

Effect of Ionizing Radiation Applied to PLA Used as Compatibilizing Agent in Reinforced eGG Shell PBAT/PLA Bio-Based Composites

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Abstract: Bio-filler from eggshells as reinforcement of bio-based polymers are based on their benefits as adequate strength and stiffness, besides friendly, degradable and renewable environment. Eggshell is an agricultural waste considered as garbage, contributing to pollution; nevertheless, it can be transformed into bio-calcium carbonate, acquiring new values. As biodegradable polymers, there were chosen PLA (poly-lactic-acid) and PBAT (butylene adipate co-terephthalate), thermoplastics capable to be processed via conventional methods. PLA is a linear, aliphatic thermoplastic polyester, high in strength and modulus, but brittle. PBAT is a synthetic polymer, very flexible, based on fossil resources with high elongation at break, but low strength. It will be required the use of compatibilizers, for reducing interfacial tension exhibited by PLA/PBAT immiscible blend, considering their extreme glass transition temperatures: 62 °C for PLA and - 30 °C for PBAT. Herein it was used ionizing radiation for inducing compatibilization by free radicals, improving dispersion and adhesion of blend phases, without using chemical additives, at room temperature. PLA, acting as compatibilizer, was previously e-beam and gamma radiated, at 150 kGy. PBAT/PLA 50/50 blend with 15 phr of bio-filler from avian eggs 125 µm particle size and both compatibilizers were homogeneized in a co-rotating twin-screw extruder, within a temperature profile 120 to 145 °C, from hopper to die. Characterization involved: Differential Scanning Calorimeter, Thermogravimetric Analyses, Fourier Transform Infrared Spectroscopy, Wide Angle X-Ray Diffraction, Tensile Strength and Elongation at Break.

Keywords: Ionizing radiation, eggshell, PLA, PBAT, compatibilization.

1. INTRODUCTION

The waste management associated with petroleum-based synthetic plastics has contributed for keeping focus on environmentally compatible materials from renewable sources, capable to substitute petroleum-based polymers in some applications [1]. Natural polymers, biopolymers and synthetic polymers based on renewable resources are the basis for the twenty-first century portfolio of sustainable and efficient plastics. The interest on these polymers is considerable, due to a decrease of world resources in oil; in addition, the lack of safe disposal and efficient degradation methods of synthetic polymers are posing an ecological threat to vegetation and animals [2]. A

large amount of plastic waste is being generated rapidly all over the world, being the origin of complex problems in aquatic environment, such as plastic debris pollutes, plastics entangling marine life, consumption of plastic items by marine fauna, etc... [3]. As alternative to synthetic plastics, biodegradable polymers are a newly emerging field [1-16], considering that *biodegradable* is the ability of a material to undergo biodegradation.

Bioplastics: a material is defined as bioplastic if it is either biobased, biodegradable or features both properties. Biobased materials are made from organic (carbon based) materials that contain in whole or part biogenic carbon, replacing petrol/fossil carbon with bio/renewable carbon. Herein there were used PBAT (Poly (butylene adipate-co-terephthalate)) and Polylactide or poly (lactic acid) (PLA) bioplastics blends. PBAT is an aliphatic-aromatic random co-polyester, fully biodegradable, prepared from 1,4 -

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butanediol, adipic acid and terephthalic acid: synthetic polymer based on fossil resources, 100% biodegradable, with high elongation at break. [17]. PBAT can be used in several applications, such as packaging materials, hygiene products, biomedical fields, industrial composting, among others [17 – 21]; nevertheless, PBAT has poor thermal and mechanical properties, that can be overcome with addition of fillers. Polylactide or poly(lactic acid) (PLA) is a linear, aliphatic thermoplastic polyester, used for different applications ranging from medical to packaging, resorbable and biodegradable under industrial composting conditions [22]; its rheological properties, especially its shear viscosity, have important effects on thermal processes. Some properties of PLA such as inherent brittleness, low toughness, slow crystallization, poor melt strength, narrow processing window and low thermal stability and high cost limit their large scale-applications [23 - 26].

PLA and PBAT binary blends showed improved properties, when compared to pure PLA. The addition of filler in PLA/PBAT blends will provide their reinforcement, based in a higher mechanical strength.

Herein, it was used as reinforcement bio-calcium carbonate from avian eggshells. Currently, egg production throughout the world is 65.5 million metric tons per year [27]. The eggshell is rich in calcium carbonate, natural bio-ceramic composite with an unique chemical composition of high inorganic (95% of calcium carbonate in the form of calcite) and 5% of organic (type X collagen, sulfated polysaccharides) components; this eggshell characteristic structure combined with substantial availability makes eggshells a potential source of bio-fillers that can be efficiently used for polymer composites [28].

Nevertheless, PLA/PBAT blends filled with calcium carbonate (CaCO₃) present poor mechanical properties due to a poor interfacial adhesion, considering different glass transition exhibited for PLA (56 °C) and PBAT (-34 °C). In order to deal with this

problem, it was used irradiation in order to improve the compatibility between immiscible polymers in the blend. Irradiation leads to changes not only in the interphase but also in the bulk of both polymers (chain scission, crosslinking, etc.). Herein it was used as compatibilizing agent PLA previously gamma and e-beam radiated at 150 kGy, air environment, 10.5 kGy.h⁻¹. Güven and collaborators have proposed the use of ionizing radiation instead of chemical compatibilizing agents for thermoplastic materials with enhanced properties [29 - 34].

2. MATERIALS

The main characteristics of PLA and PBAT are described in Table 1.

- PLA e-beam radiated at 150 kGy: compatibilizing agent, from PLA e-beam radiated at 150 kGy, dose ratio 10.00 kGy/s, Dynamitron II electrons accelerator, 1.5 MeV energy, 25 mA current, 37.5 kw power, in CTR-IPEN/CNEN-SP.
- PLA gamma radiated, ⁶⁰Co source, 150 kGy, in Multipurpose reactor, in CETER/IPEN.
- CaCO₃ (calcium carbonate) from avian eggshells: white chicken eggshells were subjected to a thorough cleaning using tap water for removing of internal membranes. Afterwards, clean eggshells were kept for 4 hours in a 100 °C water bath and finally dried at 100± 2°C for 2 h in an air-circulating oven. Eggshells were size reduced to fine powder, particle size equal to 125 µm, by using ball mills and granulometric sieve, respectively.

2.1. Samples Preparation

Composite materials were prepared according to Table 2; they were first homogeneized by melting extrusion process, using a co-rotating twin-screw extruder (HAAKE Rheomex 332p, 3.1 L/D, 19/33

Table 1: Main Characteristics of Used Polymers

Characteristics of PLA	Characteristics of PBAT
Grade: Ingeo Biopolymer 3251 D	Commercial name: Ecoflex FS
Supplier: Nature Works	Supplier: BASF
Melting Point: 168 °C	Melting Point: 110 – 120 °C
Glass transition temperature: 62 °C	Glass transition temperature: -30°C
Average molecular weight: 100,000 g.mol ⁻¹	Average molecular weight: 40,000 g.mol ⁻¹

Both PLA and PBAT were dried at 70 °C for 12 hours before processing.

Table 2: Designation of Studied Materials and Compositions

Designation	PBAT (wt%)	PLA (wt%)	Bio- CaCO ₃ 125 µm (phr)	PLA 150 kGy Gamma (phr)	PLA 150 kGy Eb (phr)
PBAT	100.0	--	--	--	--
PBAT50	50.0	50.0	--	--	--
BCCM 1	50.0	50.0	15	5	--
BCCM 2	50.0	50.0	15	--	5
PLA	--	100.0	--	--	--

compression ratio), by using a 120 to 145°C temperature profile and 50 rpm.

3. METHODS

Analyses were performed in triplicate for results achievement and characterized by following methods:

3.1. Differential Scanning Calorimetric Analyses (DSC)

Thermal behavior was examined in a DSC-50 Shimadzu apparatus, according to ASTM D3418-08. Heating was carried out from 30 °C to 270 °C, nitrogen atmosphere, N₂ flow rate = 50 ml/min and temperature rate = 30 °C/min. DSC is a thermal analysis technique capable to measure the temperature and heat flow associated with transitions in materials as a function of temperature and time.

3.2. Thermogravimetric Analyses (TG)

Thermogravimetric analyses provide 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 were performed using two apparatus: a TGA Shimadzu, model 51 and a TGA Mettler Toledo, according to ASTM E1641-07, by using 10 – 20 mg of sample, within a 25 to 600°C program, at a 20°C.min⁻¹, in a nitrogen flow of 50ml.min⁻¹.

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

FTIR is a sensible method for identifying chemical modification in a material, and so, is capable to detect

chemical modifications in a polymeric material. This method detects vibrational movements imparted from chemical bonds for the material that is being analyzed. As each chemical group absorbs vibrational energy at a given value, it is possible to differentiate them via infrared spectrum. Spectra were obtained from a Perkin-Elmer, Universal ATR Sampling Accessory – Spectrum 100 FT – IR Spectrometer. Setup collection sample was adjusted for 64 scans, within a 4,000 to 650 cm⁻¹ range.

3.4. Wide Angle X-Ray Diffraction Analysis (XRD)

X-rays diffraction is a technique used for determining anatomic structure: it consists on a constructive interference of a wave from X-rays incident beam in relation to an uniform atomic spacing.

In this technique it is applied Bragg's Law, defined by $n\lambda = 2d\sin\theta$ where $n\lambda$ is an entire value for wavelength generated by an specific target according to a give electronic transition and $\sin\theta$ is the angle where it occurs the constructive interference; therefore, it is possible to determinate interplanar distances (d) for each crystalline plane. The identification of crystalline phase of a material is given from a database defined by JCPDS (Joint Committee on Powder Diffraction Standards) that compares position of obtained peaks with intensity relationship.

It was used herein a X-Ray diffractometer, Rigaku Multiflex, graphite monochromator, 40 kV, 20 mA, X-rays tube, copper anode $\lambda_{Cu_{ka}} = 1,5418\text{\AA}$, scanning 2θ within 3 ° to 60 °, speed 0.06°/4 seconds, fixed time. It provides, among other, information on samples crystallinity, via diffractograms, distinguishing between amorphous and crystalline state.

3.5. Tensile Strength and Elongation at Break

Tensile and elongation at break essay is a relevant instrument for evaluating loss of properties and evolution of degradative process of the polymer.

Parameters that contribute for mechanical behavior of polymers are: chemical structure, crystallinity degree, molar mass, moisture, reinforcing agent present, among others. All these properties are modified during degradation processes. In case of reinforcing agents, the concentration is not changed; nevertheless, their interaction can be modified in consequence of chemical modifications suffered by the polymer. Tensile and elongation at break tests were accomplished at $25 \pm 5^\circ\text{C}$, in an EMIC model DL 300 universal essay machine, 20 kN load cell, $50 \text{ mm} \cdot \text{min}^{-1}$, in accordance with ASTM D 638-14. Specimens were conditioned at $25 \pm 5^\circ\text{C}$ and $50 \pm 5\%$ relative humidity, for 24 hours, prior test.

4. RESULTS AND DISCUSSION

4.1. Differential Scanning Calorimetric Analyses (DSC)

DSC heating curves for analyzed materials shown in Table 3 are shown in Figures 1 and 2:

Table 3: Thermal Characteristics for Samples and Constituents

	PEAK ($^\circ\text{C}$)	FUSION ENTHALPY (J/g)
PLA	158.9	22.34
PBAT	160.8	3.07
PBAT50	157.0	13.53
PLA 150 kGy eb	103.6	58.63
PLA 150 kGy g	101.5	66.21
BCCM 1	152.39	1.26
BCCM 2	152.13	2.01

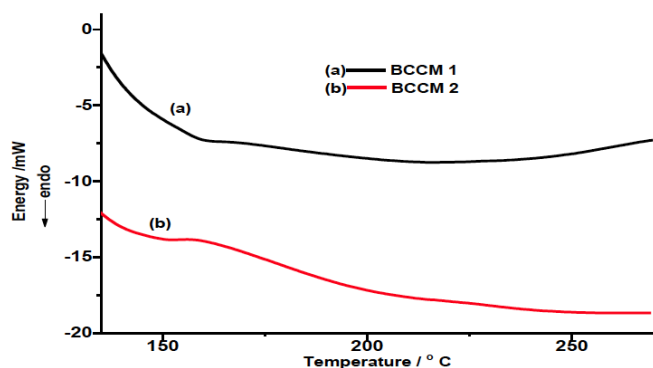


Figure 1: Melting curves for analyzed samples.

In Table 3 it is listed a summary of thermal characteristics for samples and constituents.

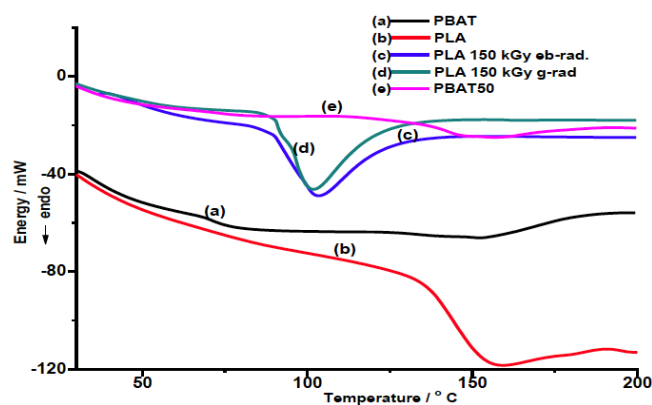


Figure 2: Melting curves for constituents.

Values presented in Table 3 for Peak and Fusion Enthalpy ratify curves presented in Figures 1 and 2.

Melting curves shown for BCCM1 and BCCM 2 samples presented very close results, emphasizing for BCCM 2 a value slightly higher.

Both PLA 150 kGy eb and PLA 150 kGy g (compatibilizing agents) presented well defined peaks, as expected for irradiated materials.

DSC curves found for samples constituents, in Figure 2, were expected, according to literature values.

4.2. Thermogravimetric Analyses (TG)

In Figures 3 and 4 are shown obtained curves for samples and constituents, respectively.

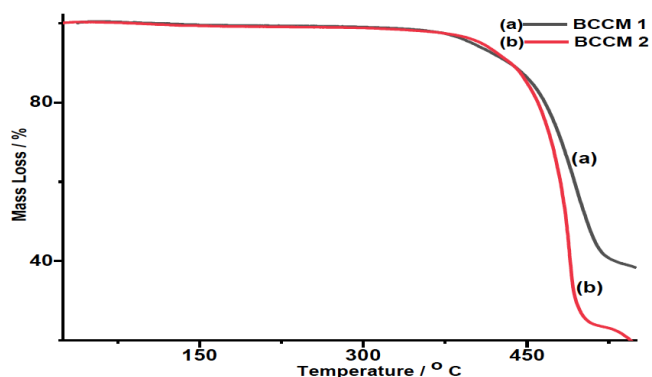


Figure 3: TG curves for samples.

In Table 4 it is listed a summary of thermal characteristics for samples and constituents.

Both BCCM 1 and BCCM 2 samples presented very close results for peak temperature as well for mass loss.

Samples constituents presented identical behavior for PBAT, PLA and PBAT50.

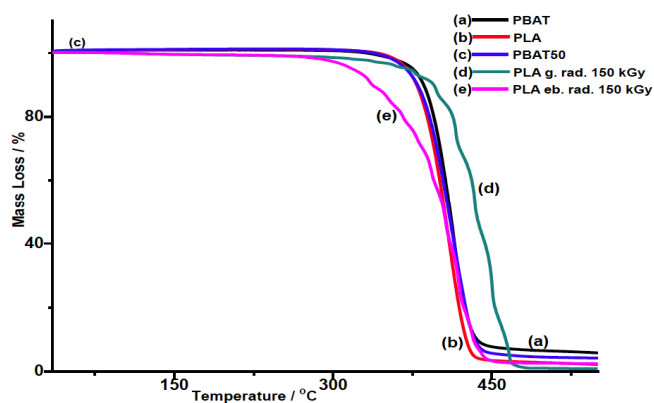


Figure 4: TG curves for constituents.

Table 4: Thermal Characteristics for Samples and their Constituents

	PEAK (° C)	Weight Loss (%)
PLA	380.80	89.33
PBAT	390.10	91.39
PBAT50	380.4	96.69
PLA 150 kGy, eb	450.2	95.91
PLA 150 kGy, g	475.1	99.80
BCCM 1	450.1	90,01
BCCM 2	474.8	89,90

PLA 150 gamma radiated presented a higher thermal stability when compared to PLA e-beam radiated [35].

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

FTIR spectra for samples and their constituents are shown in Figures 5 and 6.

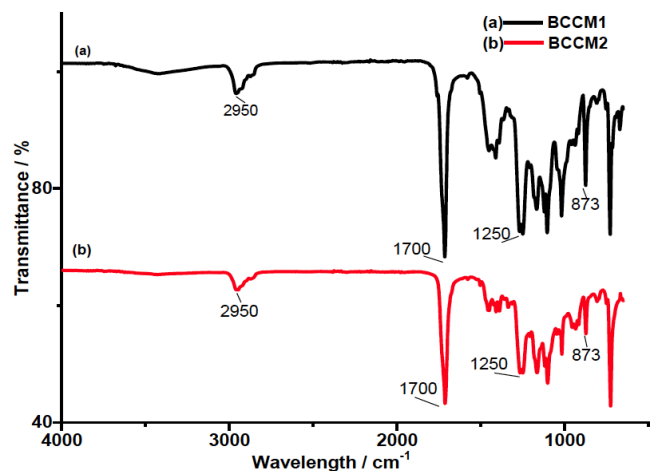


Figure 5: Infra-red spectra for samples: BCCM1 and BCCM2.

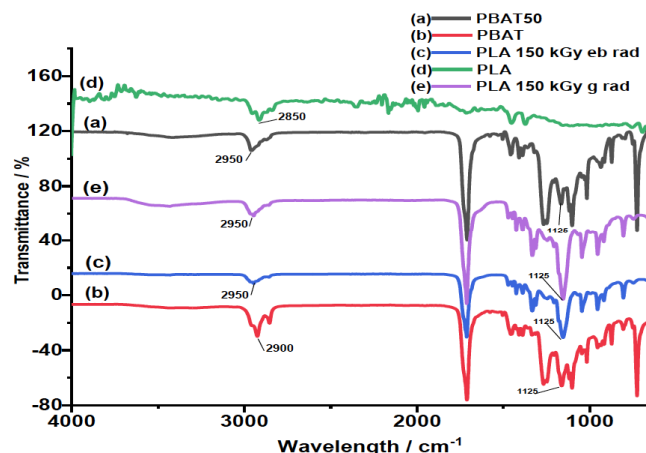


Figure 6: Infra-red spectra for constituents.

FTIR spectra for samples and their constituents, in Figures 5 and 6 were expected according to literature values.

In Figure 5, absorption peaks at 2950 and 1700 cm^{-1} were due to the stretching and bending vibrations of C-H bond. C-O stretching peak was observed at 1250 cm^{-1} . The peak at 873 cm^{-1} was assigned to carbonate of micro particles. It is evident the higher intensity peaks registered for BCCM-1 sample.

In Figure 6, IR absorption peaks at 2850, 2900, 2950 and 1700 cm^{-1} were due to the stretching and bending vibrations of C-H bond. C-O stretching peak was observed at 1125 cm^{-1} .

4.4. Wide Angle X-Ray Diffraction Analysis (XRD)

Wide angle X-ray diffraction pattern for samples and their constituents is given in Figures 7 and 8, respectively.

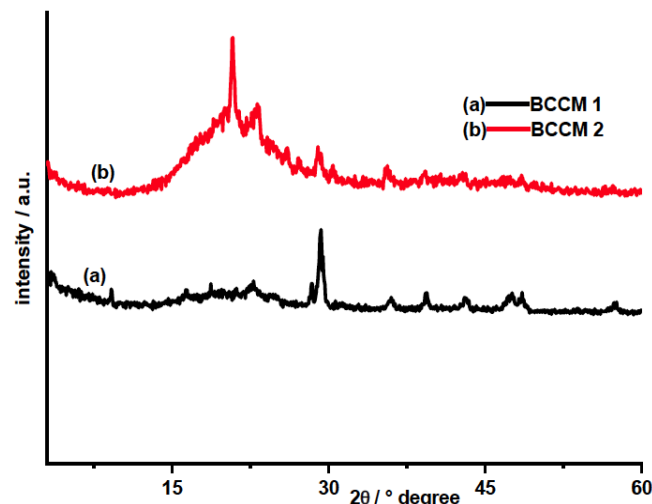


Figure 7: DRX diffractograms for samples

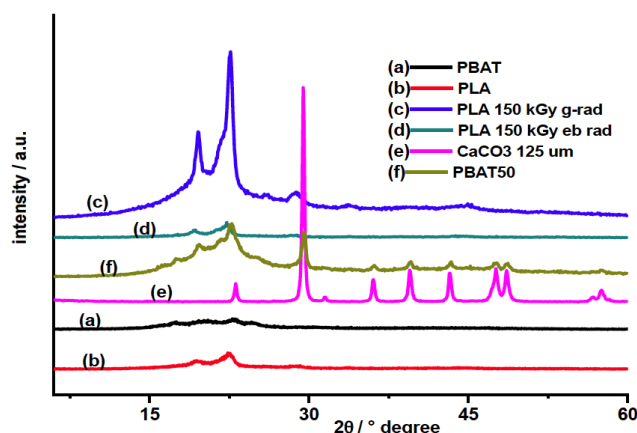


Figure 8: DRX diffractograms for constituents.

Diffractograms presented by samples, in Figure 7, indicated for BCCM 1 and BCCM 2, different interaction with constituents: the presence of 2 θ peak at 29.7° confirmed that micro CaCO_3 was incorporated into the matrix (BCCM-1) and is from calcite type.

In Figure 8, diffractogram referring to PLA compatibilizer gamma radiated showed a better peaks development and intensity when compared to e-beam radiated compatibilizer, that will contribute for further effective results in mechanical essays [36].

4.5. Tensile Strength and Elongation at Break

Tensile Strength and Elongation at Break for samples and their constituents are presented, respectively, in Figures 9 and 10.

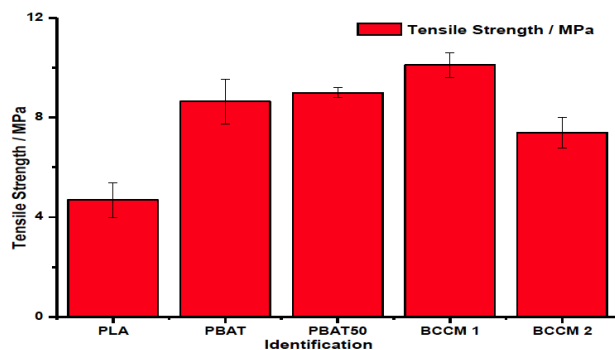


Figure 9: Tensile Strength for samples and constituents.

Mechanical properties of composites depend to a greater extent upon the uniform distribution of filler in polymer matrix and interfacial adhesion between filler and matrix.

Herein, BCCM 1 sample presented a higher Tensile Strength when compared to PBAT50 (PBAT/PLA 50/50), pointing toward the effectiveness of reinforcing agent associated to PLA gamma radiated 150 kGy

used as compatibilizer. Elongation at break results for both BCCM 1 and BCCM 2 were approximately 65% lower when compared to PBAT50.

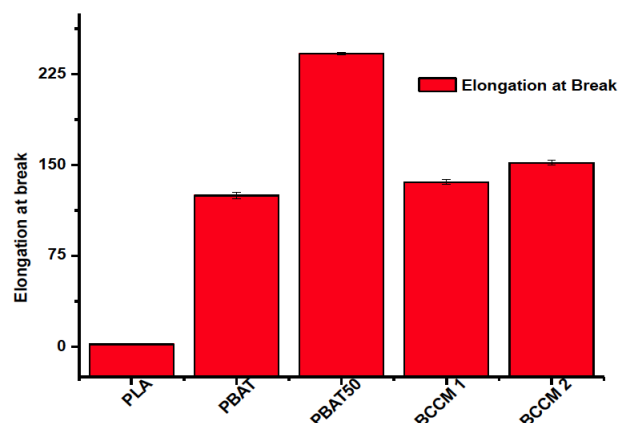


Figure 10: Elongation at break for samples and constituents.

CONCLUSIONS

In this paper, there were studied two compatibilizing agents for providing an effective interaction between immiscible PLA/PBAT blend reinforced with eggshell (bio-calcium carbonate): PLA e-beam and PLA gamma radiated. Evaluation comprised DSC, TG, ATR-FTIR, XRD, Tensile Strength and Elongation at Break. Melting curves (DSC) for both evaluated samples did not present significant differences as well their constituents. Thermogravimetric analyses pointed toward a negligible difference for both evaluated samples as well for their constituents, except for gamma radiated PLA that exhibited a higher thermal stability when compared to e-beam radiated PLA. Results for ATR-FTIR were expected according to literature values, emphasizing for PLA gamma radiated slightly higher intense peaks when compared to PLA e-beam radiated at 1700 and 1125 cm^{-1} , corresponding respectively to C-H bond and C-O stretching peak. XRD investigation pointed toward more defined and intense peaks for PLA gamma radiated in comparison with PLA e-beam radiated, predicting effective results in mechanical essays and corroborated by tensile strength shown by PBAT/PLA 50/50 containing 15 phr of bio-calcium carbonate $125\text{ }\mu\text{m}$ and 5 phr of PLA 150 kGy gamma radiated. Finally, PLA e-beam radiated at 150 kGy was not effective to act for compatibilizing in PBAT/PLA 50/50 reinforced with bio-calcium carbonate; efficacy of compatibilizing action was proved for PLA gamma radiated at 150 kGy.

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