 – 1200 cm^{-1}, and in the range of 700 – 800 cm^{-1} some of bands overlapped with medium and weak signals of PP. Increase of C-O signals at 1144 cm^{-1} also confirmed participation of BAD additive in grafting and cross-linking reactions.

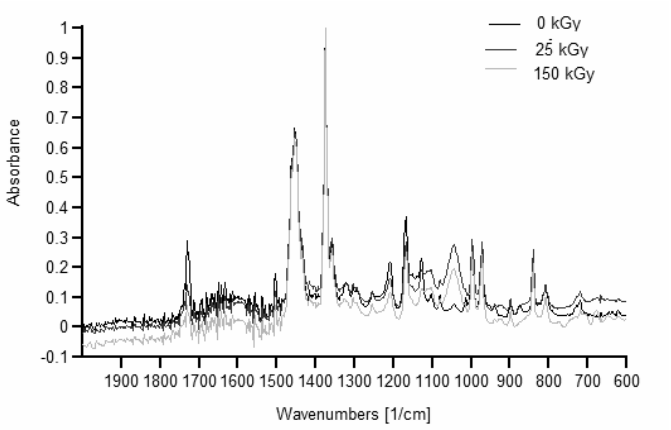

Fig.9. FTIR spectra of PP grafted with 3 wt.% BAD irradiated with different absorbed doses (D_{abs}, kGy)

Changes in helicoidally structure of PP are recognized as effect of BAD grafting to PP confirmed from decrease and broadening of absorbance bands attributed to block copolymer groups, indicating decrease of crystallinity confirmed also by DSC data.

IV. Conclusions

Changes of mechanical and thermomechanical properties of polypropylene modified with BAD cross-linking promoter and irradiated with accelerated electrons up to a dose of 150 kGy highly improve mechanical properties. Values of tensile strength increase with the irradiation dose and the values of elongation at break decrease indicating the grafting and cross-linking processes that increase with the content of grafted monomers to the backbone of polypropylene.

Specimens grafted with the BAD monomer showed the increased radiation stability due to the existence of the aromatic ring enabling a considerable amount of energy to be absorbed without any rupture of the bonds. Increased deformability observed for entire PP is reduced by the process of irradiation-induced grafting of 3 wt.% bisphenol a dimethacrylate to PP backbone.

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Ingars Reinholds, Valdis Kaļķis, Jānis Zicāns, Remo Merijs Meri, Ilze Elksnīte. Ar elektronu starojumu apstarota polipropilēna, kam piepotēts akrilātu monomērs, termomehāniskās un deformatīvās īpašības.
Pētītas ar paātrinātiem elektroniem (enerģija 5 MeV) apstarota polipropilēna (PP) blokkopolimēra mehāniskās un struktūras īpašības pie jonizējošā starojuma (JS) absorbētām dozām 25...150 kGy. Kā radiācijas šķērssaistīšanās aģents izmantots difenola-a-dimetakrilāta monomērs (DAD) ar koncentrāciju 0,5...3 m.%. Termiskās izturības un struktūras pētījumiem izmantota diferenciālās skenējošās kalorimetrija un infrasarkanā spektrometrija. Uzņemot izometriskās karsēšanas un dzesēšanas diagrammas orientētiem (ε = 100%) plēves veida paraugiem, noteiktas termorelaksācijas spriegumu ($\sigma_{TR,}$ MPa) un paliekošo nosēdspriegumu ($\sigma_{PN,}$ MPa) vērtības. Novērots, ka pamatā mainās σ_{TR} vērtības, kas ir relatīvi augstas (0,4...0,9 MPa), salīdzinot ar kompozīcijām, kuras veidotas uz augsta vai zema blīvuma polietilēna bāzes (0,2...0,4 MPa). Novērota arī stiprības īpašību (E, $\sigma_{b,}$ un ε_b) palielināšanās kompozīcijām PP/DAD, salīdzinājumā ar tīru PP, ja palielina radiācijas promotora saturu. Mehānisko īpašību izmaiņas, salīdzinot PP un PP/DAD pie relatīvi mazas JS absorbētās dozas 25 kGy, norāda uz to, ka JS inducētās radikāļu reakcijās notiek galvenokārt DAD monomēra piepotēšanās, kā rezultātā izmainās materiāla virsmolekulārā struktūra un 2,6 reizes palielinās trūkšanas pagarinājums ε_b (51% tīram PP un 135% PP ar DAD saturu 3 m.%). Otrā sildīšanas cikla kalorimetriskie dati (maksimālās kušanas temperatūras, kušanas entalpijas un kristāliskuma pakāpes izmaiņas) liecina par jaunas šķērssaistītas PP-DAD struktūras izveidošanos, ko apstiprina arī infrasarkano spektru izmaiņas.

Ингарс Рейнхолдс, Валдис Калькис, Янис Зицанс, Ремо Мери Мерий, Ильзе Елксните. Термомеханические и деформационные свойства облученного электронным излучением полипропилена с привитым мономером акрилата.
Исследованы механические и структурные свойства блок-сополимера полипропилена (ПП), облученного ускоренными электронами (энергия 5 МэВ) до поглощенных доз ионизирующего излучения (ИИ) 25...150 кГр. В качестве сшивающего агента использован мономер бисфенол-а-диметакрилат (БАД) с концентрацией 0,5...3 мас.%. Для исследования термической стойкости и структурных свойств использованы дифференциальная сканирующая калориметрия и инфракрасная спектроскопия. По результатам диаграмм изометрического нагрева и охлаждения ориентированных (ε =100%) пленкообразных образцов судили о величины терморелаксационных напряжений (σ_{TP}, МПа) и остаточных напряжений (σ_{OC}, МПа). Установлено, что в основном изменяются величины σ_{TP}, которые относительно более высокие (0,4...0,9 МПа) по сравнению с величинами, которые имеются у композиций на основе полиэтилена высокой или низкой плотности (0,2...0,4 МПа). Обнаружено также увеличение прочностных свойств (E, $\sigma_{p,}$ un ε_p) у композиций ПП/БАД, по сравнению с чистым ПП, если увеличить содержание БАД. Изменения механических свойств композиции, сравнивая ПП и ПП/БАД при относительно низкой поглощенной дозе ИИ равной 25 кГр, указывает, что в в основном происходит реакция привитой сополимеризации БАД и ПП, и при этом изменяется надмолекулярная структура, о чем свидетельствует увеличение прочности при разрыве ε_p в 2,6 раза (51% у чистого ПП и 135% у ПП, содержащего 3 мас.% БАД). Калориметрические данные второго цикла нагрева (изменение максимальной температуры плавления, энтальпии плавления и степени кристалличности) указывает на образование новой сшитой структуры ПП-БАД, что подтверждает в основном изменения инфракрасных спектров.