

Compressive Strength of Gamma-Irradiated Polymer Concrete

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Polymer concrete (PC) was developed by using different concentrations of silica sand as aggregate and a commercial unsaturated preaccelerated (orthophthalic) polyester resin. The PC specimens were submitted to gamma radiation at several dosages: 5, 10, 50, 100, and 150 kGy. Mechanical performance was evaluated including the compressive strength, the compressive strain at yield point, and the compression modulus of elasticity. As expected, mechanical properties depend on both the resin concentration and the applied radiation dose. Improvement achieved in mechanical properties is related to the morphological characteristics observed by scanning electron microscopy. POLYM. COMPOS., 29:1210–1217, 2008. © 2008 Society of Plastics Engineers

INTRODUCTION

Polymer concrete (PC) is a particulate composite where thermoset resins binds inorganic aggregates instead of the water and cement binder typically used in Portland cement concrete (PCC). We thus have a polymeric matrix and dispersed particles of strengthening phases. The polymer constitutes the continuous phase hence the composite behavior is largely determined by the properties of the polymer, which are dependent on time, structure, and temperature. Polyesters or epoxy resins have been used

for the purpose, with polyester resins the most common due to low prices and corrosion resistance.

Let us first make a survey of PCs used so far. Silica sand, gravel, or fly ash as aggregates have been used; catalysts or hardeners are added to the resin prior to mixing and casting. For the case of sand, there are various options: (a) sand particles composed mainly of quartz with the specific gravity of 2.65 g/cm³, with grain sizes ranging from 5 to 0.07 mm (200 mesh); (b) sand with particle sizes between 0.84 and 0.59 mm (20–28 mesh) used in fracture studies [1]; (c) well-graded blasting sand (20–28 mesh) used in the foundry industry for the compressive and fracture behavior determination; (d) sand with fineness modulus of 2.35; and (f) coarse aggregate with the diameter of 9.5 mm for manufacturing epoxy-PC, oven-dried for 24 h at (120°C [2]. We recall a wide range of uses of epoxy resins [3–5].

Several silica sands have been used in the foundry industry: (a) Type 6/16 [particle size distribution 2.36–0.85 mm and average grain size = 1.35 mm]; (b) Type 16/24 [particle size distribution 1.0–0.5 mm and average grain size = 0.67 mm]; (c) Type 110 [particle size distribution (1.0–0.063 mm) and average grain size = 0.12 mm] [6], and (d) sand with 0.245 mm average diameter (60 mesh) [2].

In principle, the mix design of PC typically involves an aggregate gradation to provide the lowest possible void volume that will require the minimum polymeric binder concentrations necessary to coat the aggregates and to fill the voids [2].

The composition of PC is determined by its applications; in the last decade PC are being used increasingly in

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the construction, in highway pavements or in underground wastewater pipes. Moreover, PC is used very efficiently for manufacturing thin overlays since it has the advantage of providing lightweight, and for manufacturing precast components for bridge panels, buildings, machine bases, and transportation components. Moreover, PC has been shown as suitable for the use in high pressure, high temperature environments such as the utilization of geothermal energy due to its durability in hot acidic springs.

From the stability point of view, traditional concrete can trap some water, which on freezing cracks the concrete. Furthermore, cement can be attacked by acids that would lead to deterioration. For a compact polymer, with a minimum of open voids combined with a hydrophobic nature and chemical inertness, these problems can overcome [2]. Moreover, in impregnated hardened cement, the unsaturated liquid absorption depends on the permeability. The amount of absorbed polyester is a function of the accessibility of the pores to be filled with liquid unsaturated polyester, and also of the impregnation time [7].

PC shows a longer maintenance-free service life than PCC, because of its durability and physical properties superior to those of PCC, fast setting times (curing within 1 or 2 h), and low permeability [2, 8]. Moreover, improved mechanical strength (compressive and flexure) and chemical resistance are also advantages of PC in comparison to ordinary PCC.

Different compositions of PC have been obtained: (a) 80% by mass of foundry sand and 20% by mass of epoxy resin [9], (b) 10, 14, and 18% of weight fractions of the polyester resin, with the viscosity between 40 and 59 poise at room temperature and the specific gravity of 1.07 [1]. In the case of plain mortar, different formulations have been in use: (a) 20 wt% of unsaturated orthophthalic polyester resin diluted in 44% styrene. The resin system is preaccelerated by the manufacturer, the initiator used was methyl ethyl ketone peroxide (2 phr); or (b) 20 wt% of epoxy resin based on a diglycidyl ether of bisphenol A and an aliphatic amine hardener. Such a system has low viscosity, and is processed with a maximum mix to hardener ratio of 2:1 [8].

The epoxy PC has common compressive strength in the range from 80 to 100 MPa and compressive strain at break of 0.006 mm/mm, in comparison to PCC (35 and 0.003 MPa, respectively); compression modulus of elasticity of 11.5 GPa and bending strength of 38.7 MPa. Nevertheless, PC does display brittle characteristics [8], what has limited its usefulness for load-bearing applications. We recall a definition of brittleness which involves the elongation at break [10]. Therefore, some reinforcement is needed in the tensile zone, for example glass fibers used for reinforcement of polymer laminates. Glass fibers are noncorrosive, nonconductive, nonmagnetic, and have low density and high modulus [8].

Another option involves a modification of physico-chemical properties of the resins and its bond with the aggregates. Evident precautions for the aggregates are:

very low moisture concentration (sometimes oven-dried before mixing needed) and purity. The literature does not provide much guidance in this area.

Different resins have been used for manufacturing PC: (a) epoxy resins with low viscosity (500–600 mPa) and the flexural strength of 70 ± 5 MPa; (b) orthophthalic preaccelerated polyester resins with the flexural strength of 119 MPa [9]. Curing of polyester resins must be well controlled in order to obtain good workability for a high viscous PC specimens and to avoid the presence of water because it can damage the hardening process of the resin [2, 9].

Different initiators and promoters have been used to begin the free-radical polymerization process. For example, in the case of polyester PC: (a) 1.5% by weight (of the polyester resin) of methyl ethyl ketone peroxide (MEKPO) as initiator and 0.3% by weight of the cobalt naphthenate as promoter; or (b) MEKPO and an amine as initiators and cobalt naphthenate (6 wt% concentration) as the promoter. For methyl methacrylate PC, benzoyl peroxide (BZP) as the initiator and dimethyl-*para*-toluidine (DMPT) as the promoter.

In the first stage of the reaction, the resin is usually in the liquid state. When the temperature passes through the reaction temperature, the physical state of the compound changes abruptly and the crosslinking reaction is slowed down [8]. The phase transition occurs faster as a result of curing. The partially cured resins typically exhibit two transitions changing as curing proceeds. This is interpreted as an effect of the physical structure of the gel formed at a very early stage of the reaction on the curing process [11].

When the glass-rubber transition of the cured resin passes through the reaction temperature, the reaction is slowed down but it is not stopped completely. The oscillator strength of the transition is found to decrease as curing proceeds. This is explained as the effect of crosslinking by which the polar groups in the resin chains become fixed.

A typical crosslinked chain fragment has the following chemical structure: in the uncured resin and in early stages of the reaction the chain segments, the thermally stimulated movement which produces the glass-rubber transition is determined by the polar carbonyl groups. As the curing reaction proceeds, more and more of these groups are joined by the bulky styrene. At this second stage, the transition depends mainly on movement of apolar hydrocarbon groups [11, 12].

It is well known that the gamma radiation causes structural modifications of polymers via three main processes: scission, crosslinking, and grafting of chains involving generation of free radicals [13–17].

There is a monotonous increment in the conversion percentage up to about 8 kGy in polyester resins irradiated with gamma rays. At this stage a gel fraction and the styrene monomer are present. Although the glass-rubber transition is below the reaction temperature up to doses of about 8 kGy, the samples behave as fairly elastic gels

from about 3 kGy. This means that multiple-phase products are formed in that stage of the reaction when the glass-rubber transition is below the reaction temperature (the curing temperature = 35°C) [11]. For a loss of 5% of its initial weight, the gamma irradiated polyester resin (at 50 kGy) shows an increment of temperature of 35°C (from 260 to 290°C) as compared with that of unirradiated polyester resin. Moreover, there is no significant difference in thermal decomposition for both kind of resins (un- or irradiated) at higher decomposition temperatures up to 500°C [7].

When the epoxy resin is irradiated by gamma rays, chain scission occurs; the density of crosslinks decreases while T_g decreases to 121°C at 6750 kGy. Chain length between crosslinks is assumed to increase and the mobility of the chains increases. However, because that T_g value is far higher than the experimental temperature = 195°C, the chains still cannot flow. On the other hand, creep is found to increase in the hot condition. This result can not be explained by the change of elastic modulus; the modulus has hardly changed with the irradiation, as the bending test results show [18].

In general, when gamma radiation is applied, first radicals are formed first at the molecular end groups. Possible reactions of these groups include scission (which is always detrimental) as well as crosslinking. However, when the chains with the radicals are restrained by the surrounding chains, the radicals may rebound with the original chain, resulting in the recovery of the chain instead of scission—a so-called “cage effect.” At low temperatures when recovery occurs, the total number of the scissions is believed to be proportional to the number of the radicals present. At high temperatures, the chains have strain energy caused by the stress.

When using gamma radiation, the radicals formed are believed to help a given chain to escape from the restraint of the surrounding chains. The so-called cage breaks, with short chains breaking first [18].

We expect that the ionizing energy can improve compatibility between polyester resin (matrix) and the silica sand (aggregate) by means of the structural and surface modification of both components. Thus, improvement of the mechanical properties of PC can be obtained.

In this work we report on gamma radiation effects on polyester PC (polyester + PC) systems, focusing on the mechanical improvement. Based on this, we propose a novel technology for manufacturing PC—different than costly and time consuming current procedures such as chemical attack or thermal treatment.

EXPERIMENTAL

Specimen Preparation

For preparing the PC specimens, natural silica (0.150 mm : mesh 100) from a local company (GOSA™, Tlalne-pantla, Mexico) was used, as well as a commercial unsat-

urated preaccelerated polyester resin (orthophtalic): a viscous liquid resin with a styrene monomer concentration of 30% (Polylite 32493-00™, Reichhold, Atlacomulco, Mexico). The proportions of the initiator methyl ethyl ketone peroxide (MEKPO) added to the polymer resin for initiating the free-radical polymerization process was 1 mL/100 g of the resin weight.

Five different PC lots identified by A, B, C, D, and E were prepared, each one a different day. The proportions of the polyester resin in the PC were 38, 43, 54, and 58% by weight. Also a 100% polyester resin specimen as a reference has been prepared. After mixing, the concrete cubic specimens ($5 \times 5 \times 5 \text{ cm}^3$) were placed in a controlled temperature room at $(23.0 \pm 3.0)^\circ\text{C}$ up to 72 h.

Mechanical Tests

The compressive tests of the PC cubic specimens were carried out in an Instron Universal Testing machine Model 1125 according to the ASTM C-109M standard. The charge speed was between 91 and 184 kg/s, holding the charge until reaching the maximum value to assure the reliability of the test.

Morphological Characterization

The surfaces of the silica sand and polyester resin, before and after irradiation, as well as the fractured zone of the manufactured PC were analyzed by scanning electron microscopy (SEM) in a JEOL model JSM-5200 machine, in the secondary-electron mode. In the present case this mode provided good contrast between the constituents. In other cases, such a polymer matrix + metal micropowder, backscattered electrons provide better results [19].

Irradiation Procedure

The PC was exposed to varying gamma radiation doses using a ^{60}Co source. The experiments were performed in air at the room temperature; the dosages were 5, 10, 50, 100, and 150 kGy at the dose rate of 6.10 kGy/h. The irradiation was provided by a 651 PT Gammabeam Irradiator manufactured by the Atomic Energy of Canada. (AECL, Chalk River, Ontario), and located at the Institute of Nuclear Sciences of the National Autonomous University of Mexico.

COMPRESSIVE STRENGTH

In Fig. 1 we show the compressive strength for nonirradiated PC. We see that compressive strength increases along with the resin concentration increase from 49.7 to 83.3 MPa; moreover, the value for the nonirradiated polyester resin (identified by the number 100) is the lowest of all, namely 42.1 MPa. In fact, it has been observed that the compressive strength of modified mortar systems is

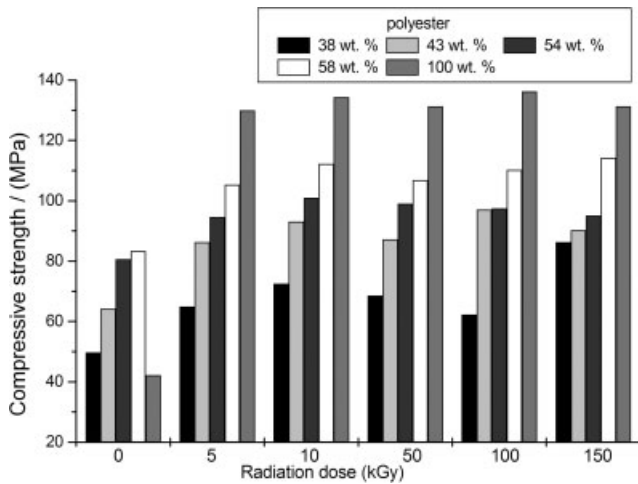


FIG. 1. Compressive strength for non- and irradiated polyester + PC at different applied doses.

increasing when the polyester resin concentration formed in the porous matrix increases [7].

In general, for irradiated-PC the compressive strength increases when the resin concentration increases too, for each applied radiation dose. Moreover, the radiation effects produced on the resin and the silica sand allow to have the highest values for irradiated PC with 58% of resin which means an 42% higher than conventional values reported for PC (80 MPa).

Nevertheless, for irradiated-polyester resin the values vary from 129.8 to 136.2 MPa, that is between 208 and 223% more than the nonirradiated resin. Thus, given a small difference of 4.9% in the compressive strength in the entire dosage range (from 5 to 150 kGy), the compressive strength of the irradiated-resin is almost independent of the dosage.

More detailed inspection of the radiation effects is provided in Figs. 2 and 3. We see in Fig. 2 the compressive strength behavior for PC with 38 and 43 wt% of the resin. The irradiated-PC with 38% of resin show three well

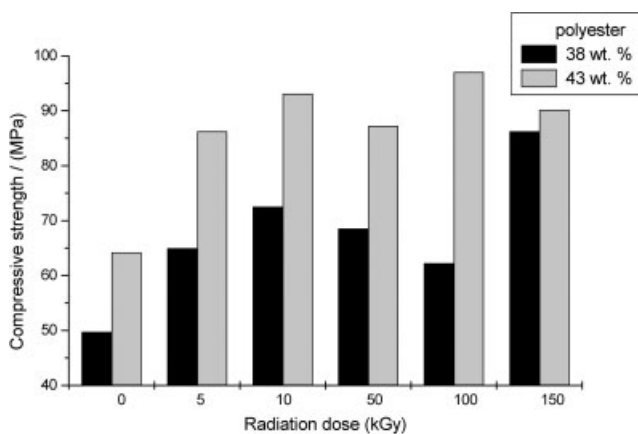


FIG. 2. Compressive strength for irradiated polyester + PC with 38 and 43% of the resin.

defined stages: in the first the values increase from 0 to 10 kGy; then the values decrease for 100 kGy (second stage); and finally they increase again for 150 kGy. Thus an “increment-decrement-increment” phenomenon is present; we call it a periodical behavior. A similar behavior is observed for 43 wt% of the resin. In this case there are not three but four stages (I: from 0 to 10 kGy; II: from 10 to 50 kGy; III: from 50 to 100 kGy, and IV: from 100 to 150 kGy). An explanation of the periodical behavior might be related to the fact noted in Introduction: irradiation causes chain scission, but it also produces relaxation and cage breaking.

The compressive strength values for PC with 38% of resin vary from 49.7 to 72.5 MPa for all irradiated-PC in the range from 0 to 100 kGy; only for 150 kGy the value is larger than conventional values reported for PC. In the case of PC with 43% with exception of the nonirradiated specimen, all PCs have compressive strength values higher than 80 MPa.

We can observe for both concentrations of resin a similar behavior from 0 to 10 kGy. For higher doses the compressive strength values show different behavior, reaching a maximum value at 150 kGy for PC with 38% of resin, and at 100 kGy for PC with 43% of resin.

Thus the compressive strength response of the PC with its periodical behavior with respect to the polyester resin concentration and the radiation dose has consequences for the concrete application. For PC with 38% of resin, application of radiation among 10 and 100 kGy is not enough for reaching the conventional compressive strength values reported for PC alone. It is necessary to apply the dose of 150 kGy. Nevertheless, for PC with 43% of resin, all irradiated specimens have values above 80 MPa. Thus, for concentrations of the resin below 43% it is necessary to apply more ionizing radiation so as to reach desired values.

In Fig. 3, the PC with 54% of the resin shows a two-stage behavior: I: from 0 to 10 kGy where the compressive

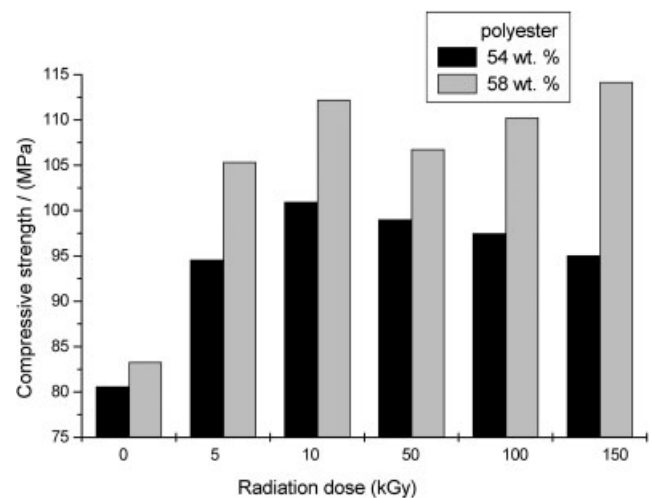


FIG. 3. Compressive strength for irradiated polyester + PC with 54 and 58% of the resin.

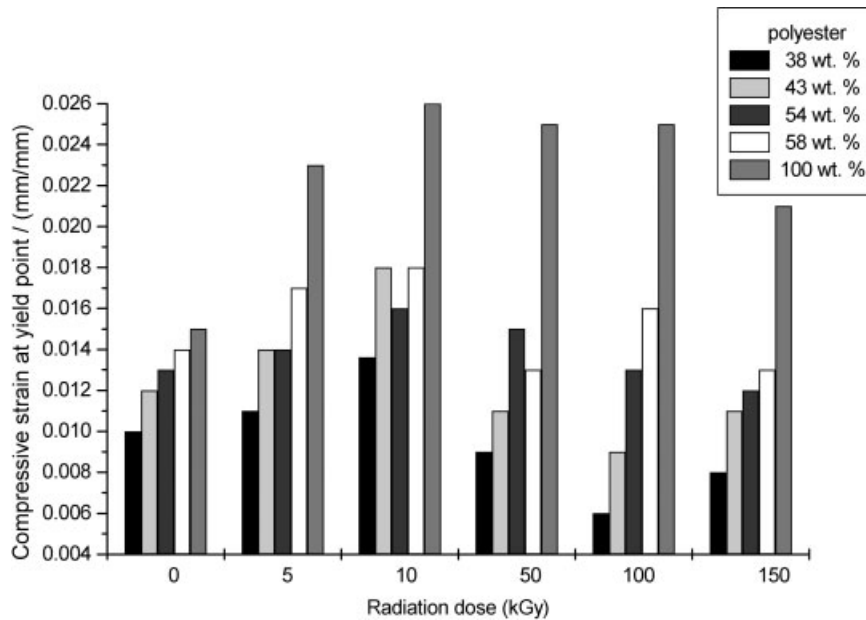


FIG. 4. Compressive strain at yield point for irradiated polyester + PC at different applied doses.

strength is increasing; and II: from 10 to 150 kGy, when the values are decreasing. For this resin concentration all irradiated-PCs have more than 80 MPa strength (from 94.5 to 100.9 MPa). When increasing the concentration to 58% of the resin, the highest values for all irradiated-PCs are obtained (from 105.3 to 114.1 MPa). We observe a three-stage behavior: (I) an increase from 0 to 10 kGy, (II) a decrease from 10 to 50 kGy, and (III) an increase from 50 to 150 kGy.

Summarizing, we find three important features: (a) for all irradiated PCs the compressive strength increases from 0 to 10 kGy; for higher dose the behavior varies according to the resin content. For lower concentrations of the resin (38 and 43%) a maximum value is found at 10 kGy; (b) the highest compressive strength values correspond to the high concentration of resin, but the differences of values for all applied doses (from 10 to 150 kGy) are small (8.3%). Thus, for economical and time saving reasons is better to irradiate at low doses.

COMPRESSIVE STRAIN AT YIELD POINT

We now consider the compressive strain at yield point; see Fig. 4.

In general, the compressive strain values increase when the resin concentration increases. Similarly to compressive strength results, high values are found for high resin concentrations. Nevertheless, all PC samples, including irradiated and nonirradiated, have values higher than 0.01 mm/mm (the standard value for polyester + PC). Moreover, the compressive strain values for the polyester resin are the highest, showing a well defined two-stage behavior: (I) an increase from 0 to 10 kGy and (II) a decrease from 10 to 150 kGy.

The compressive strain analysis can be performed in two parts according to the resin concentration. First, for 38 and 43% of the resin (see Fig. 5) we observe three well-defined stages: (I) an increase from 0 to 10 kGy, (II) a decrease from 10 to 100 kGy, and (III) an increase from 100 to 150 kGy. That is, for both concentrations we have the same behavior; an improvement of 36 and 80% is obtained (for irradiated-PC at 10 kGy for 38 and 43% of the resin, respectively), when comparing with the standard compressive strain data reported in the literature for polyester + PC [1].

We observe a similar behavior (three stages) for both the compressive strength and the compressive strain for PC with 38% of resin content.

In the second range, which is for the samples containing 54 or 58% of the resin, a different behavior is found (Fig. 6). PC with 54% of resin has the same behavior as the compressive strength values (two stages) with a maxi-

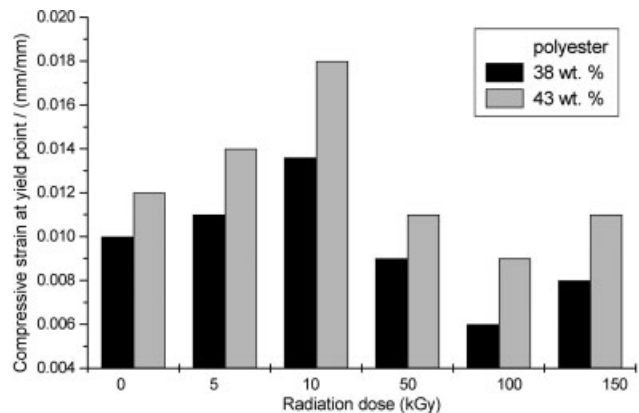


FIG. 5. Compressive strain at yield point for irradiated polyester + PC with 38 and 43 wt% of the resin.

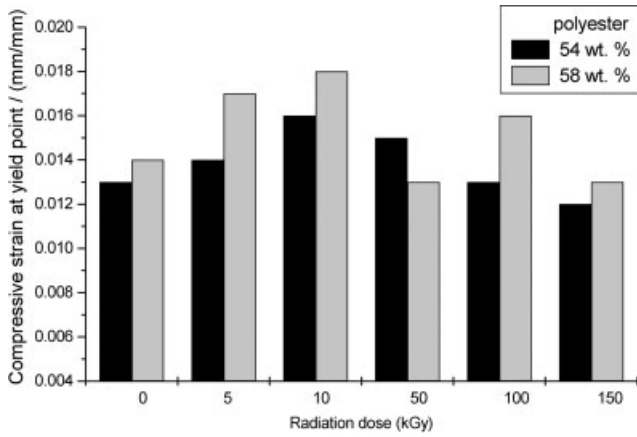


FIG. 6. Compressive strain at yield point for irradiated polyester + PC with 54 and 58% of the resin.

imum compressive strain value at 10 kGy. However, when increasing the resin concentration further to 58%, more stages are present (four) than for compressive strength; nevertheless, a maximum is reached for 10 kGy. It is important to mention that for these two resin percentages, all PC samples have values up to 0.01 mm/mm, which is more than the polyester + PC value reported in the literature.

For the case of the pure polyester resin, the compressive strain increases until a maximum at 10 kGy (0.026 mm/mm), but for higher applied doses the values decrease; see again Fig. 4. Nevertheless, the highest compressive strain values are seen for the polyester resin, with an improvement of 73% with respect to the nonirradiated resin.

COMPRESSION MODULUS OF ELASTICITY

We shall now consider compression modulus of elasticity, E_c . Indeed, a PC is useful because of its high compressive deformation resistance and not too high com-

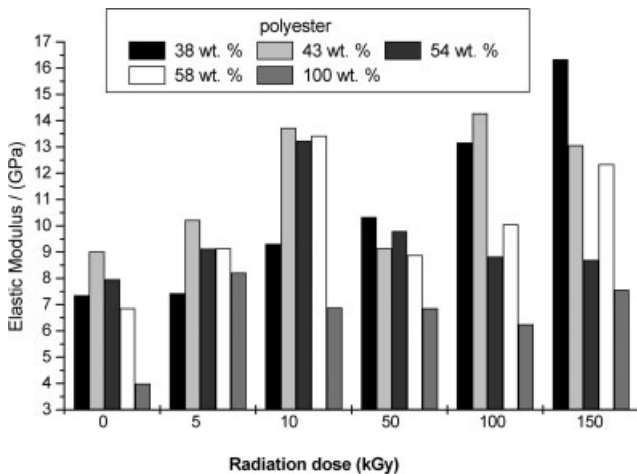


FIG. 7. Compression modulus of elasticity for irradiated polyester + PC at different applied doses.

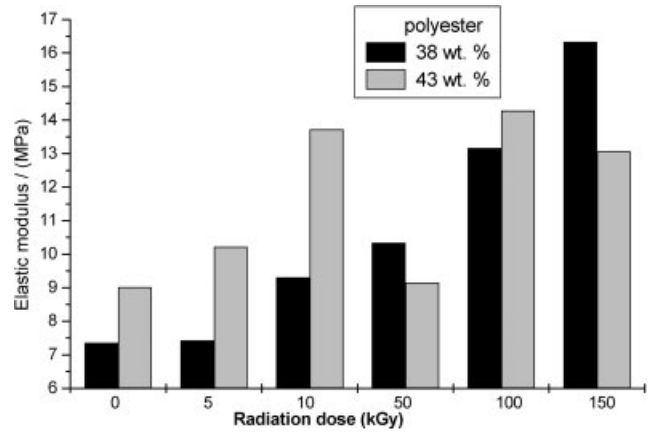


FIG. 8. Compression modulus of elasticity for irradiated polyester + PC with 38 and 43% of the resin.

pressive strains. For the polyester + PC the compression modulus of elasticity values oscillate around 6.7 GPa on the average [1].

In Fig. 7 we show E_c as a function of two parameters: the resin concentration and the applied radiation dose. In general, the values for irradiated-PC are higher than for nonirradiated, covering a range from 7.4 to 16.3 GPa.

For 38% of resin, E_c increases when the radiation dose increases (Fig. 8). The behavior seems different to those observed for compressive strength and compressive strain. The standard values reported in the literature [1] (6.7 GPa) are reached for all PC samples. However in the case of irradiated-PC with 43% of resin (Fig. 8), four stages are present (as compressive strength behavior). There is a simple behavior from 0 to 10 kGy. For higher radiation doses also a periodical behavior is seen here. Thus, concentrations of the resin higher than 38%, the elastic modulus behavior depends strongly on the radiation dose.

E_c values for specimens with 54% of resin follow the same behavior as the compressive strength and strain:

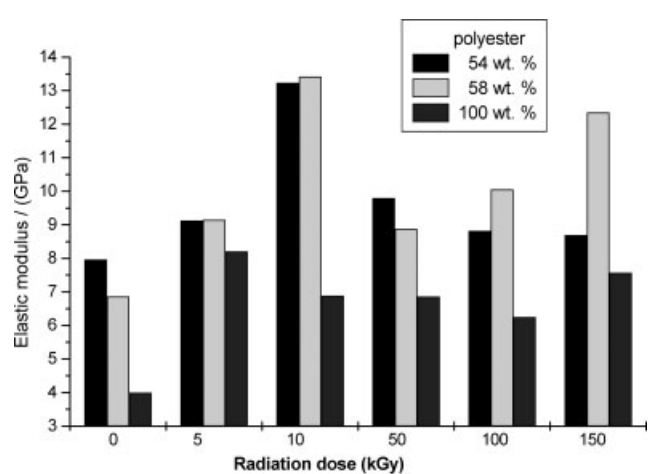


FIG. 9. Compression modulus of elasticity for irradiated polyester + PC with 54 and 58% of the resin.

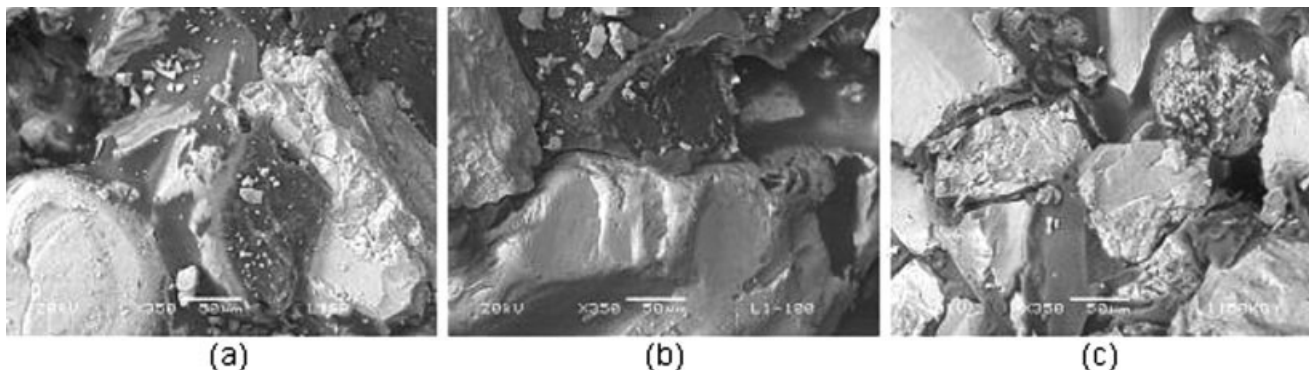


FIG. 10. SEM micrograph of PC with 38% of the resin after compressive test: (a) nonirradiated, (b) irradiated at 100 KGy, and (c) irradiated at 150 KGy.

increases with increases of the radiation dose, reaching a maximum at 10 kGy (13.2 GPa) and going down for higher dosages (Fig. 9). For 58% of the resin, a three-stage behavior is present: (I) an increase from 0 to 10 kGy, reaching a maximum at 10 kGy (13.4 GPa); (II) a decrease at 50 kGy; and (III) an increase for 100 and 150 kGy.

In general, the values for 58% of resin are higher than for the pure polyester resin (identified by 100 number in the Fig. 9). Thus a hardened PC has been obtained, and the influence of the silica sand and the gamma radiation is evaluated.

MORPHOLOGICAL ANALYSIS

The mechanical performance, before and after irradiation with gamma particles, can be connected to the morphology evaluated by SEM.

In Fig. 10 we show the fracture zone after testing the PC with 38% of resin. For nonirradiated PC an analysis of both silica sand and polyester resin was carried out (Fig. 10a). First, sand particles are well visible with sizes below 50 μm , contributing to the material resistance against deformation under a compressive load. Then, the particles tend to disintegrate; the original particles had the diameters of 150 μm on the average. The resin has a homogeneous surface and totally covers the sand particles.

When increasing the irradiation dose, the mechanical features are improved—as we have seen above. However, for the high level radiation, namely 100 kGy, the mechanical parameters decrease. In the micrograph corresponding to 100 kGy (Fig. 10b), we observe less disaggregate sand particles whose sizes are not smaller than nonirradiated PC. Moreover, crack formation in the resin is seen, thus, a separation between the sand particles and the resin occurs. This causes lowering of both the compressive strength and strain values. On the other hand, we get high elastic modulus. At the highest radiation dose of 150 kGy, an evident separation between resin and the particles is seen. Some small particles, with the diameter below 50 μm on the average, are observed (Fig. 10c).

In principle, when the PC has more space between resin and the particles (caused by gamma radiation), the elastic modulus increases but the compressive strength decreases. Moreover, the morphology of the nonirradiated resin shows a homogeneous surface. For higher doses striations are seen (Fig. 11b), apparently a consequence of the resin contraction. Then, if a sand particle is present in a nonirradiated resin matrix, it will be surrounded by the resin. When the radiation has been applied, the resin will start its polymerization, what will cause the following effects: breakdown of the resin (cracks) and resin contraction with pulling out from the sand particles. Moreover,

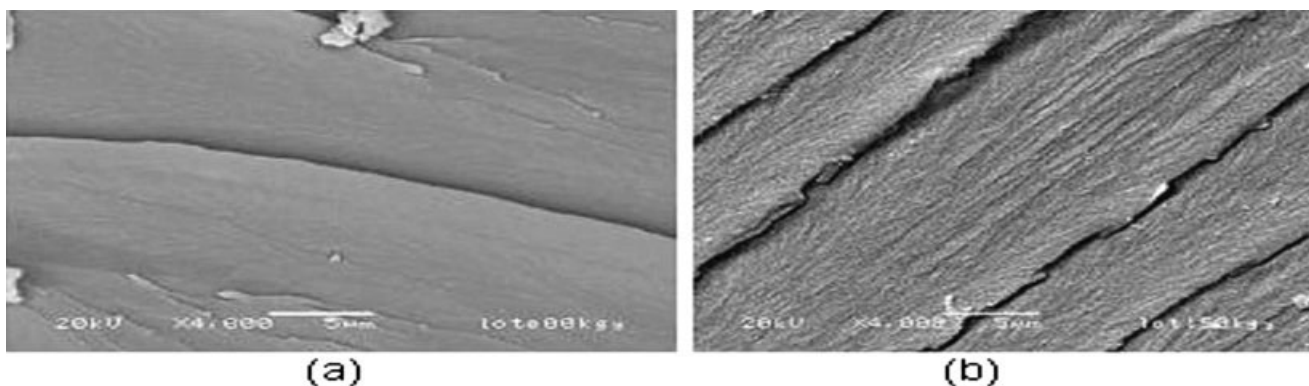


FIG. 11. SEM micrograph of the polyester resin: (a) nonirradiated, (b) irradiated at 150 KGy.

as already reported by Ismail et al., the interaction between cement constituents and polyester formed in the pores under the effect of gamma irradiation contribute to an improvement in the mechanical strength [7].

CONCLUDING REMARKS

As expected, mechanical properties depend on both the resin concentration and the applied dose. For nonirradiated PC, all sand particles are covered by a homogeneous resin. Under irradiation with gamma particles, we observe crack formation of the resin and the separation between sand particles and resin, what causes changes in the mechanical properties. This work is part of a larger project focused on the effects of the gamma radiation on polymers and concrete, including hydraulic and PC [20, 21].

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