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## Recovery of cotton fibers from waste Blue-Jeans and its use in polyester concrete



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### HIGHLIGHTS

- Polyester concrete with waste cotton fibers from Blue-Jeans was elaborated.
- The effects of gamma radiation on compressive and flexural properties were studied.
- The highest mechanical performance is obtained with 1 wt% of waste cotton fibers.
- Irradiation dose of 300 kGy provides the highest mechanical values.
- Mechanical improvements were related with SEM, FT-IR and XRD analyzes.

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### ABSTRACT

Currently, the consumer tendency causes that the garments are dismissed more quickly, which generate increment of textile waste, such as Blue-Jeans. In this work, polyester concrete with waste cotton fibers was elaborated, and a novel treatment by gamma irradiation was carried out. The results show up to 40% improvement on the compressive strength, as well as 7% on the flexural strength. Additional improvements for irradiated concrete were obtained, when 300 kGy of irradiation dose was applied. Modifications on the surface, chemical structural and crystallinity of irradiated waste cotton fibers, were related with improvements on the mechanical properties of concrete.

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### 1. Introduction

Environmental pollution is not a topic for the future, but one that we currently have, which having caused serious damages to our ecosystems, some of them irreversible. Many investigations are increasingly on designing strategies to remedy damages, through waste reduction, and recycling and reuse of materials. Waste production is accelerating, it is estimated that in developed

countries each person produces 2 kg of solid waste daily on average, and in the case of Latin-American countries, 1 kg daily [1].

As we known industrial activities provoke environmental impact as those developed by textile industry, which is one of the most developed in the world. Fortunately, great demand on the use of natural textile fibers is happen, mainly due to current fashion, which is governed for the use of comfortable, light and skin-friendly clothing. Blue-Jeans (Denim), are an example of such clothing. One-third of worldwide production of cotton corresponding to cotton fibers, which are used in the textile industry; mainly for manufacturing of Denim garments. Worldwide cotton production

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in 2014 was 25.8 million tons and an annual growth of 2.1% in the next 10 years is expected [2].

Final disposal of textile waste is causing soil contamination, canal obstructions and drainage systems. Moreover, water pollution is generating by production and manufacturing of clothes; mainly through the dyeing, process which using high content of substances such as sulfur, naphthol, soaps, enzymes and dyes [3]. Some chemical products used in the manufacturing, sometimes are able to react with disinfectants, such as chlorine, and produce compounds with carcinogenic properties. In an investigation, the salinity and alkalinity of soils were severely affected, by textile industry water, and long-term effects on the low crop yields were observed [4].

The use of natural fibers such as jute, flax, coconut fiber, henequen and cotton, as reinforcements in building materials, is arousing great scientific interest; due to the improvements on the mechanical properties of them, sustained on the fiber characteristics as low environmental impact and cost. For example, polymer concrete (mixture of a thermoset resin and mineral aggregates), is three to five times more resistant than hydraulic concrete, but this shows fragility at the failure point, limiting its use when large loads are applied. Thus, for solving such problem the use natural fibers as reinforcing materials has been proposed [5,6].

The composite material elaborates with epoxy resin, jute fiber and palm oil; having a jute/palm oil ratio of 1:4, 1:1 and 4:1, as a novel material with application in the construction, aeronautic and automotive industries was proposed [7]. The results show higher electrical voltage and Young's modulus values, for higher jute fiber concentration. Other investigation is concerning on films of a commercial polyurethane and cellulose nanocrystals; the effect of the latter on the mechanical, thermal, morphological and water absorption properties of the composite was carried out. The results show well dispersion of the cellulose nanocrystals in the polymer matrix, which are able to act as nucleating agents and as mechanical reinforcements. Moreover, increment of Young's modulus and tensile strength are observed [8].

Plastic Wood Concretes (PWC) are a new class of materials that combine the characteristics of plastic and wood; but with the drawback of a high moisture absorption. Polymer concrete elaborated with a polymer matrix, mineral aggregates and wood, showed improvement on the rigidity property for higher concentrations of wood (up to a maximum of 55 wt%). Moreover, the addition of minerals generated higher strength and reduction in moisture absorption [9].

Mechanical behavior of concrete elaborated with epoxy resin, sand and recycled textile fibers (cotton, polyester, silk and rayon), was investigated [10]. It is important to mention that cotton contains a high content of cellulose fibers. Textile fiber concentrations were 1 and 2 wt%. Specimens had a resin/sand ratio of 10/90 for evaluating flexural strength behavior, as well as a ratio of 12/88 for evaluating compressive strength. The results show diminution on the mechanical properties for higher concentrations of textile fibers; nevertheless, mechanical failure occurs less violently.

Mechanical performance of an elaborate composite with polyester resin as matrix, and peanut shells as reinforcing materials was studied. Concentrations of 5, 10, 15 and 20 wt% peanut shells were used. The results show the highest flexural strength values (80 MPa), for a concentration of 15 wt% of peanut shells [11].

Polymer concrete elaborated with unsaturated polyester resin and solid residues, obtained from the extraction process of olive oil, were studied [12]. Concentration of the residues were from 10 to 60 wt%. A silanized process (based in the use of mercaptopropyltrimethoxysilane), was used for improving of the resin-residues interface. The results show highest flexural strength (50 MPa), for concrete with 30 wt% of residues treated with silane, in comparison with those that used untreated residues, namely 44

MPa. Reduction of the water absorption by the fibers was caused by the silane application, and in consequence improvement of the interface.

Improvement of the compatibility between fibers and polymer matrix is very important, due to nature of both: a hydrophobic matrix and hydrophilic fibers. Moreover, in a fiber reinforced composite with optimal mechanical performance, the interfacial bond between the polymer matrix and the fibers must be optimized, to achieve effective transfer of stresses between the two phases. The correct compatibility between fibers and matrix is achieved from polymer chains that will encourage entanglements and inter-diffusion with the matrix [13].

Physical and chemical treatments have been developed for compatibility improvement, some of them are environmentally friendly, in particular those based on interaction of ionizing radiation with matter, as gamma irradiation is [14,15]. Such kind of energy acting over polymers provoke cross-linking and scission of polymer chains, as well as formation of new compounds. Rupture of chemical bonds cause changes in the chemical structure, which modifies the crystallinity [16,17].

This work proposes the use of waste cotton fibers (obtained from Blue-Jeans), as reinforcing materials in polymer concrete elaborated with polyester resin and marble particles; as well as use of gamma irradiation for improvement of the mechanical features of concrete. Both proposal for solving in some measure the environmental problem generated by textile wastes.

## 2. Experimental

### 2.1. Preparation of waste cotton fibers

Cotton fibers were obtained from waste Blue-Jeans (Called Waste Denim), labeled by manufacturer as 100% cotton. Waste Denim was cut in square pieces of 1 x 1 cm (Fig. 1a), and then they were reduced in size, by using a knife mill, trough to 4 cycles of 15 s each one (Fig. 1b).

The concentration of each component of the waste cotton fibers are shown in the Table 1, where the main component corresponds to cellulose; which can vary depending on provenance and climatological conditions during its cultivation [18].

The mechanical properties of waste cotton fibers in the Table 2, are shown.

### 2.2. Preparation of polyester based composite

In a first stage of the experiments, composite materials were elaborated with marble particles and unsaturated polyester resin (Poliformas Plásticas, PP-70X60). The concentration of polyester resin was from 20 to 40 wt%, this was pre-accelerated with copper octoate, and for its polymerization methyl ethyl ketone peroxide (MEKP), was used as catalyst. We called such concrete as Control composite (i.e. concrete without textile fibers).

In a second stage, waste cotton fibers were added to the concrete, in concentrations of 0.5, 1.0 and 1.4 wt%. Finally, in a third stage, concrete with waste cotton fibers was irradiated with gamma rays, covering dosages 100 to 900 kGy.

### 2.3. Compressive and flexural strength

Compressive and flexural strength tests for the concrete specimens were carried out according to EN-196-1 standard test, in a Universal testing machine model 70-S17C2 (Controls™, Cernusco, Italy). Three-point-flexural test with a distance between supports of 10 cm, was carried out.

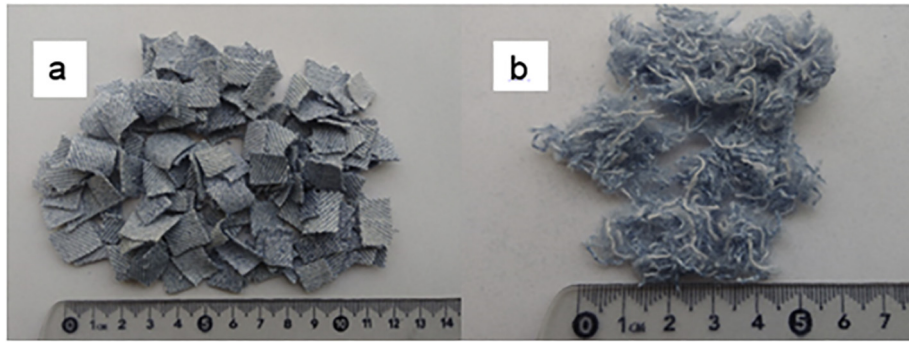


Fig. 1. a) Square pieces of waste Blue-Jeans, and b) Cotton fibers from Blue-Jeans.

**Table 1**  
Composition of each waste cotton fiber component [18].

Component	Typical composition (wt.%)	Waste composition (wt.%)
Cellulose	95.0	88.0–96.0
Protein	1.3	1.1–1.9
Ash	1.2	0.7–1.6
Peptic substances	0.9	0.7–1.2
Organic acids	0.8	0.5–1.0
Wax	0.6	0.4–1.0
Total sugars	0.2	0.1–1.0

**Table 2**  
Mechanical properties of waste cotton fibers [19].

Property	Value
Density, g/cm <sup>3</sup>	1.5–1.6
Elongation, %	7.0–8.0
Tensile strength, MPa	287–597
Young's modulus, GPa	5.5–12.6

#### 2.4. Morphological characterization of waste cotton fibers

Waste cotton fiber surfaces were analyzed by scanning electron microscopy (SEM), in a JEOL model JSM-5900LV in the secondary electron mode, at 20 keV.

#### 2.5. X-ray diffraction

The X-ray diffraction patterns of non-irradiated and irradiated waste cotton fibers were obtained in a BrukerD8 Advance diffractometer, operated at 35 kV. The diffraction pattern was obtained by scanning the sample in an interval angle ( $2\theta$ ) from 10° to 30°, and a rate of 0.5°/min.

#### 2.6. FT-IR spectrophotometry

The FT-IR spectra of non-irradiated and irradiated waste cotton fibers were obtained in a spectrometry prestige 21, with a HART diamond accessory. Samples were read in the infrared interval, from 4500 cm<sup>-1</sup> to 550 cm<sup>-1</sup>, with a resolution of 8 cm<sup>-1</sup> and 32 scan.

#### 2.7. Irradiation procedure

Waste cotton fibers (placed in a capillarity tube), and the concrete specimens were exposed at different gamma irradiation dosages (100–900 kGy), at dose rate of 3.5 kGy/h, in air at room temperature. The irradiation was provided by using a Transelektro irradiator LGI-01 provided with a <sup>60</sup>Co source, manufactured by

IZOTOP Institute of Isotopes Co. Ltd., Budapest, Hungary; and located at the Instituto Nacional de Investigaciones Nucleares Mexico (ININ).

### 3. Results and discussion

#### 3.1. Compression behavior of polymer concrete without textile fibers

In a first stage of the experiments, polymer concrete specimens were prepared varying polyester resin concentration (20 wt%–40 wt%), for to obtain those with highest compressive values. In Fig. 2, the compressive strength and compressive strain results are shown. Respect to compressive strength, the values are increasing when the resin concentration increase. A value of 83 MPa was obtained for composite with 20% of resin, while for those with 40% of resin was 113 MPa, which means 40% of increment. For a composite having lower concentration of marble powder and higher concentration of resin, better contact is done between such components, showing more homogeneity, and consequently a strength composite is obtained.

Compressive strain values show two well-defined stages, in the first one, a constant value is observed for concrete with 20% and 30% of resin; while in the second an improvement of 22% is observed for concrete with 40% of resin, such increment is due to the flexural behavior of the resin.

Young's modulus results are shown in Fig. 3; the maximum value, 2.74 GPa, was obtained for concrete with 30% of resin; which is a rigid composite (less flexibility); then 30% of resin is the most adequate concentration to cover marble particles and provide greater rigidity to polymer concrete.

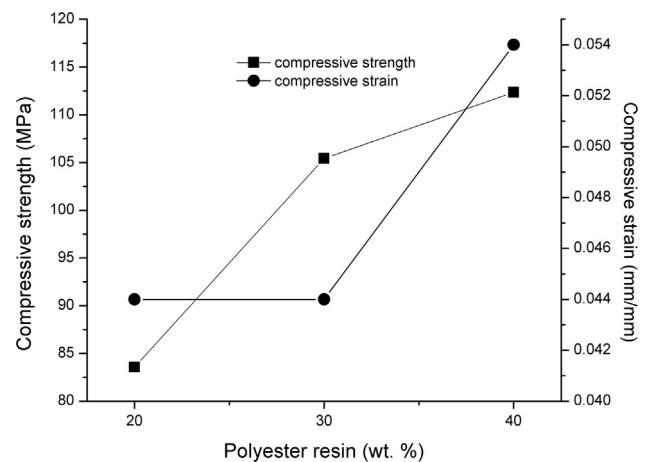


Fig. 2. Compressive strength and strain of polymer concrete.

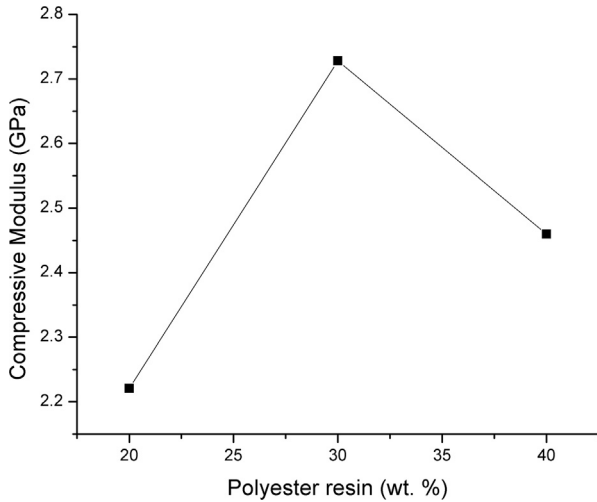


Fig. 3. Compressive modulus of polymer concrete.

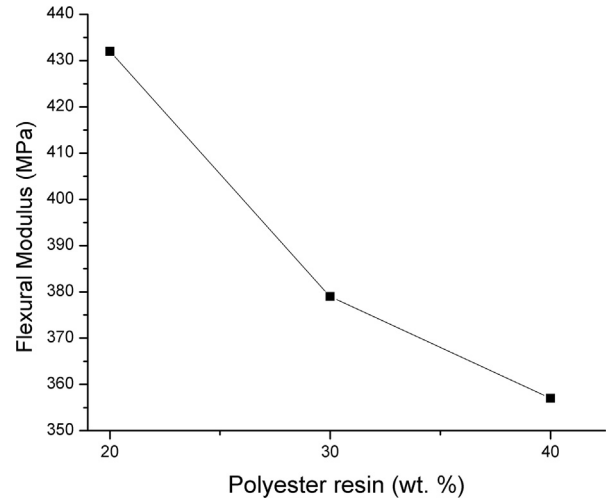


Fig. 5. Flexural modulus of polymer concrete.

3.2. Flexural behavior of polymer concrete without textile fibers

Flexural strength results of polymer concrete are shown in Fig. 4; the values increase according to resin concentration increase too. The maximum value, 7.8 MPa, was obtained for concrete with 40% of resin; more concentration of polyester resin provides more flexibility to polymer concrete; moreover, a greater contact between marble particles and the polyester resin is done.

Flexural strain values have similar behavior that those for flexural strength: they increase when polyester resin concentration increase. The maximum value was obtained for concrete with 40% of resin. Then, major flexibility in the polymer concrete is obtained when marble particles concentration diminishes.

In Fig. 5, it is corroborated higher flexibility of polymer concrete for higher concentrations of the polyester resin. The values are ranging from 360 MPa to 432 MPa, which means 20% of difference between them.

According to previous results, minimal differences on the mechanical properties were found for concrete with 30 and 40% of resin (6% of difference for compressive and 9% for flexural behavior). Thus, it was deciding for the second stage of the experiments to elaborate polymer concrete with 30% of polyester resin, different concentrations of marble particles and waste cotton

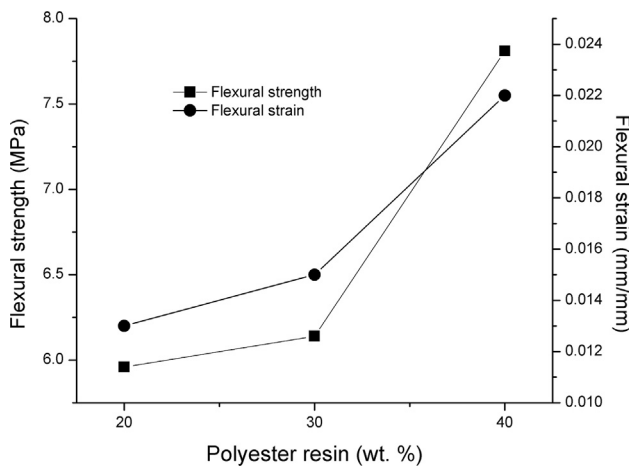


Fig. 4. Flexural strength and strain of polymer concrete.

fibers. Such polyester resin concentration was choosing for saving production costs.

3.3. Compression behavior of polymer concrete with waste cotton fibers

In Fig. 6 compressive strength and strain values of polymer concrete with fibers are shown. For compressive strength the values decrease when the concentration of the waste cotton fibers increases; being until 8% lower than that for control concrete (without fibers); while for compressive strain, an increment of 7% was obtained. In general, addition of waste cotton fibers generates lower resistance and higher deformation of polymer concrete. Such results can be attributed to a poor interface between polymer matrix and the cotton fibers. In general, modifications of the surfaces and moisture of textile fibers can improve such interface.

Addition of the fibers generate higher Young's modulus values, as it can be seen in Fig. 7. The highest value was obtained for polymer concrete with 1.0 wt% of waste cotton fibers, it means 12% improvement. In general, addition of waste cotton fibers generates lower compressive strength; caused by the poor interface between fibers and polymer matrix. Nevertheless, Young's modulus is increasing.

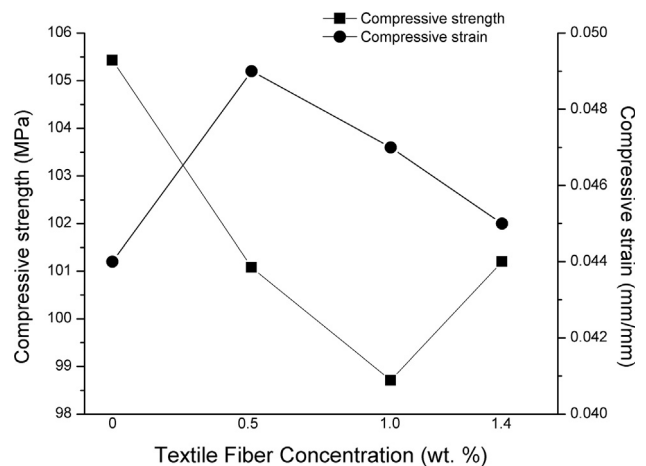


Fig. 6. Compressive strength and strain of polymer concrete with waste cotton fibers.

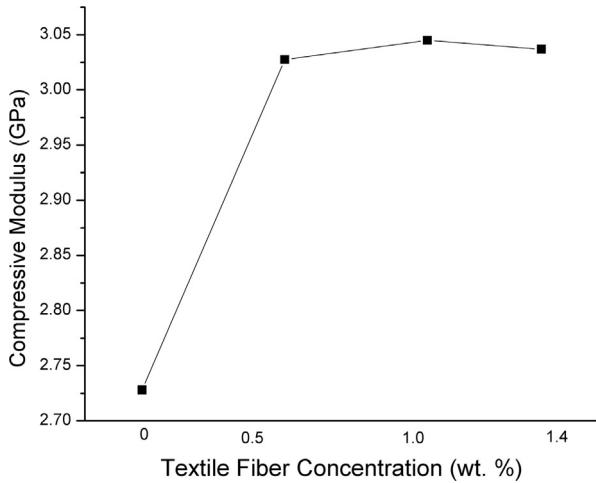


Fig. 7. Compressive modulus of polymer concrete with cotton fibers.

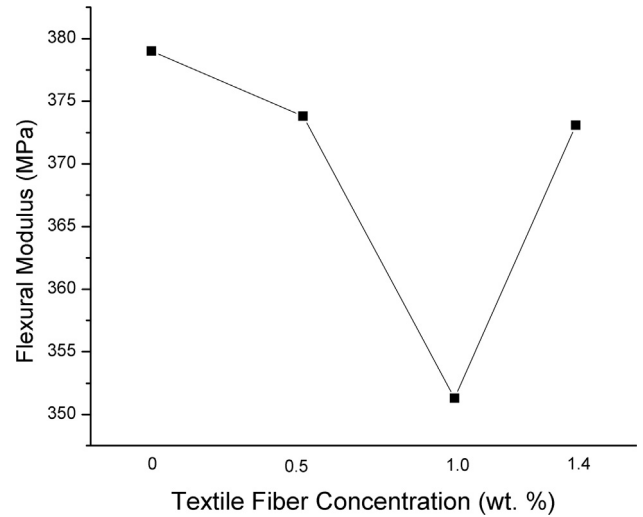


Fig. 9. Flexural modulus of polymer concrete with waste cotton fibers.

3.4. Flexural behavior of polymer concrete with waste cotton fibers

Flexural strength behavior of polymer concrete with fibers is shown in Fig. 8. It can be observed an increment of 10% for polymer concrete with 1.0% of fibers respect to control concrete (without fibers), such difference indicates an opposite behavior than those for compressive strength, where the values decrease.

In the case of flexural strain, an improvement of 26% is obtained for composite with 1.0% of fibers. This difference suggests that the dispersed fibers into the polymer concrete provide higher deformation, as consequence of their flexural characteristics.

Respect to flexural modulus values, as it can be observed in Fig. 9, lowest value is obtained for polymer concrete with 1.0% of fibers, this means 7% lower than that for control concrete. Then, polymer concrete is more flexible when using 1% of the waste cotton fibers.

The better compressive and flexural results were found for concrete with 1 wt% of waste cotton fibers. Thus, for the third experimental stage, we decide elaborate polymer concrete with 1% of waste cotton fibers and irradiate them at different gamma irradiation dosages (from 100 kGy to 900 kGy). In this stage, the main idea was improving the interface characteristics between polymer matrix and the waste cotton fibers, through modifications of them by using gamma irradiation, and in consequence, improvement on the mechanical properties of polymer concrete.

3.5. Compression behavior of irradiated polymer concrete with waste cotton fibers

Compressive strength and strain of irradiated polymer concrete elaborated with 30 wt% of polyester resin, 69 wt% of marble, and 1 wt% of waste cotton fibers, are shown in Fig. 10. An improvement of 37% in compressive strength was obtained for polymer concrete irradiated at 300 kGy, respect to that for control concrete (not irradiated one). For higher irradiation dosages, compressive strength values are almost constant. Compressive strength increments are due to physical and chemical modification of the fibers and resin, caused by gamma irradiation; mainly surface and crystallinity modifications of the waste cotton fibers, which generate improvement on the interface fibers-polymer matrix.

Respect to compressive strain, a maximum increment of 15% was achieved for polymer concrete irradiated at 100 kGy; however, for higher irradiation dosages the values notably diminish. This corroborate that ionizing radiation generate a rigid polymer concrete.

Compressive properties of irradiated polymer concrete can be related with morphological changes of irradiated waste cotton fibers, as it is shown in Fig. 11. For non-irradiated cotton fibers smooth and not rough zones are observed, fibers have smaller sizes than 50 μm. When they are irradiated, surface morphology

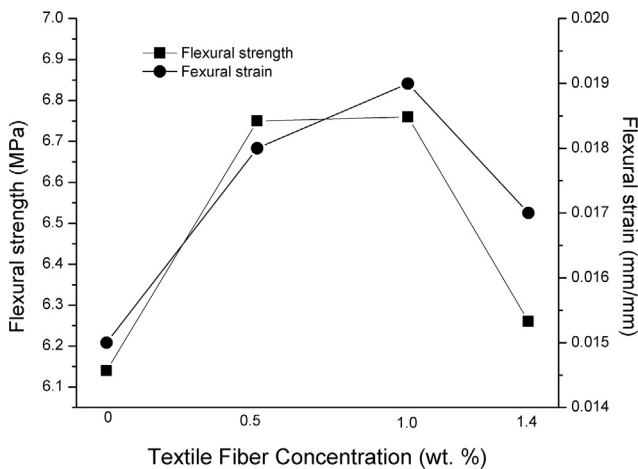


Fig. 8. Flexural strength and strain of polymer concrete with waste cotton fibers.

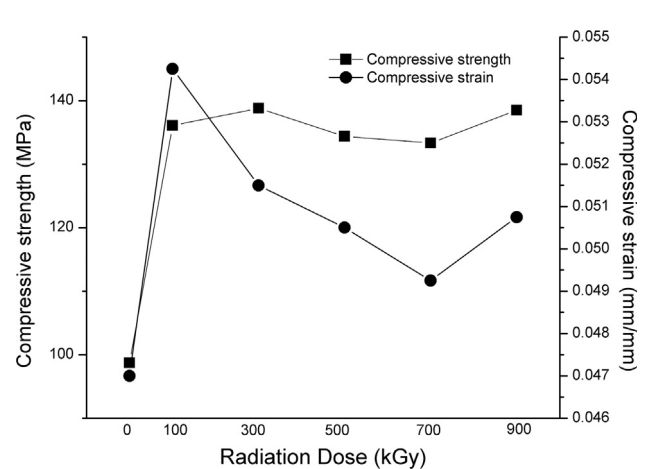


Fig. 10. Compressive strength and strain of polymer concrete with waste cotton.

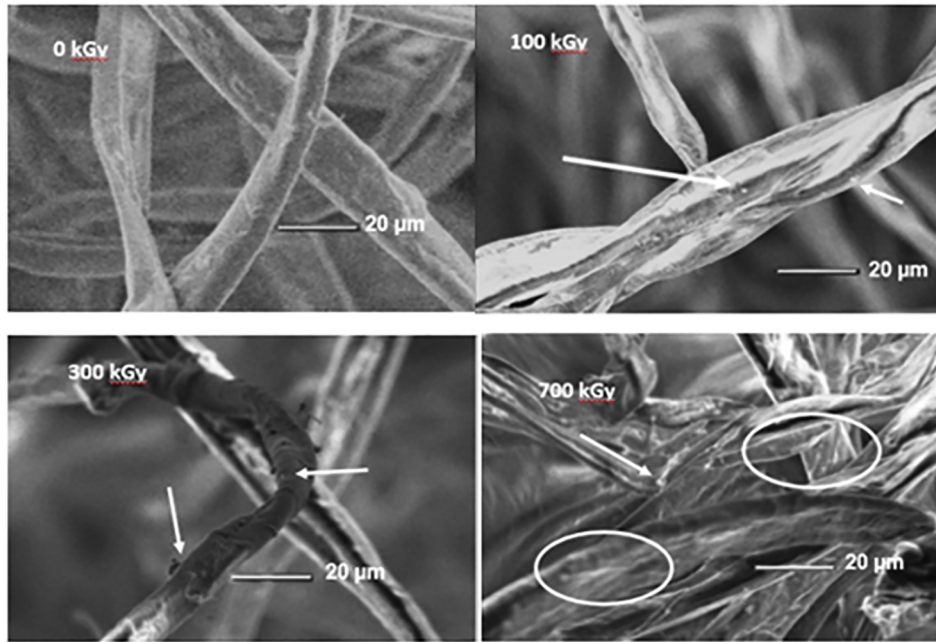


Fig. 11. SEM images of non-irradiated and irradiated waste cotton fibers, irradiated at different dosages.

changes; at 100 kGy, presence of detached particles are observed (indicated by arrows), according to the mechanical properties, such morphology helps to have highest compressive strain values. Appearance of cracks and rougher surface are observed on irradiated fibers at 300 kGy (indicated by arrows and circles), cracks promote also higher compressive strength. Finally, for higher irradiation dose, 700 kGy, more notable is the presence of detached particles and much deeper cracks (indicated by circles), these produce detrimental results on the compressive strength and strain. Nevertheless, more cracks on the rougher surface is beneficial for the interaction between waste cotton fibers and the polymer matrix.

In the case of compressive Young's modulus (Fig. 12), it is noted decrease in the values of irradiated polymer concrete. The lowest value is obtained at 500 kGy of irradiation, which means 7% lower than that for non-irradiated one. Then, polymer concrete is more ductile after irradiating with gamma particles. In general, higher

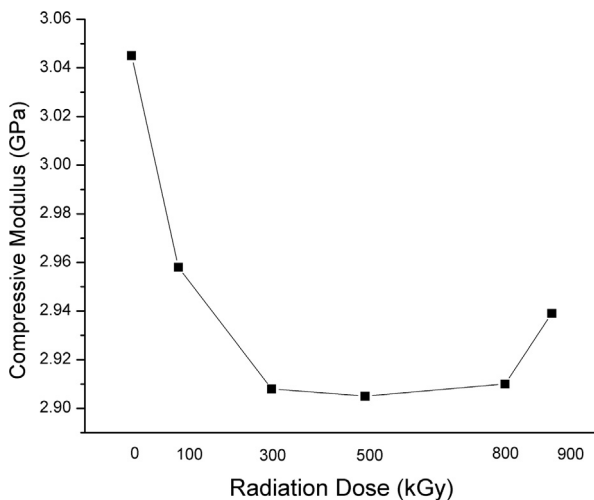


Fig. 12. Compressive modulus of polymer concrete with cotton fibers, irradiated at different dosages.

flexibility caused by gamma irradiation is due to the improvement on the transfer of tensions between the components of polymer concrete. The irradiated cotton fibers cause better interaction with the polymer matrix, due to the change in its structural, morphological and crystallinity properties.

Diminution on the flexural modulus can be related with structural changes of the irradiated waste cotton fibers, observed by FT-infrared spectroscopy; as it is shown in Figs. 13 and 14. The FT-IR spectra of the waste cotton fibers show the presence of two types of cellulose: crystallized I and crystallized II. In the spectrum ranging from  $3600\text{ cm}^{-1}$  to  $2800\text{ cm}^{-1}$  (Fig. 13), two peaks are observed, one at  $3328\text{ cm}^{-1}$  and other at  $3273\text{ cm}^{-1}$ , which corresponding to OH groups in tension of the intramolecular hydrogen bridges in the crystalline cellulose type II; such molecular group is consequence of the chemical treatment with NaOH given to Blue-Jeans (Denim).

According to the nature of cotton fibers, for smaller crystallinity easier is absorbance of water molecules [20], which can be

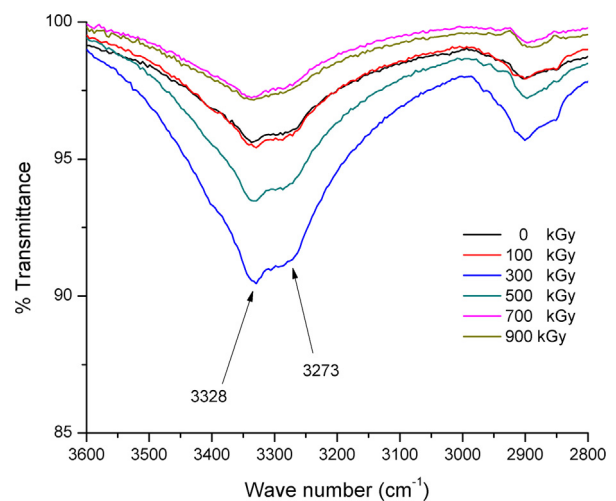


Fig. 13. FT-IR spectra of non- and irradiated waste cotton fibers ( $3600\text{--}2800\text{ cm}^{-1}$ ).

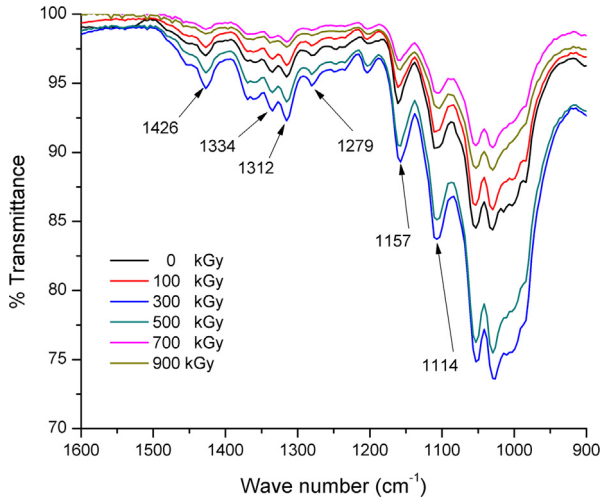


Fig. 14. FT-IR spectra of non- and irradiated waste cotton fibers (1600–900 cm<sup>-1</sup>).

detected by peaks corresponding to the deformation bonds of OH. Respect to irradiation of the fibers, peaks show lowest percentage of transmittance (or higher absorbance), at 300 kGy of dose. Even more, at this dose compressive modulus values are the lowest.

In the spectra ranging from 1600 cm<sup>-1</sup> to 900 cm<sup>-1</sup> (Fig. 14), were detected peaks at 1334, 1312 and 1279 cm<sup>-1</sup> corresponding to the presence of crystallized cellulose II. While cellulose I was evidenced by the peaks at 1426, 1157 and 1114 cm<sup>-1</sup> [21]. Respect to irradiation process, transmittance percentage decrease for irradiated waste cotton fibers, mainly for those irradiated at 300 kGy.

3.6. Flexural behavior of irradiated polymer concrete with waste cotton fibers

Flexural strength results are shown in Fig. 15, where an improvement of 12% is observed when polymer concrete is irradiated at 100 kGy respect to non-irradiated one. As it can be observed, flexural strength values increase for composite irradiated at 100 kGy, but for higher dosages gradual diminution on the values are done.

In the case of flexural strain, a maximum difference of 7% is obtained for polymer concrete irradiated at 100 kGy. Highest values for both flexural strength and strain at 100 kGy can be assumed, due to: a) improvement on the interface polymer

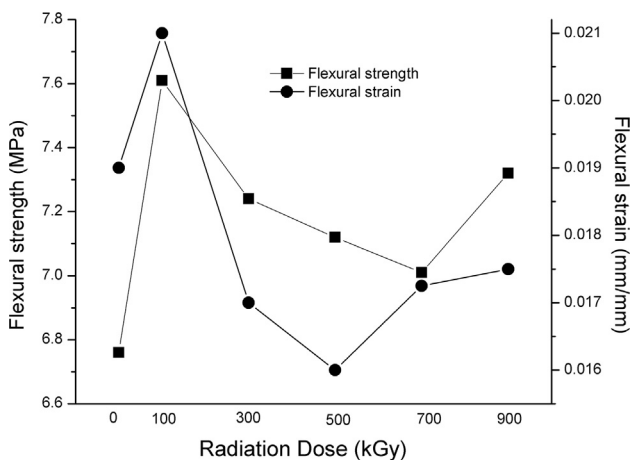


Fig. 15. Flexural strength and strain of polymer concrete with waste cotton fibers, irradiated at different dosages.

matrix-cotton fibers, and b) increment of the degree of polymerization of polyester resin, caused by ionizing radiation.

Fig. 16 illustrates the flexural modulus values; a maximum value of 410 MPa was obtained for polymer concrete irradiated at 300 kGy, which means an improvement of 30% respect to non-irradiated one; or otherwise lowest flexibility of polymer concrete was done. Such behavior is due to the improvement of the polymer/fibers interface, caused by the dispersion of the fibers into the concrete, as well as application of gamma irradiation.

Flexural properties of irradiated polymer concrete can be related with crystallinity of the irradiated waste cotton fibers, as it is shown in Fig. 17. The spectra show three well-defined peaks, at 2θ = 14.6°, 2θ = 16.4° and 2θ = 22.5°. The peak at 14.6° is associated to the plane (1 0 1) of the crystallized cellulose I; while the peak at 16.4° to the plane (1 0 1), but of the crystallized cellulose II. Finally, the peak at 22.5° is associated with the plane (0 0 2) of the crystallized cellulose I.

Crystallinity of the waste cotton fibers can influence on both flexural strain and flexural modulus; highest intensities of crystallinity were obtained for waste cotton fibers irradiated at 300 kGy and the lowest intensities at 900 kGy. The crystallinity index, I<sub>c</sub>, was calculated according to the peaks' intensity, by the formula:

$$I_c = 100 \left( \frac{I_M - I_m}{I_M} \right)$$

Where I<sub>M</sub>, is the maximum intensity of the peaks, in this work located at 2θ = 22.5°; while, I<sub>m</sub>, is the intensity attributed to the amorphous phase, located at 2θ = from 6° to 19°. In Fig. 18, the crystallinity index values are shown. For non-irradiated waste cotton fibers, the crystallinity index was 86%; such percentage increase 2% for irradiated fibers at 300 kGy. For higher dosages (up to 900 kGy), crystallinity indexes were gradually decreased, with values lower than that for non-irradiated.

Application of gamma irradiation on the polymer concrete cause physical and chemical modifications on their components: cotton fibers and polyester resin. In the case of waste cotton fibers modifications in the crystallinity index have effects on the flexural behavior, mainly at 300 kGy, where maximum flexural modulus is done, thus higher crystallinity in the fibers provoke higher hardness into the concrete. Moreover, at high radiation dose (300 kGy), the degree of polymerization of the polyester resin is increased, respect to not-irradiated one.

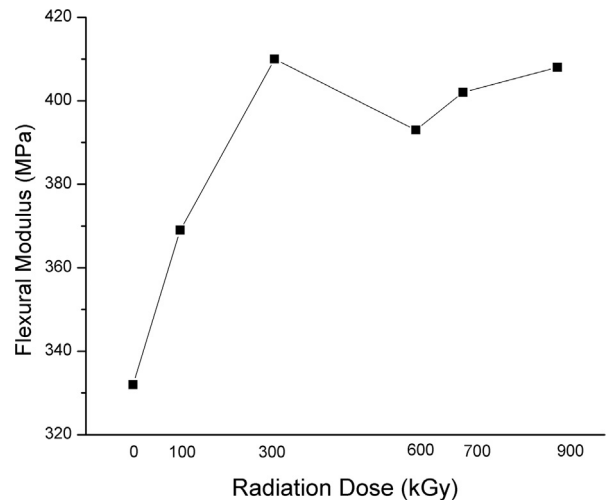


Fig. 16. Flexural modulus of polymer concrete with waste cotton fibers, irradiated at different dosages.

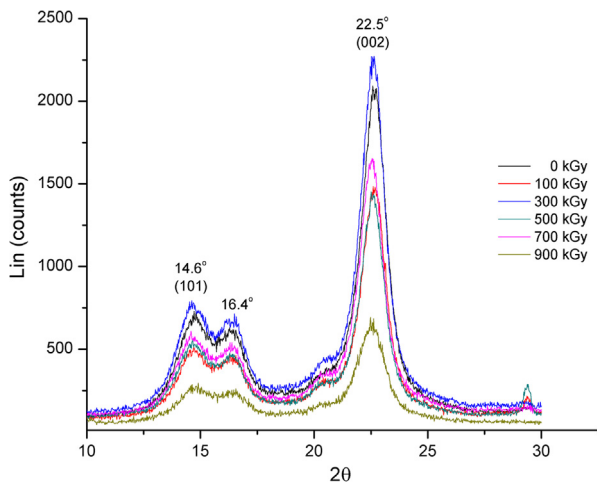


Fig. 17. X-ray spectra of waste cotton fibers, irradiated at different dosages.

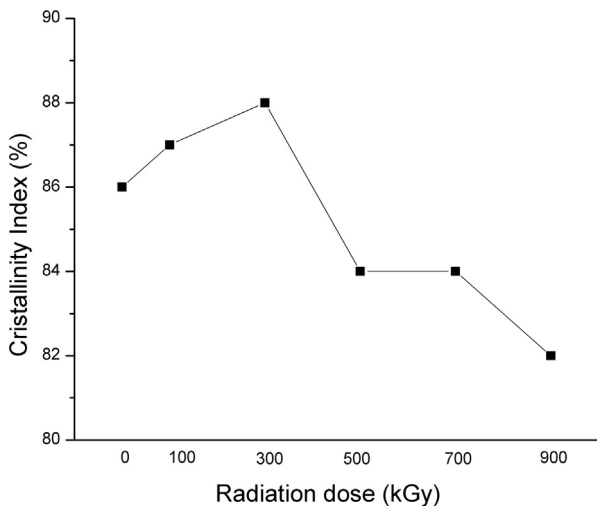


Fig. 18. Crystallinity Index of the waste cotton fibers.

#### 4. Conclusions

In the first stage of this work, highest mechanical strength values were obtained for polymer concrete elaborated with 30 wt% of polyester resin and 70 wt% of marble particles. Subsequently in the second stage, corresponding to fiber reinforced polymer concrete, lowest compressive strength and highest flexural strain were obtained for concretes with 1.0 wt% of waste cotton fibers, thus a more elastic material was obtained. Finally, in the third stage,

related to effect of gamma irradiation on polymer concrete, improvement of the mechanical properties were notable at dosages between 100 kGy and 300 kGy. Moreover, mechanical properties of polymer concrete were related to structural modifications of irradiated waste cotton fibers, mainly on the surface morphology and crystallinity index observed at 300 kGy. Finally, the FT-IR spectra show lower intensity for signals of cellulose II at irradiation dosages from 300 to 500 kGy; such irradiated fibers provided improvement on the matrix/fiber interface.

#### Conflict of interest

The authors declare that none of them has a direct financial relationship with the commercial trademarks mentioned in this paper that might lead to a conflict of interests for any of the authors.

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