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Short vegetal-fiber reinforced HDPE—A study of electron-beam radiation treatment effects on mechanical and morphological properties

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ABSTRACT

The effects of electron-beam radiation treatment on fiber-matrix adhesion and mechanical properties of short piassava fibers reinforced high density polyethylene (HDPE) matrix were studied. Glycidyl methacrylate (GMA) was added at 2.5% and 5.0% (on piassava fiber wt) as a cross-linking agent and the effects upon the properties of the resulting composites treated by electron-beam radiation were also examined. HDPE reinforced with short piassava fiber composites was prepared by melt-mixing processing, using a twin screw extruder machine. The materials were irradiated with 100 and 200 kGy using a 1.5 MeV electron beam accelerator, at room temperature in presence of air. Material samples were submitted to mechanical and thermo-mechanical tests and SEM analyses. Correlation between properties was discussed. The comparison of mechanical and thermo-mechanical properties of the composites showed that electron-beam radiation treatment produced a significant improvement in mechanical properties, when compared with the non-irradiated composite sample and neat HDPE. Scanning electron microscopy (SEM) studies of the composite failure surfaces indicated that there was an improved adhesion between fiber and matrix. Examination of the failure surfaces indicated dependence of the interfacial adhesion upon the radiation dose and GMA content. Better interfacial adhesion between fiber and HDPE matrix was observed for composites with 5.0% GMA addition and treated with electron-beam radiation dose of 200 kGy. It can be concluded that GMA addition followed by electron-beam irradiation treatment, at the doses studied in this work, effectively improved the HDPE properties and led to the obtaining of composite materials with superior properties suitable for several industrial applications.

Introduction

Studies on polymer composite materials with fiber reinforcement matrix were initially carried out with man-made fibers such as glass, aramid, carbon and others, because they satisfy the desired conditions, generally can withstand maximum load and transfer strength to the matrix constituent influencing and enhancing their properties. Lately the interest in fibers of vegetable origin for replacing man-made fibers has increased dramatically because vegetable fibers are available in abundant volume throughout the world, they are renewable and biodegradable. Other relevant factors are the low demand for energy and environmental impacts of these composite products, compared with more conventional materials [1,2].

Both thermoplastic and thermoset materials can be reinforced and made into composite materials, but composites with very short fibers tend to have thermoplastic matrices. This is because fibers must be able to go through small clearances, such as the gap between the extruder screw and the extruder wall or the gate that connects the mold cavity with the runner system in both injection molding and transfer molding when being extruded or injection molded. Moreover, thermoplastics often need the additional strength or additional stiffness gained from reinforcing with short fibers [3].

High density polyethylene (HDPE) is a linear thermoplastic polymer widely used, presenting balanced mechanical properties, chemical resistance, impermeability to water, low cost and easy processing advantages [4,5]. The molecular weight, the molecular weight distribution and the amount of branching determine many

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of the mechanical and chemical properties of the end product. HDPE has been used with natural fibers to prepare composites with good performance [5–7]. A promising approach for the controllable modification of the polymer materials properties is based on ionizing radiation treatment. In general, the cross-linking and degradation processes occur, simultaneously, in amorphous regions of polymers as an effect of ionizing radiation. Cross-linking of HDPE into three-dimensional networks leads to improvement of properties such as tensile strength, chemical resistance and thermal characteristics [8].

Piassava (*Attalea Funifera* Mart) is a Brazilian lignocellulosic fiber extracted from the leaves of a palm tree of natural occurrence in the Atlantic rain forest and its exploitation is an extractive activity that represents the main source of income to approximately 2000 small-scale farmers, processors and their families. Piassava fibers have been described as harder than other lignocellulosic fibers and have higher lignin content (around 48%) than any of the other common lignocellulosic fibers [9]. This could be responsible for its inherent flexural rigidity and water proof resistance. The main use of these fibers is for industrial and domestic brooms, industrial brushes, ropes, baskets, carpets and roofs. It is estimated that around 50% of the fiber is discarded during the cut, cleaning and baling and as residue by the transformation industries [10].

A promising approach to the controllable modification of the polymer materials properties is based on ionizing radiation treatment, like electron-beam and gamma irradiation. The irradiation of polymeric materials with ionizing radiation (gamma rays, X-rays, accelerated electrons, ion beams) leads to the formation of very reactive intermediates. These intermediates can follow several reaction paths which result in rearrangements and/or formation of new bonds. The ultimate effects of these reactions can be the formation of oxidized products, grafts, scissionning of main chain (degradation) or side chains or cross-linking in amorphous regions of the polymers. The degree of these transformations and the predominance of degradation or cross-linking effect depends on the structure of polymer (presence or absence of radiation resistant groups such as aromatic groups, double substitution on a carbon atom on the main chain) and of the conditions of treatment, such as, irradiation atmosphere, presence or absence of some additives and dose rate. Dose is defined as the amount of energy absorbed per unit mass. The dose of one kGy is the absorption of one Joule of energy per gram of material [11–13]. When the application of radiation processing in polymer and plastics industries is considered, the cross-linking effects are seen to dominate the field. Cross-linking is a broadly used method for the modification of polymer properties. This process involves the formation of three-dimensional structures causing substantial changes in material properties. Cross-linking leads to the formation of insoluble and infusible polymers in which polymer chains are joined together to form three-dimensional network structure. Such networks can impart superior mechanical and thermal properties to starting polymers. The degree of cross-linking is proportional to the radiation dose. Scission reduces cross-linking efficiency and degrades the properties of polymers such as chemical resistance, mechanical, and thermal properties [14–16]. Radiation-induced cross-linking is also widely accepted for the high speed curing of coatings, for the production of composite materials, and for improves of the properties, and of the interface adhesion between matrix and filler of the polymeric composite materials. Under irradiation with high energy beams, HDPE predominantly undergoes cross-linking in the amorphous regions; the cross-linking effectively increases intermolecular bonds in this region of HDPE, resulting in an improvement in material properties, such as better mechanical, thermal and chemical properties [16–18].

Glycidyl methacrylate (GMA) has attracted enormous interest partially because of its reactive epoxide group, which can undergo a number of chemical reactions under appropriate conditions. Moreover, some homo- and copolymers based on GMA have found a variety of practical applications, such as surface modification and compatibilizers for polymer blends. GMA contains α, β-unsaturated carbonyl structure, which may lead to cross-linking or strong interfacial adhesion. In some studies, GMA was used as a cross-linking agent, by which hydrogels could be prepared, and also functional polymers were synthesized by the ring-opening reactions of epoxide groups in GMA units [19]. GMA has been used as a compatibilizing agent to prepare rubber wood with styrene [20], and to prepare styrene–acrylonitrile copolymer/organically modified nanoclay [21]. Hassan et al. investigated the effect of gamma irradiation on some properties of reclaimed rubber/acrylonitrile–butadiene rubber/GMA blend and its swelling in motor and brake oils. Results showed that the tensile strength, hardness values, and swelling resistance increased with radiation dose applied. Hassan et al. investigated that the swelling percentage steadily decreases with increasing radiation dose due to the radiation-induced cross-linking [22].

Table 1
Formulation of the composites.

Composites	GMA (wt%)[a]	Piassava fibers (wt%)
HDPE/piassava fiber (COMP)	0	30
HDPE/2.5GMA/piassava fiber (COMP1)	2.5	30
HDPE/5.0GMA/piassava fiber (COMP2)	5.0	30

[a] Based on the piassava fiber weight.

Material and methods

Material

The materials used in this study were HDPE resin (HDPE JV060U—commercial grade by Braskem S/A) with MFI = 6.41 g/10 min at 190 °C/2.16 kg, specific density = 0.957 g/cm^3, piassava fiber from agro-industrial residues and glycidyl methacrylate (GMA) as a cross-linking agent.

Piassava fibers preparation

Piassava fiber residues were scraped, washed, and kept in distilled water for 24 h. The fiber was then dried at 80 ± 2 °C for 24 h in an air-circulating oven. The dry fiber was reduced to fine powder (piassava), with particle sizes equal to or smaller than 250 μm by using ball mills and then it was dried again at 80 ± 2 °C for 24 h to reduce its moisture content to less than 2%.

Glycidyl methacrylate addition

Glycidyl methacrylate "GMA" (2.5% and 5.0% in weight; based on the piassava fiber weight) was added into dry piassava fiber as a cross-linking agent. The mixtures were added in the HDPE matrix.

Composites preparation

HDPE/piassava fiber composites were prepared by melting extrusion process, using an extrusion machine twin screw "extruder AX 16LD40" made by AX Plásticos Máquinas Técnicas Ltda. The compounded materials passed through the different zones of the extruder and were finally extruded (Table 1). The extrudates coming out of the extruder were cooled down by using cold water for a better dimensional stability, and they were wound up manually. Finally, the HDPE/piassava fiber composite was dried, pelletized by a pelletizer, fed into an injection molding machine and specimen test samples were obtained.

Table 2
Tensile response for the irradiated and non-irradiated neat HDPE and HDPE/piassava fiber composites.

Materials	Properties	Radiation dose (kGy)		
		NI	100	200
Neat HDPE	Tensile strength at break (MPa)	5.6 ± 0.18	17.1 ± 0.6	21.5 ± 1.2
	Young's modulus (MPa)	200 ± 6.9	326 ± 16.5	249 ± 21
COMP[a]	Tensile strength at break (MPa)	18.7 ± 0.9	21 ± 0.5	20.8 ± 0.8
	Young's modulus (MPa)	388 ± 6.6	422 ± 5.2	423 ± 5.2
COMP1[b]	Tensile strength at break (MPa)	19.8 ± 0.7	20.6 ± 0.8	22.2 ± 0.3
	Young's modulus (MPa)	413 ± 12	413 ± 5.2	435 ± 8.1
COMP2[c]	Tensile strength at break (MPa)	18.9 ± 0.9	22.4 ± 0.6	23 ± 0.4
	Young's modulus (MPa)	408 ± 24	461 ± 9.5	460 ± 6.2

[a] HDPE/piassava.
[b] HDPE/piassava + 2.5% GMA.
[c] HDPE/piassava + 5.0% GMA.

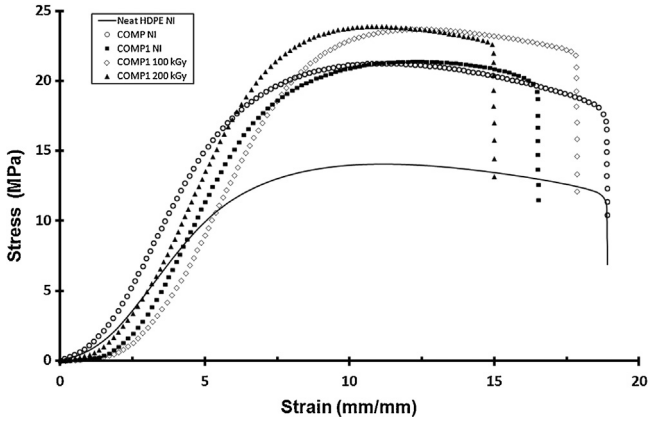

Fig. 1. Tensile stress–strain curves for non-irradiated (NI) neat HDPE, non-irradiated (NI) HDPE/piassava fiber composites (COMP), and for irradiated and non-irradiated HDPE/piassava fiber composites with 2.5% of GMA (COMP1).

Electron-beam irradiation

HDPE/piassava fiber composites and neat HDPE were irradiated at radiation dose of 100 and 200 kGy using an a electrostatic accelerator (Dynamitron II, Radiation Dynamics Inc., 1.5 MeV energy, 25 mA current and 37.5 kW power), at room temperature, in air, dose rate 14 kGy/s. The irradiated and non-irradiated samples were submitted to mechanical tests (tensile, flexural, and impact strength), thermo-mechanical tests (heat distortion temperature (HDT) and Vicat softening temperature), and SEM analysis and the correlation between their properties and radiation dose applied was discussed.

Characterization

Mechanical and thermo-mechanical tests

Neat HDPE and HDPE/piassava fiber composites were characterized by: tensile (ASTM D 638), flexural (ASTM D 790), impact strength (ASTM D 256-06), heat distortion temperature (HDT) (ASTM D 648) and Vicat softening temperature (ASTM D 1525).

Scanning electron microscopy (SEM)

The scanning electron microscopy (SEM) analyses for the neat HDPE and HDPE/piassava fiber composites cryo-fractured (under liquid nitrogen) samples were carried out using a LX 30 (Philips) instrument. The cryo-fractured surface of samples were coated with a thin layer of gold and observed by scanning electron microscopy.

Results and discussion

Mechanical and thermo-mechanical test results

Tensile test results

Figs. 1–2 show the tensile stress–strain curves for neat HDPE and irradiated and non-irradiated HDPE/piassava fiber composites. From these figures it can be seen that tensile strength and modulus of neat HDPE increase significantly by piassava fiber addition, but the greater increases are due to the piassava fiber and GMA addition followed by electron-beam irradiation treatment.

Table 2 shows the tensile response obtained for the irradiated and non-irradiated neat HDPE and HDPE/piassava fiber composites. The results presented show the average values calculated from the data obtained in tests for five test specimens. These results showed an increase of around 250% in tensile strength at break of neat HDPE due to addition of the piassava fiber with GMA, and of around 300% for the composites consisting of HDPE reinforced with piassava

Table 3
Flexural test results for the irradiated and non-irradiated neat HDPE and HDPE/piassava fiber composites.

Materials	Properties	Radiation dose (kGy)		
		NI	100	200
NEat HDPE	Flexural strength (MPa)	17.05 ± 0.3	20.2 ± 0.4	23.6 ± 0.5
	Flexural modulus (GPa)	0.68 ± 0.03	0.78 ± 0.03	0.93 ± 0.04
COMP[a]	Flexural strength (MPa)	22.5 ± 0.7	14.8 ± 0.5	15.7 ± 0.4
	Flexural modulus (GPa)	0.46 ± 0.02	0.83 ± 0.03	0.90 ± 0.03
COMP1[b]	Flexural strength (MPa)	24.07 ± 0.7	16.1 ± 0.1	17.3 ± 0.1
	Flexural modulus (GPa)	0.49 ± 0.02	0.93 ± 0.05	0.97 ± 0.01
COMP2[c]	Flexural strength (MPa)	24.0 ± 0.6	15.1 ± 0.4	17 ± 0.2
	Flexural modulus (GPa)	0.49 ± 0.01	1.0 ± 0.08	0.96 ± 0.03

[a] HDPE/piassava.
[b] HDPE/piassava + 2.5%GMA.
[c] HDPE/piassava + 5.0%GMA.

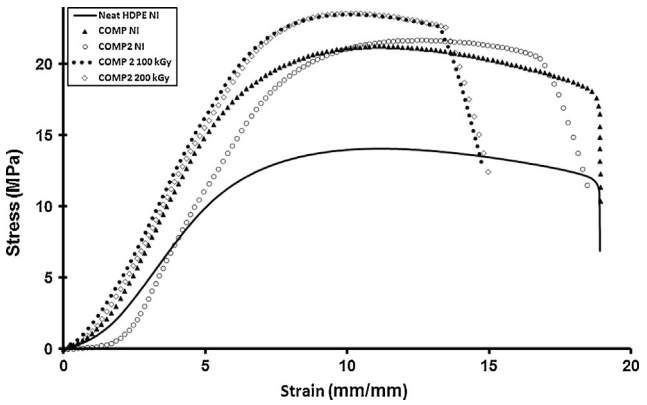

Fig. 2. Tensile stress–strain curves (MPa) × strain (mm/mm) for non-irradiated (NI) neat HDPE, non-irradiated (NI) HDPE/piassava fiber composites (COMP), and irradiated and non-irradiated HDPE/piassava fiber composites with 5.0% of GMA (COMP2).

fiber and GMA treated by electron-beam irradiation. It can be distinctly observed that addition of piassava fiber with GMA led to the increase in Young's modulus of around 100% and of around 130%, when the composite are also treated by electron-beam irradiation.

It is clear from these results that, as widely reported in the literature [8,12,16,17], electron-beam irradiation induced predominantly cross-linking in the amorphous regions of HDPE, whereas the cross-linking effectively increases intermolecular bonds in this region, resulting in an improvement in material properties, such as better mechanical properties. Furthermore, the GMA addition, a radiation cross-linking agent, likely increased even further the cross-linking degree by radiation of HDPE.

Flexural test results

Table 3 presents the flexural test results for the irradiated and non-irradiated neat HDPE and HDPE/piassava fiber composites. The results presented show the average values calculated from data obtained in tests for five test specimens. As it can be seen clearly in Table 3, flexural strength of HDPE/piassava fiber composites presented gains of around 30% due to piassava addition, and 40% due to piassava and GMA addition, in comparison with neat HDPE. However, after electron-beam radiation reductions in flexural strength of the composites can be observed, whereas for irradiated neat HDPE at 100 kGy increased by 18% and at 200 kGy by 38%, but these values are even smaller than that of the non-irradiated composites containing GMA. Concerning the flexural modulus, non-irradiated composites presented a reduction, in comparison with neat HDPE, while the irradiated composites presented equal or greater flexural modulus than the irradiated neat HDPE, depending on the radiation dose applied.

Notched Izod impact, HDT and Vicat test results

The test results of notched Izod impact and the thermo-mechanical test results (heat distortion temperature "HDT" and Vicat softening points) of neat HDPE and HDPE/piassava fiber composites are showed in Table 4. As expected that the notched Izod impact of neat HDPE decreased due to the piassava and GMA addition. The heat distortion temperature (HDT) of neat HDPE

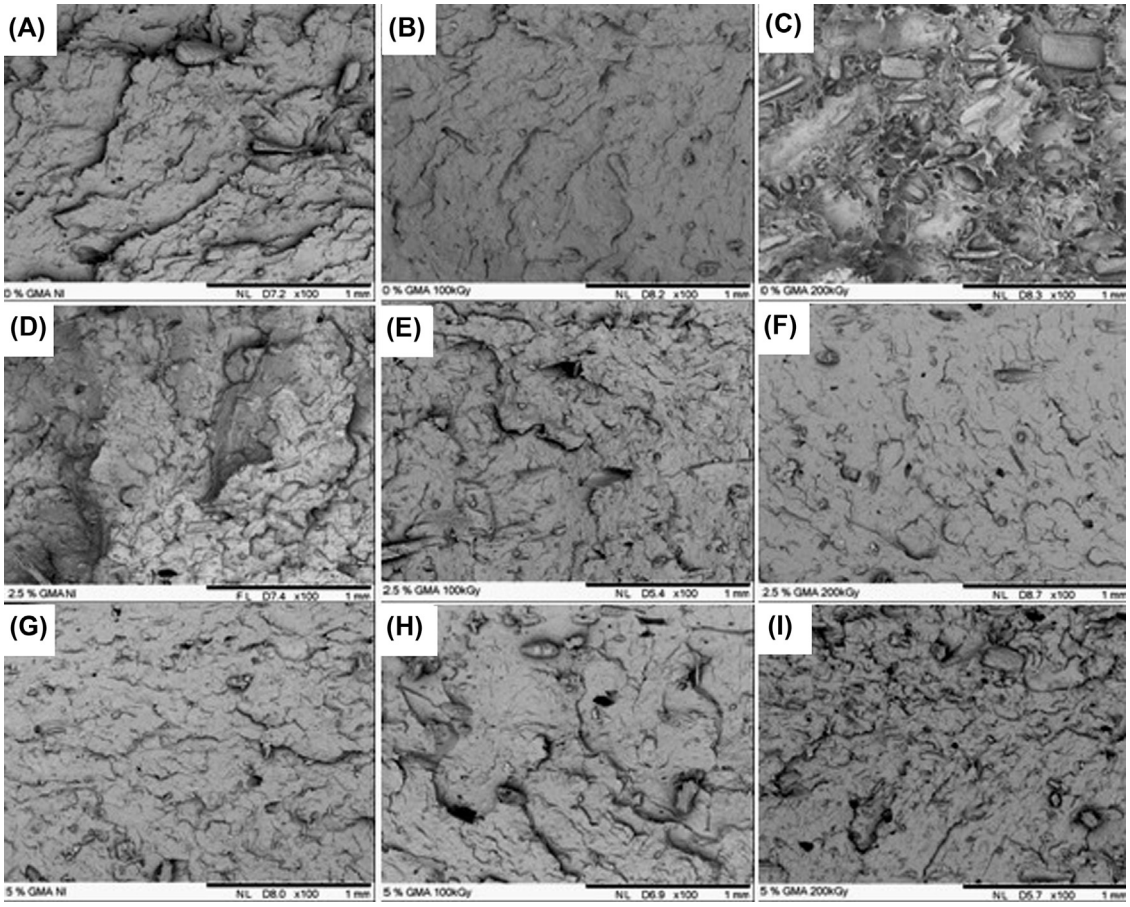

Fig. 3. SEM micrographs for cryo-fractured surfaces of the HDPE/piassava fiber (500×): (A) non-irradiated HDPE/piassava fiber; (B) HDPE/piassava fiber, 100 kGy; (C) HDPE/piassava fiber, 200 kGy; (D) non-irradiated HDPE/piassava + 2.5% GMA; (E) HDPE/piassava + 2.5% GMA, 100 kGy; (F) HDPE//piassava + 2.5% GMA, 200 kGy; (G) non-irradiated HDPE/piassava + 5.0% GMA; (H) HDPE/piassava + 5.0% GMA, 100 kGy; (I) HDPE/piassava + 5.0% GMA, 200 kGy.

Table 4
Notched Izod impact, heat distortion temperature (HDT) and Vicat softening points test results for the irradiated and non-irradiated neat HDPE and HDPE/piassava fiber composites.

Materials	Properties	Radiation dose (kGy)		
		NI	100	200
Neat HDPE	Notched Izod impact (J/m)	77.3 ± 1.6	318 ± 3.1	750 ± 2.7
	HDT (°C)	56.3 ± 0.3	57.2 ± 0.1	60 ± 0.2
	Vicat (°C)	126 ± 0.2	128 ± 0.3	130 ± 0.4
COMP[a]	Notched Izod impact (J/m)	50.0 ± 0.2	53.3 ± 0.1	66.7 ± 1.2
	HDT (°C)	101 ± 0.2	105 ± 0.3	108 ± 0.1
	Vicat (°C)	128 ± 0.3	129 ± 0.3	130 ± 0.3
COMP1[b]	Notched Izod impact (J/m)	36.7 ± 0.1	36.7 ± 0.1	43.4 ± 0.9
	HDT (°C)	103 ± 0.3	104 ± 0.1	108 ± 0.7
	Vicat (°C)	128 ± 0.2	129 ± 0.3	129 ± 0.8
COMP2[c]	Notched Izod impact (J/m)	36.7 ± 0.1	36.7 ± 0.1	53.3 ± 0.6
	HDT (°C)	106 ± 0.3	109 ± 0.3	107 ± 0.5
	Vicat (°C)	128 ± 0.3	129 ± 0.1	130 ± 0.4

[a] HDPE/piassava.
[b] HDPE/piassava + 2.5%GMA.
[c] HDPE/piassava + 5.0%GMA.

increased of around 30% due to the piassava. The addition of GMA and electron-beam irradiation showed no marked improvement in HDT. Vicat softening points of neat HDPE showed a slight due to piassava fiber and GMA addition, and electron-beam irradiation.

Scanning electron microscopy (SEM)

Fig. 3 presets the SEM micrographs of cryo-fractured surfaces of non-irradiated and irradiated HDPE/piassava Fiber composites. As can be seen in Fig. 3 non-irradiated HDPE/piassava fiber composites shows several cracks and voids between fiber and matrix; irradiated HDPE/piassava fiber composites with 100 kGy shows fiber pull-out, little deformation and fracture with flat surfaces that suggest a brittle failure processes; irradiated HDPE/piassava fiber composites with 200 kGy shows several cavities or voids between fiber and matrix that result in poor adhesion between fiber and matrix strength; the cryofractured surface of irradiated HDPE/piassava fiber + 2.5% GMA and HDPE/piassava fiber + 5.0% GMA with 100 and 200 kGy suggest that extensive plastic deformation takes place before fracture, characteristic of material with more ductile fracture. It can be observed some fibers break and a better interfacial adhesion between fiber and HDPE matrix in comparison with the surfaces of the other composites.

Conclusion

The objective of the present work was to evaluate the effects of electron-beam radiation treatment on fiber-matrix adhesion and mechanical properties of short piassava fibers reinforced high density polyethylene (HDPE). Glycidyl methacrylate (GMA) was added as a cross-linking agent and their effects upon the properties of the resulting composites treated by electron-beam radiation were also examined. The comparison of mechanical properties of the composites showed that electron-beam radiation treatment produced a significant gain in mechanical properties. Scanning electron microscopy (SEM) studies of the composite failure surfaces indicated that there was an improved adhesion between fiber and matrix due to radiation treatment and GMA addition. The better interfacial adhesion between fiber and HDPE matrix was observed for composites samples containing GMA and treated with 200 kGy of electron-beam radiation. The gain in mechanical properties for the irradiated composites was even higher when 5.0% (on piassava fiber wt) of GMA was added, due to the better interfacial adhesion between fiber and HDPE matrix. So, it can be concluded that GMA addition followed by electron-beam irradiation treatment, at the doses studied in this work, could lead to the obtaining of composite materials with better mechanical and morphological properties than untreated ones and could be a good alternative to replace conventional HDPE compounds for several applications.

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