Construction and Building Materials 47 (2013) 86–91

Contents lists available at SciVerse ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Luffa fibers and gamma radiation as improvement tools of polymer concrete

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HIGHLIGHTS

- Polymer concrete with silicious sand, unsaturated polyester resin and luffa fibers was elaborated.
- The effects of gamma radiation and the luffa fiber concentration on compressive and flexural properties were studied.
- We show that the compressive strain and the elasticity modulus are higher than plain concrete.
- The higher gamma dose provides the highest elasticity modulus.

ARTICLE INFO

Article history:
Received 29 January 2013
Received in revised form 23 April 2013
Accepted 4 May 2013

Keywords:
Polymer concrete
Luffa fibers
Gamma radiation
Mechanical properties

ABSTRACT

This work presents a study on the effects of luffa fibers and gamma radiation as tools for mechanical improvement of polymer concrete based on a polyester resin/foundry sand mixture. Different concentrations of irradiated and non-irradiated fibers (0.3, 0.6 and 0.9 wt%) and higher irradiation doses were used. The results show that the compressive strength and flexural strength values decrease gradually when increasing irradiated-fiber concentration, respect to plain concrete (without fibers and non-irradiated). Conversely an opposite effect occurs when polymer concrete is gamma irradiated, i.e. both the degree of polymerization and cross-linking of the polymeric resin are increasing. Moreover, the values for compressive and flexural strain as well as dynamic elasticity modulus increase when increasing irradiated-fiber concentration. The highest bending deformation is obtained with 0.9 wt% of fibers and 100 kGy of radiation dose.

1. Introduction

Polymer Concrete (PC) is a composite material comprising a thermosetting resin bonded to mineral aggregates. Mechanical improvements of polymer concrete are based primarily on the properties of polymer resin and mineral aggregates as well as their concentrations. The mineral aggregates involve several linked characteristics: specific area, interfaces with the matrix, strength and deformability, geometry and size. For example aggregates with irregular geometry and high surface area improve the anchorage with the matrix [1,2]. Moreover, some authors refer to different aggregate sizes ranging from 0.063 to 2.36 mm (mesh 230 to mesh 8) and different concentrations: from 70 to 90 wt% of polymer resin; the most common at 80 wt% of mineral aggregate and 20 wt% of polymer resin [3,4].

The mix design of polymer concrete involves an aggregate size gradation to provide the lowest possible void volume that will require the minimum polymeric binder concentration necessary to coat the aggregates and to fill the voids. Thus a variety of aggregate types have been used: silicates, gravel, limestone, calcareous rock, granite, clay, quartz, calcium carbonate, fine fly ash, phosphorgypsum, cinder, silica fume, silica sand; the last one the most used due to size distribution, ranging from 0.6 to 4.0 mm (mesh 30 to mesh 5).

New alternatives for physicochemical modifications of polymer resin and mineral aggregates have been proposed. The low hard-

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0950-0618/$ - see front matter
http://dx.doi.org/10.1016/j.conbuildmat.2013.05.038

ness of unsaturated polyester resin has been partly result adding liquid elastomers (reactive and/or non-reactive), or introducing dispersed solids, consisting of a soft immiscible phase as polymeric fibers [5–7]. Other methods, such as chemical attack or heat treatment are costly and time consuming.

When adding fibers the elastic characteristics of polymer resin is reinforced and improvement on fracture resistance is obtained, with better control and size distribution of cracks [8]. A lack of information concerning to fiber reinforced polymer concrete is present, moreover if natural fibers are contemplated. Some studies including the use of glass, carbon or boron fibers, as well as natural fibers as coconut or sugar cane bagasse. For example, when using short glass fibers or carbon fibers, the fracture properties are improved [8,9].

Natural fibers are a resource environmentally clean, renewable and biodegradable; then everyday more industries have interest in their use [10]. A natural fiber that has captured attention in applied research is luffa fiber, due to its physicochemical properties. They are from a subtropical plant of the cucurbitacea family, which produces a fruit with a fibrous vascular system (luffa), The fibers are composed mainly of cellulose (54%), hemi-cellulose (20%) and lignin (15%); with sizes between 1.5 cm and 1.5 m and a average diameter 8–10 cm [10]. It is abundant in China, Japan and other countries in Asia, Central and South America.

The study of the effects of natural fibers on the mechanical properties of resin based composites is in its early stages. One of the main characteristics of raw luffa fibers (without surface treatment) is its capacity to absorb moisture easily and its high potential as reinforced material in hybrid composites [11].

In polymer concrete physical interactions are present between polymer resin (matrix) and mineral aggregates, but no chemical bonds are present [12]. Therefore, improvements on the interfacial interaction between them are required. One proposal is to use gamma radiation to modify the surface of polyester resin and thereby achieve compatibility [13–18]. One can achieve good control of the dimensions and the elimination of internal stress; which cause reduction in mechanical strength [6].

The elastic modulus is the most often used characteristics of composites. In the case of building structures, the non-destructive tests (NDTs) take into account the acoustic impedance of the system components – important factors influencing ultrasonic wave propagation. Evaluations of different parameters such as defect detection, layer thickness or delaminations have been carried out by ultrasonic methods [19]. The dynamic elastic modulus is determined by measuring the pulse velocity along the composite and using electrical transductors located on the opposite sides of the cubic specimens of concrete. The energy supplied the ultrasound depends on how compact is the composite, including the void presence. One thus obtains the dynamic elastic modulus, $Ed = V^2\ d\ (1 + u)\ (1 - 2u)/(1 - u)$, here “$V$” is the pulse velocity; “d” is the mass density of the concrete specimen; and “u” is the Poisson ratio.

The gamma radiation applied to polymers causes three different process: breaking, cross-linking of chains, or graft. The permanence of any of these processes depends on the nature of the radiation, the chemical structure of the polymer and the applied dose [14,20]. Compared to thermal process or chemical attack, gamma radiation has more advantages in addition to spend less time and money. The advantages are: (a) initiating radiation requires no activation energy, (b) does not require catalysts or additives to initiate the reaction, (c) the initiation is homogeneous throughout the system [21], d) the process can be carried out at any temperature and can be interrupted at a specific reaction time, (e) the termination reaction is practically controlled, the polymer can be analyzed to a specific reaction step, and (f) during temperature initialization reaction is maintained, unlike the one presented in a conventional exothermic curing (without irradiation) [20–23].

The use of small concentrations of synthetic fibers in polyester resin ensures a homogeneous distribution of them. An optimal fiber concentration is required depending on the resin type. For example, improvement of 95% is obtained when using glass fibers as reinforcement and silane group agent. Moreover, with synthetic fibers the ductility increases but the modulus decreases; inclusive with long fibers the friction increases. Some works related to fiber reinforced polymer concrete show improvements on mechanical properties: adding 1 wt% of glass fibers the compressive strength is improved by 9%; or adding 2 wt% of carbon fibers an 16% is obtained [24].

In this paper polymer concrete is elaborated according to three different methods as follow: (a) PC = Plain Concrete, (b) AIF-PC = Added-Irradiated-Fiber PC and (c) DI-PC = Directly-Irradiated PC. The plain concrete does not contain fibers and non-irradiation is used. For the case of AIF-PC, luffa fibers are previously gamma irradiated and then adding to polymer concrete; and for DI-PC first non-irradiated luffa fibers are adding and mixed with foundry sand and polymer resin, after all PC is gamma irradiated. The compressive and flexural strength of all different polymer concrete were evaluated. Our research opens a window of opportunity for the use of low cost materials, as organic luffa fibers, and to promote environmental conservation.

2. Experimental part

2.1. Specimen preparation

We have prepared three different kind of polymer concrete: (a) PC, (b) AIF-PC and (c) DI-PC. The plain concrete was elaborated with 70 wt% of foundry sand and mixed with 30 wt% of polyester resin. While for the AIF-PC and DI-PC specimens the polyester resin concentration was maintained equal, but the foundry sand content was varying according to the luffa fiber concentrations (0.3, 0.6 and 0.9 wt%), used as reinforcement.

The polymer concrete specimens were elaborated with an isophthalic polyester resin (Aropol™ FS 3992) and siliceous sand (SP55-Sibelco). The resin was accelerated by 1% of cobalt octoate, and the methyl ethyl ketone peroxide (MEKP) was used as initiator. The sand with uniform granulometry had an average diameter of 245 μm (mesh 60); which was chosen as an intermediate value between particle-size ranging from 150 to 355 μm (mesh 100 to mesh 45), with which satisfactory results were obtained in previous studies [13].

Four different PC lots were prepared at different day; each lot contained six specimens. That is, for AIF-PC and DI-PC method of preparation 24 concrete specimens were made. After mixing, the polymer concrete prism specimens ($4 \times 4 \times 16$ cm) were placed in a controlled temperature room at 23.0 ± 3.0 °C for 24 h.

2.2. Mechanical tests

Mechanical behaviors of reinforced and plain PC formulations were assessed by means of flexural and compressive tests. For each PC formulation four specimens were tested. Prismatic specimens were tested in three-point bending up to failure at a constant loading rate of 1 mm/min over a span of 100 mm, as specific by RILEM CPT PCM-8 test method. The two pieces of each broken specimen in bending were posterior tested in compression by using Instron Universal testing machine, with a load cell of 100 KN, at the loading rate of 1.25 mm/min, following the procedure described in UNE 83821 test standard. The dynamic modulus of elasticity was measured by using an ultrasonic testing equipment for building materials: Ultrasonic Pulse Velocity Tester model 58-E0048 (Controls™, Cernusco, Italy), with an ultrasonic resolution of 0.1 ms.

2.3. Morphological characterization

Both irradiated and non-irradiated luffa fibers were dried in a rotovapor for 24 h. Then the luffa surfaces were analyzed by scanning electron microscopy in the secondary-electron mode by using a JEOL model JSM-6510LV machine.

2.4. Irradiation procedure

Both the luffa fibers and the polymer concrete with luffa fibers were exposed to gamma radiation dose of 50 and 100 kGy in air at room temperature. A dose rate of 3.5 kGy/h was applied by using a Transelektro irradiator LGI-01 provided with a ^{60}Co source manufactured by IZOTOP Institute of Isotopes Co. Ltd., Budapest, Hungary, and located at the National Institute of Nuclear Research (ININ-Mexico).

3. Results and discussion

3.1. Compressive strength

In Fig. 1 we show the compressive strength σ_c values for polymer concretes. (a) For plain Polymer Concrete (PC) taken as control (without fibers and non-irradiated) a compressive strength σ_c value of 27 MPa is observed; (b) for AIF-PC (fibers are previously irradiated and then adding to polymer concrete) the compressive strength values diminish according to both luffa fiber concentration and gamma radiation doses increase. The lowest value is obtained when adding 0.9% of irradiated fibers at 100 kGy. This value is 40% lower than those for PC taken as control.

Irradiated fiber concentration is critical for compressive strength decreasing as seen in Fig. 2. The compressive tested specimens have a progressive damage when adding luffa fibers with higher irradiated dose. Such diminution on the compressive strength can be attributed to: (1) weak fiber–resin matrix bond as consequence of materials nature: the luffa fibers are hydrophilic (water absorption 13.6 g/g), and the resin matrix hydrophobic and (2) fiber porosity, the pores act as flaws in the composite during compression stress, thus the resistance decreases. Moreover, porosity also prevents cracks from spreading and reaching the breaking point, because during the compression test pores act as "failures" in concrete.

(c) For DI-PC (non-irradiated fibers are mixed with foundry sand and polymer resin, then PC are gamma irradiated), the compressive values increase when increasing the fiber concentration and gamma radiation dose; the highest value is for DI-PC with 0.9% of fibers irradiated a 100 kGy, it means an increment of 27% respect to plain concrete. The present σ_c value for plain concrete (27 MPa) is lower than for PC with silica sand (62.2 MPa) reported in literature [5]. Thus, we recommend combine 0.9 wt% of non-irradiated fibers and 100 kGy of radiation dose to obtain the highest σ_c values.

An explanation of such increment is related to the multiple radiation effects on the polyester resin; as already noted, the irradiation causes chain scission but it also produces some cross-linking, chain relaxation and cage breaking. In consequence the formation of bonds into polymer chains increases the degree of polymerization of the resin matrix as seen in Fig. 3. For non-irradiated resin a homogenous surface is seen (Fig. 3a); such morphology is affected by gamma radiation because a higher number of chemical bonds is established and a more rough surface is observed (Fig. 3b), and for higher radiation dose a well-defined surface is seen, with the presence of small particles created from the cross-linking of the resin (Fig. 3c).

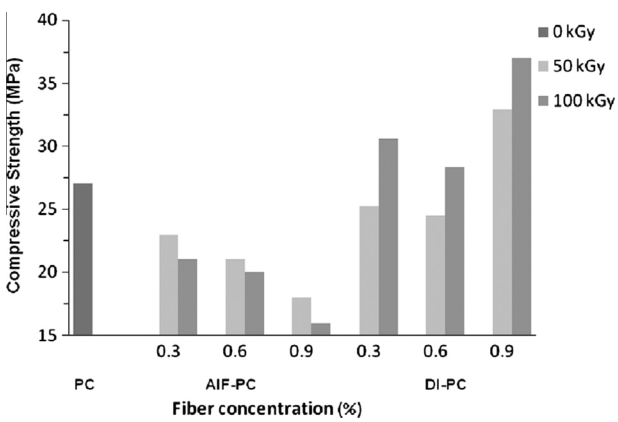

Fig. 1. Compressive strength of polymer concrete.

3.2. Compressive strain at yield point

In Fig. 4 we show compressive strain values at yield point for polymer concretes. (a) For plain polymer concrete a compressive strain value of 0.019 mm/mm is observed; (b) for AIF-PCs the com-

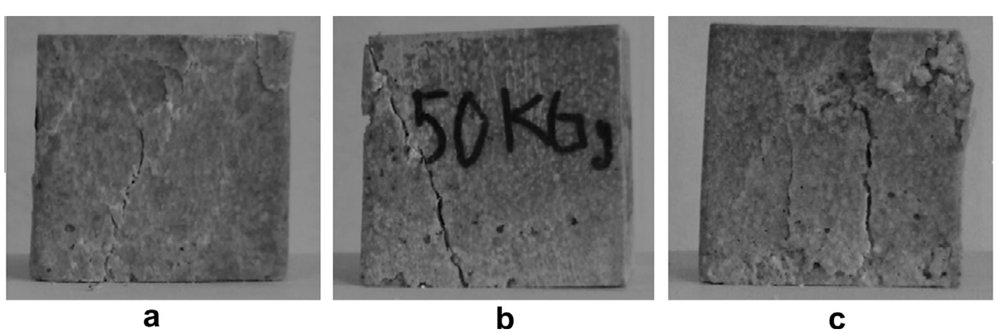

Fig. 2. AIF-polymer concrete specimens after testing: (a) non-irradiated, (b) irradiated at 50 kGy, and (c) irradiated at 100 kGy.

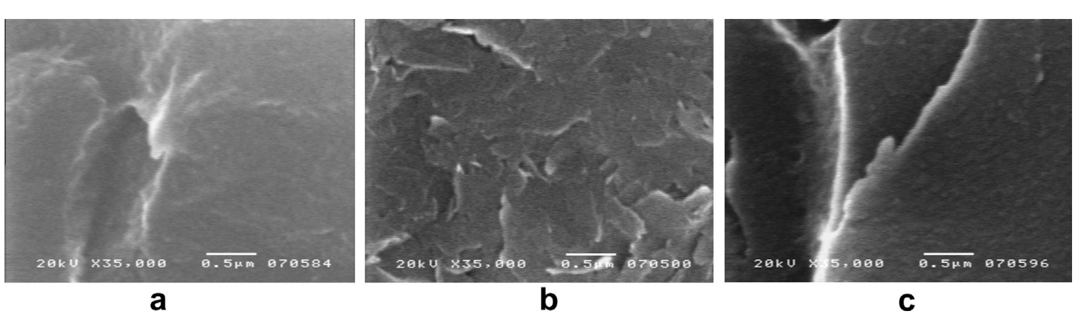

Fig. 3. SEM images of polyester resin: (a) non-irradiated; (b) irradiated at 50 kGy; and (c) irradiated at 100 kGy.

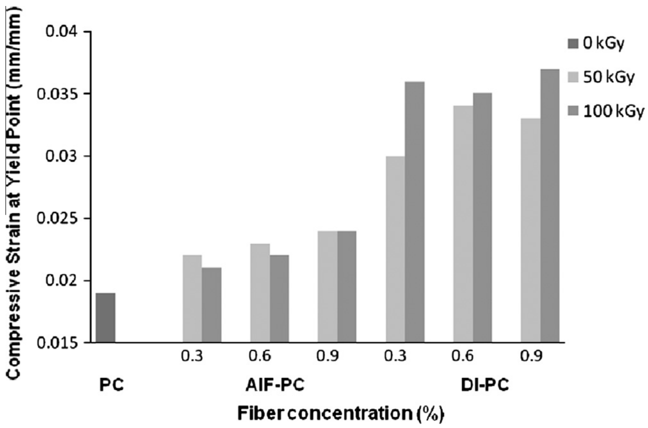

Fig. 4. Compressive strain at yield point of polymer concrete.

pressive strain values increase when increasing the fiber concentration. The largest deformation occurs on PC with 0.9 wt% of luffa fiber, it means an improvement of 13% respect to plain concrete. We recall that the values are comparable to those for pure-polyester resin (0.021–0.026 mm/mm) obtained in earlier work [14]. Moreover, minimal difference for PC with irradiated fibers at 50 or 100 kGy is observed. Thus the compressive strain of AIF-PCs does not depend on gamma doses, but of luffa concentration. As we remind, the fibers do not have a preferential arrangement in the polymer concrete and suffer mechanical deformations when the polymer concrete is submitted to the compression test; the deformations can vary according to the applied dose.

(c) The DI-PCs have a similar behavior as AIF-PC: the compressive strain values increase when increasing the fiber concentration. The highest value is obtained for PC with 0.9 wt% of irradiated luffa fibers at 100 kGy, it means an improvement of 94% than those for plain concrete; inclusive 280% respect to the standard values for a polyester-based PC reported (0.01 mm/mm) [6].

The increment of the compressive strain of DI-PC depends on irradiation of all PC components; which participate in the absorption of the applied compressive stress and produce sustained deformation. In fact, when increasing the applied radiation dose, the morphological changes are a consequence of the polyester constraints resulting from cross-linking of the chains in the polyester resin; in consequence a harder polymer concrete is obtained.

When comparing the compressive strain values with other irradiated-PCs elaborated with different mineral aggregates and fibers, we can see the advantages of using luffa fibers. The present values are in the range from 0.029 to 0.037 mm/mm, higher than for PC with silica sand (0.006–0.013 mm/mm) [5], or PC with $CaCO_3$ (0.010–0.016 mm/mm) [25], or PCs with silica sand + $CaCO_3$ (0.014–0.017 mm/mm) [13].

3.3. Flexural strength

Fig. 5 shows the flexural strength for polymeric concretes. Several behaviors are observed. (a) For plain concrete a flexural strength of 10 MPa is observed. (b) For AIF-PCs the flexural strength decreases with increasing fiber concentration. The lowest value is obtained for PC with 0.9 wt% of fiber; it means 20% lower than those for plain concrete. Moreover, the values are greater for 50 kGy than for 100 kGy. The components nature affect the physical union between fiber and resin matrix which reducing the resistance. (c) For DI-PCs two behaviors are identified: (i) flexural strength decreases for 0.6 wt% of fibers (31% lower) and after increasing for 0.9 wt% of fiber and (ii) the variations of the values are minimal for both radiation doses: no more than 5% for each fiber concentration.

In contrast to the compression strength behavior where the values increase, in the case of flexural resistance the values decrease. According to the literature when adding synthetic fibers the flexural property is lightly improved. For example, when adding 1 wt% of non-irradiated glass fiber to polymer concrete based on epoxy resin an improvement of 5% is obtained; or adding 1 wt% of non-irradiated wood fibers the improvement is of 6% [26]. Thus the irradiation of luffa fibers influence on the flexural strength.

Fig. 6 shows the flexural strain for polymer concretes. Different behaviors were observed. (a) For plain concrete a flexural strain of 0.41 mm is observed. (b) For AIF-PCs the flexural strain has the same behavior as compressive strain: the deformation increases as the concentration of fiber increases. A maximum increment of 60% for PC with 0.9 wt% of fiber irradiated at 50 kGy is obtained, respect to plain polymer concrete. (c) For DI-PCs different behaviors are observed: (i) for 50 kGy, the flexural strain increase according to the fiber concentration increases; but (ii) for 100 kGy, such deformation decrease for 0.6 wt% of fiber, and after increasing for 0.9 wt% of fiber; and (iii) higher deformation values are obtained at 100 kGy instead of 50 kGy.

3.4. Compression modulus of elasticity

Fig. 7 shows the compression modulus of elasticity E_c for polymer concretes. Some behaviors were found: (a) For plain concrete an elasticity modulus of 2.8 GPa is observed. (b) For AIF-PCs the elasticity modulus decreases with increasing irradiated-fiber concentration; Thus a more ductile polymer concrete is obtained; notable is the diminution, 58%, for concrete with 0.9% of irradiated

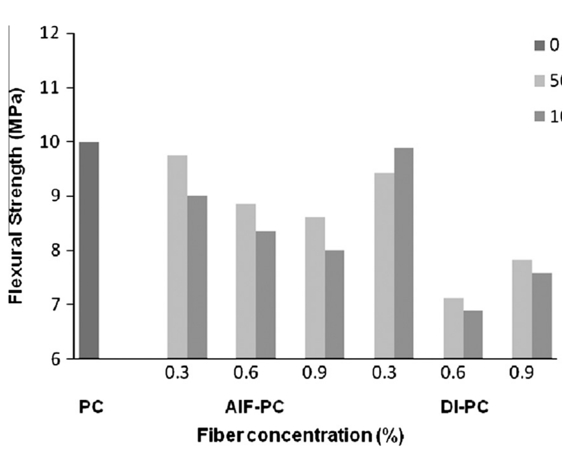

Fig. 5. Flexural strength of polymer concrete.

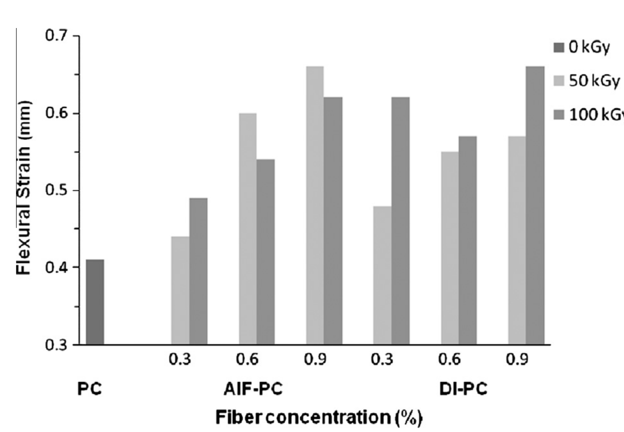

Fig. 6. Flexural strain of polymer concretes with luffa fibers.

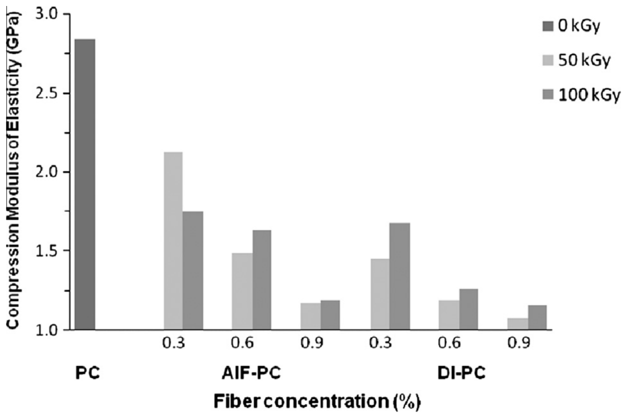

Fig. 7. Compression modulus of elasticity of polymer concretes.

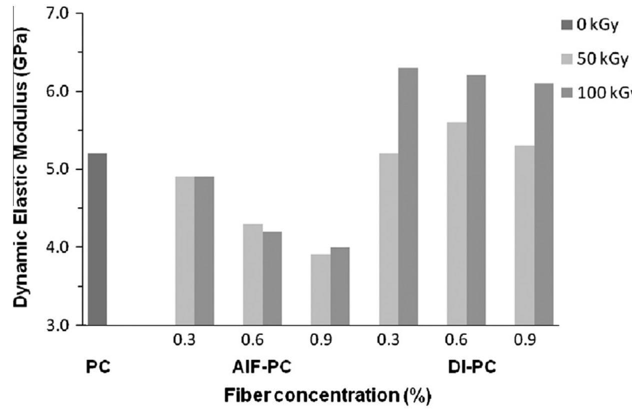

Fig. 8. Dynamic elastic modulus of polymer concretes with luffa fibers.

fibers, regarding plain concrete. Moreover, the compression modulus of elasticity exhibits similar behavior as the compressive and flexural strength. (c) The DI-PCs show the same behavior as AIF-PC concretes. Such deformations are due to luffa fiber when polymer concrete is submitted to compression test. Relevant contribution is made for DI-PC with 0.9 wt% of fibers according its ductility: it has highest strain rate (see again Fig. 4) and the lowest E_c values (Fig. 7).

To have high deformability and moderate compressive strength at the same time depends on the combination of fiber concentration and the high radiation dose. Moreover, special attention should be taken on the polyester resin behavior after irradiating. In general, non-irradiated resins show homogeneous surfaces; when irradiating it to 50 kGy results in formation of constraining regions and the elasticity modulus values increase. At 100 kGy more constrained regions are present, providing the highest elastic modulus [12].

When comparing the E_c values with others polymer concretes we observe that the present values (from 1.0 to 2.8 GPa), are lower than for PC with silica sand + $CaCO_3$ (5.6–8.0 GPa) [13], or PC with silica sand (7.4–16.3 GPa) [5] or PC with $CaCO_3$ (10.5–16.1 GPa) [17]. Thus, less ductility is achieved when using silica sand + luffa fibers.

3.5. Dynamic elastic modulus

Fig. 8 shows the dynamic modulus for polymer concretes. Some behaviors were found: (a) for plain concrete an elasticity modulus of 5.2 GPa is observed, (b) for AIF-PCs the modulus decreases with increasing fiber concentration. A diminution of 25% is obtained for polymer concrete with 0.9% of fiber, regarding to plain concrete. Conversely, (c) the DI-PCs specimens show on opposite behavior as AIF-PC concretes: the dynamic modulus increase respect to plain concrete; being 25% higher. In general terms, the values for the dynamic elastic modulus, E_d, are higher than those for compression modulus, E_c, is to say: (3.9–4.9 GPa) vs (1.2–2.2 GPa).

The mechanical performance of the PCs can be related to the morphology of the luffa fiber surfaces before and after irradiating. Fig. 9 shows images of non-irradiated and irradiated luffa fibers obtained by Scanning Electron Microscopy (SEM). For non-irradiating luffa fibers width "channels" (4–12 μm) are seen (Fig. 9a); as well as rough surfaces, particles with different lignin shapes (indicated by arrows), and thin layers of lignin and hemicellulose covering the cellulosic fibers (indicated by rectangles) as seen in Fig. 9b. For irradiated fibers at 50 kGy and 100 kGy a higher number of lignin particles are seen (Fig. 9c and d); some studies report similar results [27,28].

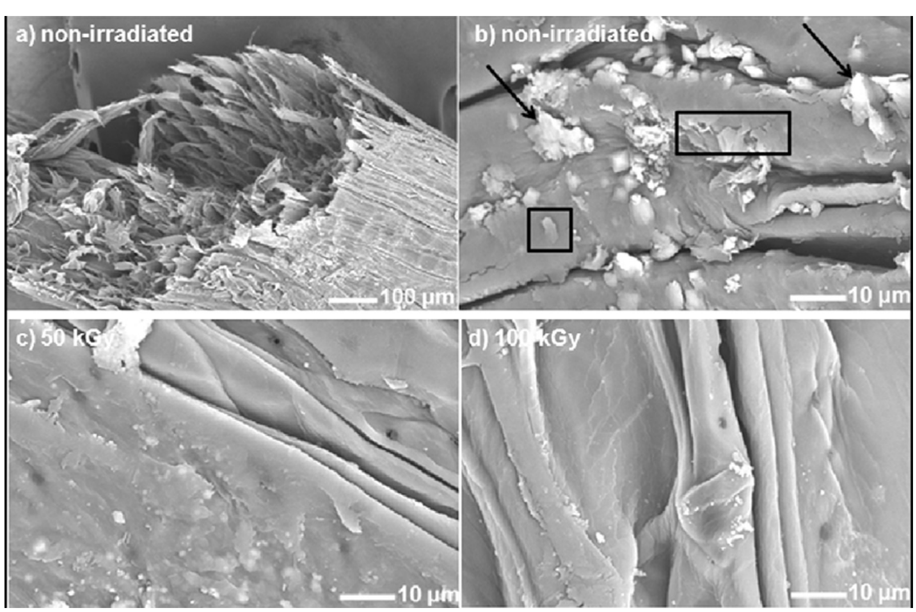

Fig. 9. SEM images of non-irradiated and irradiated luffa fibers.

According to previous results, the compressive strength and flexural values, for AIF-PCs, decrease when increasing irradiated-fiber concentration. This can be attributed to the morphological characteristics of fibers, mainly to: (a) channels, which cannot be filled completely with resin or sand after mixing process, thus creation of pores inside of concrete are formed. Such pores act as flaws in the concrete making the resistance (compressive and flexural strength) decreases markedly; providing a more ductile polymer concrete and (b) moreover, the ionizing energy generates more contact points in the luffa fibers and in consequence larger contact areas between the components: luffa fibers, polyester resin and silica sand. In turn, an increased number of contact points in the polymer concrete will resist larger loads oriented at various angles relative to the longitudinal axes of the fibers.

For DI-PCs an opposite effect occurs; it is due to increase in the degree of polymerization and cross-linking of the resin more than fiber concentration effect. When increasing the applied radiation dose on the DI-PCs, the surface is modified and the aggregate particles are fully covered. This situation is a consequence of cross-linking of the chains in the two polymers involved, the polyester resin and the luffa fibers. Moreover, for higher applied doses, the polyester resin is constrained, the surfaces show more agglomeration regions, what produces the highest dynamic modulus.

4. Conclusions

As expected, mechanical behavior depends on the combination of luffa fiber concentrations and the applied radiation doses. Conclusions are made based on the three methods of preparation: PC (Plain Concrete); AIF-PC (Added-Irradiated-Fiber PC) and DI-PC (Directly-Irradiated PC). For compressive strength a notable behavior is observed, while the values decrease gradually for AIF-PC when increasing the fiber concentration; conversely for DI-PC these increases, thus more influence of irradiated-fibers is in the first case, and irradiation of the resin in the second. In the case of flexural strength the values decrease for both methods (AIF-PC and DI-PC); conversely, the values for compressive and flexural strain at the yield point increase for both methods. The highest bending deformation is obtained with 0.9 wt% of fibers and 100 kGy of radiation. Special attention is made for elasticity modulus; both gamma radiation and the fiber concentration tend to increase the deformation respect to plain concrete. Such behaviors suggests generation either a ductile material or hard material; the first one promoted by luffa fibers and the second by irradiated resin.

Acknowledgement

Financial support of "Joint UAEM-University of North Texas Research Seed Funding Program" by Grant # UNT-UAEM 3355/2013FS is acknowledged.

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