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Unsaturated Polyester-Based Polymer Concrete Containing Recycled Cathode Ray Tube Glass Aggregate

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Abstract: Polymer concrete (PC) is a composite construction material that boasts several advantages, such as lightness, low water permeability, high resistance to corrosive environments, and chemical degradation. Consequently, it has recently attracted interest as an alternative material to the traditional ones for several civil applications. In this study, unsaturated polyester resin was considered the matrix phase of PC. Aimed to produce green PC, the commonly dispersed phase of natural aggregate was totally replaced by recycled glass aggregate (RGA) deriving from cathode ray tube (CRT) glass waste. Fine and coarse fractions of non-hazardous CRT glass were considered in different ratios. Chemical and physical analyses were carried out through XRF, particle size distribution and microstructural analysis to characterize RGA. The influence of RGA particle size and percentage on PC performance was investigated by microstructural analysis and aggregate packing, chemical resistance, water absorption, and mechanical analyses, such as bending, impact, and scratch test. Using solely the coarse fraction of RGA led to the manufacturing of a green PC with similar performance to the traditional PC and in addition lower in density. The PC quality mainly depended on the matrix crosslinking which, for PC containing fine RGA, was promoted by adding 4 wt% of silane coupling agent.

Keywords: polymer concrete; recycled aggregate; glass waste; cathode ray tube waste; unsaturated polyester; coarse fraction; fine fraction; construction material; environmentally friendly

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1. Introduction

Polymer concrete (PC) is a composite construction material composed of a polymer matrix and natural inorganic aggregate. Its use has recently increased for civil engineering applications, i.e., bridge decks, concrete crack repairing, pavement covering, hazardous waste confinement, sewage pipes, and decorative building elements. Indeed, compared to the Portland cement-based products traditionally involved for these applications, PC displays higher mechanical performances, lightness, corrosion resistance, resistance to chemicals, excellent bonding to different substrates, lower shrinkage and lower water permeability. In addition, using PC appears particularly advantageous for fast consolidation applications [1,2].

Several polymer matrices have been investigated for PC manufacturing, including unsaturated polyesters, epoxies, acrylics, and polyurethanes [3–6]. Among these, unsaturated polyester resin is the most commonly used thermosetting matrix due to its chemical resistance, thermal stability, mechanical properties, availability, and low cost [1,2]. The production process of PC involves an exothermic chemical reaction, called crosslinking, acted by the unsaturated groups of the polymer phase [7,8]. This process is called curing and results in creating a thermosetting polymer matrix for PC, which has the primary role of binder and stress distribution between aggregate particles.

Inorganic aggregate is generally used between 60% and 95% by weight and can be of different particle sizes and nature, e.g., silicate, limestone, quartz, clay, granite, etc. [1].

For example, sand and gravel are used to produce piping and structural components in amounts between 60 wt% and 80 wt%, while quartz sand is used in percentages up to 95 wt% for kitchen tops, desktops, and countertops. Moreover, additional components can also be considered in the mix to possibly improve PC properties: fly ash, silica fume, natural and synthetic fibers, nanofiller, etc. [1,2,9–11].

In an attempt to reduce the environmental impact of PC, the possibility of using recycled aggregate, both inorganic and organic type, has been investigated [1,2,12].

Several studies have focused on the use of glass waste and glass powder waste in PC due to the similarity of chemical composition between glass and natural quartz sand [13–16]. Indeed, evidence of lower porosity and lower water absorption has identified recycled glass aggregate (RGA) as a highly suitable aggregate for PC, which in contrast is highly sensitive to the moisture content of natural aggregate [1,13]. However, high contents of recycled glass powders have resulted in worsening mechanical properties due to the incomplete covering by resin of the sharp and highly compacted particles [13]. In this context, the use of a polyester matrix over cement has been recommended to create better adhesion with RGA and avoid durability issues related to the alkali-silica reaction of cement-based concrete [13].

Among all RGAs, cathode ray tube (CRT) waste valorization has recently become a hot topic. As major advances in electronics have recently occurred, a large amount of obsolete electronic devices to be discarded has become a key problem to be addressed, especially in highly developed countries [17,18]. The CRT component accounts for about 70 wt% of old-fashioned TVs and monitors and includes mainly glass (85 wt%) [19]. The treatment and separation of the CRT component from televisions and monitors generate the so-called CRT glass waste. This may have a different composition depending on the treated glass type that composed the CRT. A major part of CRT glass waste is barium (9–11 wt%)—strontium (8–10 wt%) glass and derives from the treatment of the panel part. The CRT waste from panels is non-hazardous waste and can be directly reused or recycled. Then, the second most present glass waste is lead glass (18–20 wt% PbO) which comes from the cone/funnel section, i.e., the rear part of the panel. Finally, high lead content and low melting lead glasses are derived from the neck and frit parts, i.e., the enveloping electron gun and connection between panel and cone, respectively [18].

Actually, most efforts have been spent on solving the management of lead glasses from CRT glass waste. This has been pursued by considering the closed-loop recycling of this waste to produce new CRT lead glasses, while several attempts have also considered it as a fluxing agent for the high-temperature manufacturing of ceramics. However, lead glass from CRT waste is mostly involved as recycled aggregate in cement-based materials where the requirements in terms of lead content are less strict [16–18,20].

However, RGA from CRT glass waste has not yet been considered for PC applications, and those from CRT panel glass have not gained interest. Since CRT glass waste from panels is non-hazardous waste, its use has no drawbacks and it can be potentially considered as an additional green component for applications in the construction sector. Moreover, it appears potentially suitable for use in PC after a preliminary treatment aimed to control cleanliness, particle size, and homogeneity. Indeed, even if it is amorphous and not crystalline, its chemical composition is the same as natural quartz sand used for quartz-PC.

In this framework, the current work assessed for the first time the feasibility of using RGA from CRT panel waste as a complete replacement of natural aggregate in unsaturated polyester-based polymer concrete. Therefore, a valorization of non-hazardous CRT waste was proposed and the increasing demand for sustainability of construction material was addressed.

An RGA from an Italian company specialized in recycling glass waste from CRT was considered. Considering the particle size distribution of the RGA under consideration, it was decided to design green PCs for kitchen countertops, in which approximately 95 wt% natural quartz aggregate of the same particle size is generally used [21,22]. A reference PC was produced with 8 wt% of unsaturated polyester resin and 92 wt% of natural quartz

aggregate, whereas four green PCs were designed considering the use of both coarse and fine RGA fractions. Increasing contents (i.e., 0 wt%, 10 wt%, 20 wt%, 30 wt%) of fine RGA were combined with coarse RGA to reproduce the particle size distribution and packing factor of the natural reference aggregate.

The involved RGA was chemically and physically characterized and the influence of its use in PC was investigated through density measurements, packing factor, microstructural analysis, chemical resistance, and water absorption. In addition, bending, impact, and scratch test were performed on the green PCs, and the results were compared with those of the reference PC.

2. Materials and Methods

A commercial unsaturated polyester resin by Carlo Riccò (Correggio, RE, Italy) was considered. This contained 5 ± 1% methacrylate and 27–31% styrene and was accelerated by a cobalt compound and catalyzed at room temperature with methylethylketone peroxide. Gel time, exothermic peak, and curing time for the unsaturated polyester resin were 20–38 min, 185–205 °C, and 28–53 min, respectively.

A commercial quartz sand was involved in producing a reference unsaturated polyester-based PC for top production.

RGA from CRT glass waste was considered in two particle sizes, namely, coarse and fine fractions, and used as total substitution of natural aggregate to produce environmentally friendly unsaturated polyester-based PC. These fractions were derived from the dry grinding process of CRT glass waste performed by Stena Technoworld (Angiari, VR, Italy).

Five PC mixes were designed by considering 92 wt% of aggregate and 8 wt% of unsaturated polyester resin. A traditional quarzitic polymer concrete was considered as reference, named PCRef, and was produced by considering a commercial quartz natural sand. Then, four environmentally friendly PCs containing RGA were considered. PC0 involved the use of solely coarse RGA fraction, whereas PC10, PC 20, and PC30 also considered the addition of fine RGA fraction (Table 1).

Table 1. Composition of the PCs under investigation.

Sample	PCRef	PC0	PC10	PC20	PC30
Polymer matrix (wt%)	8	8	8	8	8
Natural sand (wt%)	92	-	-	-	-
Coarse RGA (wt%)	-	92	82	72	62
Fine RGA (wt%)	-	-	10	20	30

The aggregates and resin were mixed, cast at 25 °C in a 100 × 100 × 25 mm^3 metal mold, pressed (11 bar, 25 °C, 1 h) using a hydraulic press, and cured at 80 °C for 4 h. A further post-curing of 28-days at laboratory conditions was considered before testing. Vinyltriethoxysilane was also considered, 1 wt% and 4 wt% of aggregate, as coupling agent between polymer matrix and aggregate.

Chemical, physical, and morphological characterization of aggregates was performed on representative portions obtained from a quartering process. X-ray fluorescence (XRF, Phillips PANalytical PW3710, Malvern Panalytical Ltd., Malvern, UK) analysis was involved in defining RGA composition and the presence of hazardous components. A laser particle analyzer (Malvern MASTERSIZER, Malvern Panalytical Ltd., Malvern, UK) allowed describing of the particle size distribution of natural quartz sand and RGA, while their morphological description was achieved through scanning electron microscopy (ESEM, ESEM-Quanta 200 Fei Company Oxford Instruments, Abingdon, UK). Energy dispersive spectroscopy (X-EDS, Oxford INCA-350, Oxford Instruments, Abingdon, UK) was involved in the microanalysis detection and support of XRF results.

Moreover, the key properties of the PC cubic samples containing RGA were identified and compared to those of PCRef. Five 100 × 100 × 25 mm^3 cubic samples for each PC mix were considered for this purpose.

Water absorption was computed as the ratio of absorbed water after 24 h of immersion at 25 °C in distilled water and dry initial mass. While chemical resistance was evaluated by measuring the weight loss caused by the immersion after 24 h at 25 °C in hydrochloric acid (10% wt/wt) and potassium hydroxide (10% wt/wt).

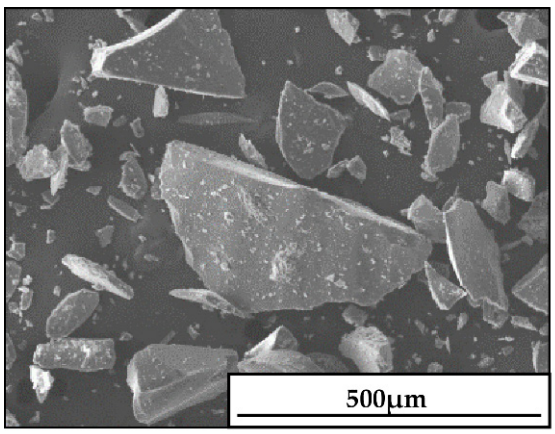
Real and apparent density were measured using a helium pycnometer (Micrometrics ACCUPYC 1330, Micromeritics instrument) and envelope density analyzer (Geopyc 1360, Micrometrics instrument, Norcross, GA, USA), respectively. Then the packing factor, or the volume of the structure filled by particles, was computed as the ratio of apparent to real density and ranging from 0 (bad compaction) to 1 (excellent compaction).

Mechanical testing was achieved by performing impact tests (ASTM D3029, Dynatup 9250HV, Instron, Norwood, MA, USA) and three points bending tests (UNI EN ISO 178, Universal Testing Machine Instron 3340). Then, a scratch test was performed (ISO 20502, Micro-Combi Tester, Anton Paar) and the critical scratch load was registered. Finally, a microstructural analysis (ESEM-Quanta200, FEI Company Oxford Instrument) was performed on residual portions of PC samples from mechanical testing.

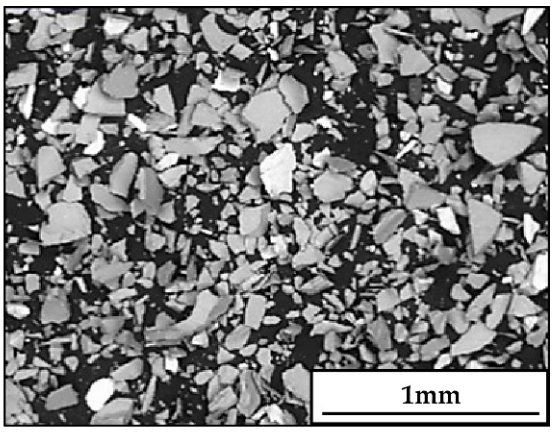
3. Results and Discussion

3.1. Characterization of RGA

XRF analysis classified the RGA from CRT glass waste as barium glass (56.9 wt% SiO_2, 12.9 wt% Na_2O, 8.5 wt% SrO, 8 wt% BaO, 7.3 wt% K_2O, 3.8 wt% Al_2O_3) with a negligible concentration of PbO (0.02 wt%). This was also confirmed by the microanalysis of Figure 1c. Figure 1 shows the flat shape and sharp morphology of coarse (Figure 1a) and fine (Figure 1b) RGA, which is attributable to the dry grinding process of CRT waste glass. This evidence was also confirmed by literature and is principally responsible for preferential orientation of particles into the matrix and inhibition of wettability with the polymer matrix, especially for fine ones [13]. Conversely, the quartz sand (99.98 wt% SiO_2) displayed a rounded morphology.

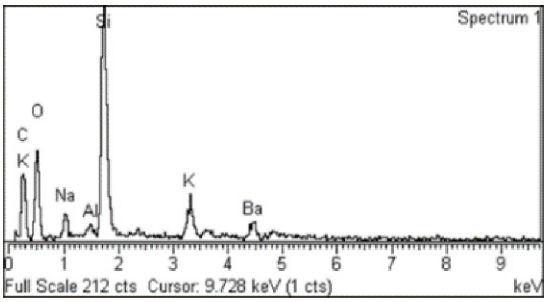


(**a**)



(**b**)

Figure 1. *Cont.*



(**c**)

Figure 1. ESEM and X-EDS characterization of RGA: (**a**) micrograph of coarse RGA, (**b**) micrograph of fine RGA, (**c**) X-EDS microanalysis.

As reported in Figure 2, the coarse RGA showed a monomodal (200 µm peak) particle size distribution compared to the bimodal one of quartz sand (300 and 35 µm peaks). Since fine RGA owned a monomodal particle size distribution with a peak at 20 µm, the combination of fine and coarse RGA allowed reproducing a particle size distribution similar to quartz sand; this was more emphasized for increased fine RGA content (Figure 2). For all the involved PCs, the coarse fraction contributed mainly to composite structure and fine fraction to compaction and porosity reduction.

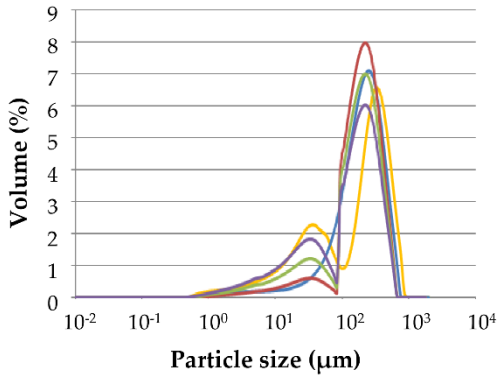


Figure 2. Particle size distribution of quartz sand (yellow), RGA mix for PC0 (light blue), RGA mix for PC10 (red), RGA mix for PC20 (green), RGA mix for PC30 (violet).

3.2. Characterization of PC

Increasing the fine RGA fraction promoted a higher compaction level and a reduced porosity, as displayed by SEM microscopy in Figure 3. Accordingly, density and the packing factor increased by passing from PC0 to PC30 (Table 2); precisely, PC30 reached a packing value equivalent to PCRef. On the other hand, a decrease of about 32% was observed when replacing natural aggregate with coarse RGA; this was mainly attributable to the interstitial voids that for PCRef were filled by fine particles and for PC0 by resin.

Due to the low resistance to alkali of unsaturated polyester resin, the chemical resistance test showed that all PCs owned a better resistance to HCl etching, so lower weight loss, than to KOH (Figure 4). Moreover, it was observed that higher content of fine RGA increased the weight loss, especially for alkali etching. In contrast, PCRef and PC0 displayed similar and the best chemical resistance and water absorption values. These values were also comparable to weight loss after HCl and KOH etching of pure unsaturated polyester resin; 0.04 and 0.45, respectively.

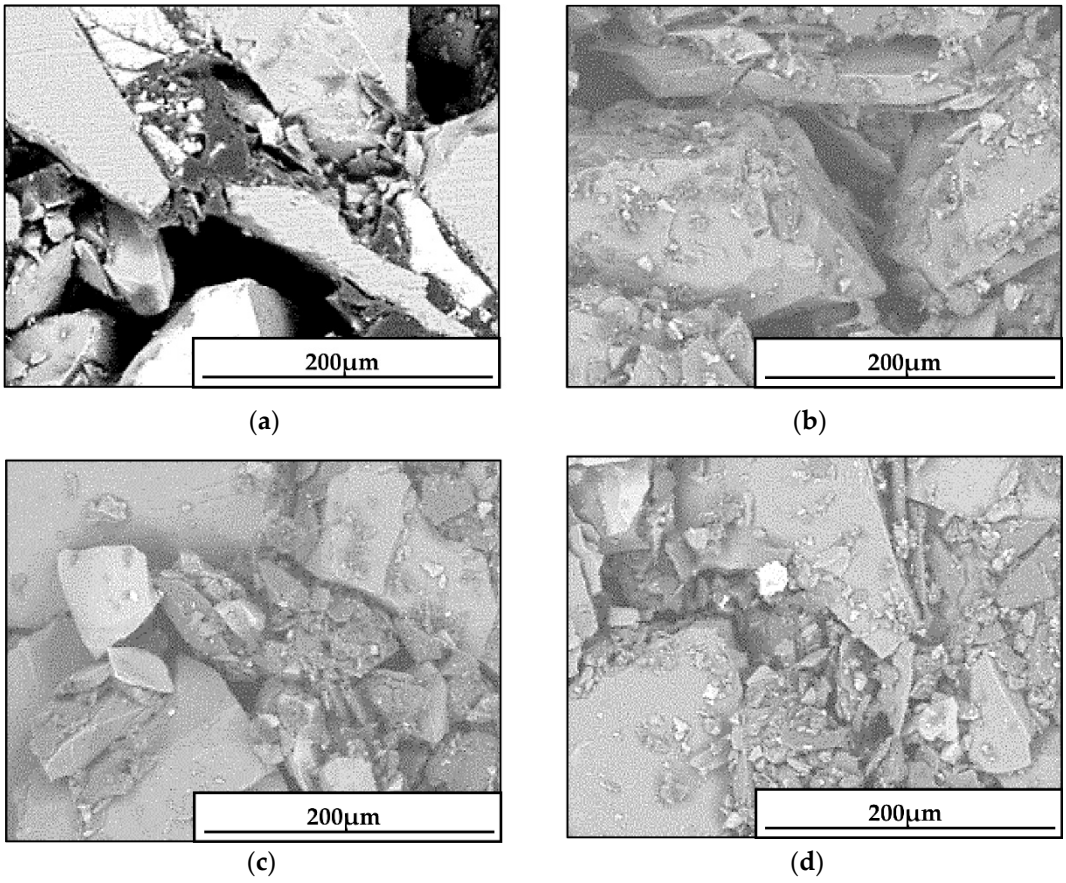


Figure 3. RGA compaction at increasing content of fine fraction: (**a**) 0 wt%, (**b**) 10 wt%, (**c**) 20 wt%, (**d**) 30 wt%. SEM micrographs at 400× magnification.

Table 2. Apparent density and real density of PCs.

Sample	PCRef	PC0	PC10	PC20	PC30
Apparent density (g/cm^3)	2.91	1.98	1.99	2.08	2.81
Real density (g/cm^3)	2.93	2.60	2.57	2.57	2.87
Packing factor (-)	0.99	0.76	0.78	0.81	0.98

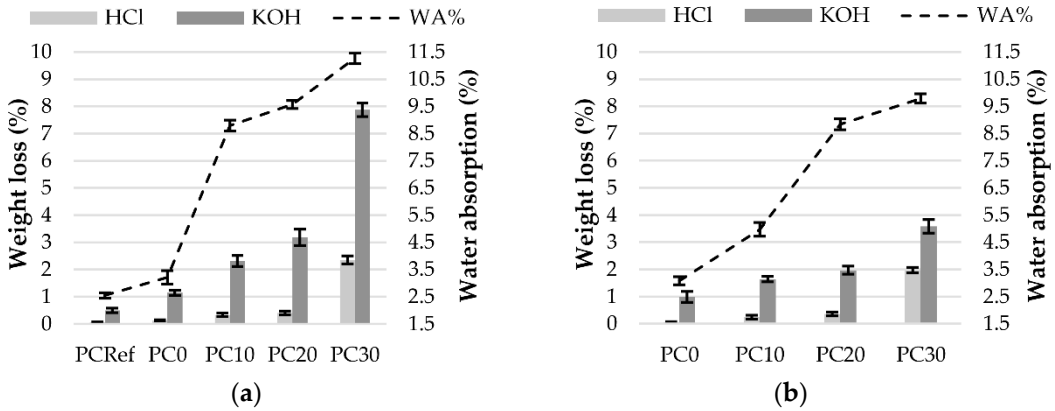


Figure 4. Correlation between chemical resistance (weight loss) and water absorption at increasing content of RGA fine fraction considering: (**a**) 1 wt% silane, (**b**) 4 wt% silane coupling agent. Error bars represent standard deviation.

Contrarily to enhancing packing factor (Table 1), the increase of fine RGA fraction also increased the water absorption (Figure 4). Though high compaction should prevent water absorption, the interaction between sharp particles and resin must be considered for PCs containing RGA [13]. In addition, a higher filler content would require a higher resin content to properly cover the greater surface area and reduce interstitial voids [1]. Accordingly, data from water absorption and chemical degradation tests pointed out that the fine RGA fraction inhibited the correct penetration of resin through the aggregate solid structure, thus the good achievement of the crosslinking mechanism and sealing of residual pores. On the other hand, the rounded shape of quartz sand did not influence this mechanism and promoted the binding action of the resin, which resulted in PC's waterproofing and higher chemical resistance. In conclusion, chemical resistance and water absorption were optimized by the rounded shape of quartz sand or by considering solely coarse RGA fraction.

The increase of silane coupling agent from 1 wt% (Figure 4a) to 4 wt% (Figure 4b) improved the chemical resistance and reduced the water absorption of PCs containing RGA. Thus, it enhanced the compatibility between phases, the crosslinking of unsaturated polyester resin and reduced the open porosity. Remarkably, the most significant improvements were observed for alkali chemical resistance (KOH). Furthermore, PC10 displayed a reduction by 44% in water absorption due to the promotion of greater interaction between glass and resin.

Results from the mechanical characterization (Figure 5) confirmed that the worse crosslinking of unsaturated polyester resin, due to the increasing presence of fine RGA fraction, also decreased the mechanical performances of PC. The dependence of good PC performance on the nature of the aggregates and the chemical/physical interaction between aggregates and matrix has also been observed in the literature [1].

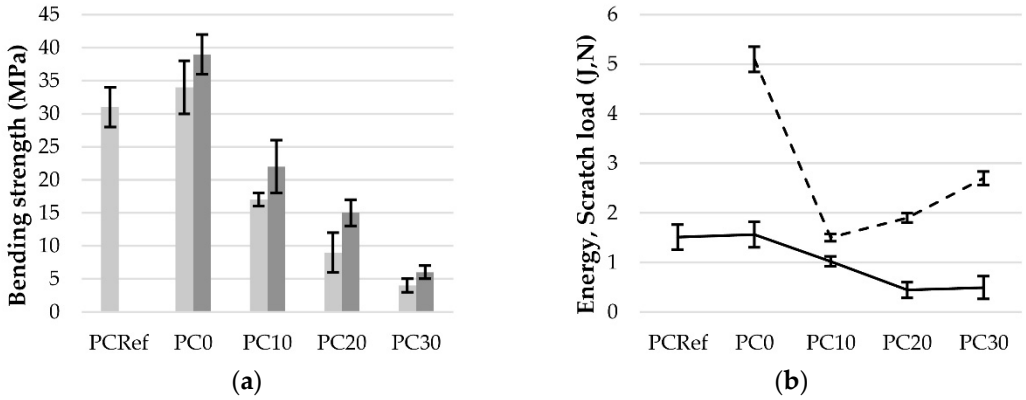


Figure 5. Mechanical characterization results for PCs: (**a**) Maximum bending stress for PCs containing 1 wt% silane coupling agents (light grey) and 4 wt% silane coupling agents (grey), (**b**) maximum impact energy (continuous line), critical scratch load (dashed line). Error bars represent standard deviation.

The flat shape of fine RGA and its high compaction level along preferrable directions within the matrix might also have contributed to particles agglomeration and their incomplete covering by the resin. This involved the creation of preferred breaking points and the pullout of particles, thus lower mechanical performance (PC10, PC20, PC30). Conversely, the use of low fine content or solely coarse fraction (PC0) has been demonstrated to promote a reinforcing action and a sort of crack-bridging effect that changes the failure mode to the failure of brittle aggregates, thus enhancing the mechanical strength of PC. Indeed, the flat shape of RGA implies a preferential orientation of aggregate perpendicularly to the impact or bending loading, whereas the complete wettability of particles is assured by not-excessive particle compaction [13].

On the other hand, the rounded shape of natural aggregate assures ease and complete covering of particles by the resin. However, a worsened performance of PCRef compared to PC0 is attributable to the easier debonding of rounded particles from the matrix during mechanical loading (Figure 5a) [13].

The addition of higher coupling agent contents enhanced the overall bending performance (Figure 5a). Thus, it was supported that good chemical, physical, and mechanical properties could only be reached by a good matrix crosslinking instead of a better compaction rate. Remarkably, PC0 displayed impact energy similar to PCRef and the best scratch critical load among the PCs with RGA (Figure 5b).

4. Conclusions

In this work, the feasibility of producing green unsaturated polyester-based polymer concrete (PC) for kitchen countertops was assessed considering the replacement of natural aggregate with recycled glass aggregate (RGA) from cathode ray tube (CRT) glass waste.

Both coarse and fine RGA fractions were considered as a total substitution to natural quartz sand (92 wt%). So, four green PCs were designed by considering different coarse to fine RGA ratios, namely, 92:0, 82:10, 72:20, and 62:30 wt%, and the same resin content (8 wt%). The innovative valorization of non-hazardous CRT glass waste from panels avoided the well-known problem of lead contamination related to other components of CRT devices and was proposed as a viable solution to the emergent issue of CRT waste management in landfills. In addition, the use of unsaturated polyester resin was suggested for the first time as an alternative to cement matrix because of its potentially better adhesion with glass aggregate and non-sensitivity to alkali-silica reaction.

It was observed that the gradual increase in the fine RGA content allowed better reproduction of the particle size distribution and packing factor of natural quartz aggregate, but it also gradually worsened the chemical resistance, water absorption, and mechanical performance of the PC. In fact, fine RGA particles inhibited the correct crosslinking of unsaturated polyester resin through the solid structure of aggregates, mainly due to its sharp morphology and high compaction. This problem was partially faced by considering 4 wt% of silane coupling agent, which enhanced the compatibility between RGA and polymer matrix, thus promoting a better crosslinking of the thermosetting resin. Remarkably, the use of a 4 wt% coupling agent was more effective in reducing alkali etching, which is a well-known issue for unsaturated polyester resin.

On the other hand, PCs containing low contents of fine RGA fraction resulted in lightness and good chemical, physical, and mechanical properties. Significantly, the PC with solely coarse RGA displayed scratch critical load, chemical resistance, and water absorption similar to the reference PC sample and even exceeded it in bending strength. Thus, the PC performance was mainly related to the good cross-linking of the polymer matrix and covering of the aggregate by resin, which were promoted by low aggregate compaction and the addition of a coupling agent.

In conclusion: it was demonstrated that RGA from CRT glass panel waste could be considered as an alternative to natural aggregate in PC production with consequent environmental and economic benefits. Future works should investigate how to further enhance the interaction between fine fraction aggregate and resin phase. This could be achieved by considering a rounded morphology of fine RGA to be obtained through a wet grinding process. In addition, a variation of the resin composition and a different inorganic fine fraction could be involved to have both good compaction and good crosslinking of the polymer matrix.

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