

An integrated recycling approach for GFRP pultrusion wastes: recycling and reuse assessment into new composite materials using Fuzzy Boolean Nets

Ana C. Meira Castro, Joao P. Carvalho, Maria C.S. Ribeiro, João P. Meixedo,
Francisco J.G. Silva, António Fiúza, Maria L. Dinis

Abstract

In this study, efforts were made in order to put forward an integrated recycling approach for the thermoset based glass fibre reinforced polymer (GFRP) rejects derived from the pultrusion manufacturing industry. Both the recycling process and the development of a new cost-effective end-use application for the recyclates were considered. For this purpose, i) among the several available recycling techniques for thermoset based composite materials, the most suitable one for the envisaged application was selected (mechanical recycling); and ii) an experimental work was carried out in order to assess the added-value of the obtained recyclates as aggregates and reinforcement replacements into concrete-polymer composite materials. Potential recycling solution was assessed by mechanical behaviour of resultant GFRP waste modified concrete-polymer composites with regard to unmodified materials. In the mix design process of the new GFRP waste based composite material, the recyclate content and size grade, and the effect of the incorporation of an adhesion promoter were considered as material factors and systematically tested between reasonable ranges. The optimization process of the modified formulations was supported by the Fuzzy Boolean Nets methodology, which allowed finding the best balance between material parameters that maximizes both flexural and compressive strengths of final composite.

Comparing to related end-use applications of GFRP wastes in cementitious based concrete materials, the proposed solution overcome some of the problems found, namely the possible incompatibilities arisen from alkalis-silica reaction and the decrease in the mechanical properties due to high water-cement ratio required to achieve the desirable workability.

Obtained results were very promising towards a global cost-effective waste management solution for GFRP industrial wastes and end-of-life products that will lead to a more sustainable composite materials industry.

Keywords:

GFRP pultrusion wastes, Mechanical recycling, Concrete-polymer composites, Mix design optimization, Fuzzy Boolean Nets

1. Introduction

Pultrusion is one of the most well-known and cost-effective techniques for manufacturing fibre reinforced polymer (FRP) structural components with a constant cross-section in a continuous manner (Bank, 2006; Stewart and Sumerack, 2000). Typically, in the manufacturing process of FRP pultruded profiles, plain glass

(GF), carbon (CF) or aramid (AF) reinforcing fibres are pulled through a thermoset resin bath for impregnation (usually a poly-ester, vinyl ester or epoxy resin), and after wetting process, the reinforcement is allowed to enter into a heated forming die, where it attains the shape of the die cavity and cures. Finally, outside the die, the already consolidated composite part (GFRP, CFRP or AFRP profile) is pulled by a continuous pulling system and then a cut-off saw cuts the part into a desired length. Obtained pultruded FRP profiles are widely used in infrastructures of wastewater treatment plants, as internal or external reinforcement of concrete structures, for retrofitting and rehabilitation purposes of structural elements (Hollaway, 2010) and, more recently, in composite construction systems together with moulded gratings and sandwich panels

(Correia et al., 2011a). Over the last 60 years, the pultrusion manufacturing technique has grown and developed strongly from its conception and first steps in the early 1950' to present as a well-established and efficient industrialized process. However, due to a growing concern regarding the sustainability of composite materials industry and the rising of consumption of resources, technological developments towards a better eco-efficiency, clean production process and sustainable materials management of pultruded FRP profiles are still needed and opportune (Lindahl et al., 2013).

In the actual framework of the pultrusion sector, and in general in that of the composite materials industry, production wastes, non-conform and end-of life products are usually landfilled due to their limited recycling ability even when the thermoplastic-based products are considered (Halliwell, 2006) (Fig. 1).

According to a recent market report of LUCINTEL (2012), a leading global market research firm, the global glass fibre market is expected to reach an estimated \$11.2 billion in 2017, and accordingly, the pultrusion sector will also contribute with its share to this scenario. The increasing production will lead, thereby, to increasing production wastes and, in the near future, to larger amounts of end-of-life products. Hence, cooperation with other companies in order to revalorize by-products and production wastes, and promoting the recycling and the reuse of the recyclates into new added value products are critical and required steps towards a better eco-efficiency performance of this sector. Moreover, due to the more restrictive EU waste management legislation, with increasing landfill taxes and limiting capacity, landfill and disposal will no longer be available solutions (Pickering, 2006). Recycling and reuse will be set for FRP scrap materials; thus FRP producers and suppliers must address this problem if they do not want to risk losing their market share to metal and other more easily recycled materials (Conroy et al., 2006; Halliwell, 2010).

Though, two distinct and reliant issues must be solved before to efficaciously proceed with the recycling approach. The first issue relies on the best recycling process for these materials and the second one concerns the end-use application for the obtained recyclates. Both matters are mutually interdependent and must take into account several economic issues in order to reach to a global cost-effective waste management solution.

Under this scope, the current study is aimed at assessing an integrated recycling approach for GFRP pultrusion wastes that embraces both the recycling process and the end-use application for the recyclates. Among the several available recycling techniques for thermoset based GFRP products the most suitable one for the intended use is selected, and a novel application for the obtained recyclates, as aggregate and reinforcement replacement for a concrete like composite, is developed. Decision-making process that endorses the choices applied in this study is supported by the state of the art on existing recycling methods and related end-use applications briefly reported in the next subsection.

1.1. Available recycling techniques for thermoset composite materials and related end-use applications for the recyclates

Presently, there are several available processes that can be used to get some value from thermoset FRP waste materials: incineration, thermo and/or chemical recycling methods, and mechanical recycling processes. The most popular is incineration that allows some energy recovering from the heat produced during the combustion process due to the high calorific power of FRP materials. However, in general, incinerator facilities charge more for incinerating FRP wastes because both the high calorific content and the toxic emissions tend to overload the system, meaning they cannot process as much domestic waste (Conroy et al., 2006). Additionally, the air pollution resulting from FRP scrap incineration must also be considered.

For fibre and partial energy recovering, thermo-chemical decomposition processes could be applied. The most common thermal process is pyrolysis which consists on heating the scrap material in an inert atmosphere in order to recover the polymer material as oil. This kind of atmosphere prevents combustion, and as result the air pollution effects are less harmful in this process than in incineration. Another advantage is that the recovered oil can be used either as fuel or be refined to regenerate resin feedstock chemicals. As limitation of this technique, the surface fragilities induced by the thermal stress on the recovered fibres, reducing thus its original strength have been reported (Pimenta and Pinho, 2011). Oxidation in fluidised bed is another thermal process for FRP recycling and it consists in combusting the polymer matrix in a hot and oxygen-rich flow. Recovered fibres by this process are clean and show very little surface contamination by char deposition; though, strength and fibre length degradation also occur as stated in Pickering et al. (2000) and Pickering (2006).

The chemical methods of recycling involve dissolution of the resin by means of chemical products and are based on a reactive medium (e.g., catalytic solutions and supercritical fluids) under low temperature (Morin et al., 2012). Being a thermal stress-free process allows the fibres to retain most of their original strength. Though, this method involves the use of hazardous solvents and, additionally, it requires the previous granulation of scrap material in order to improve the specific surface, which causes length reduction of recovered fibres. Reduced adhesion to polymer matrix in posterior applications is another common drawback of chemical recycling methods (Pimenta and Pinho, 2011).

Mechanical recycling, with size reduction by shredding, crushing or milling processes, is another option mainly considered for fibre reinforced composite materials in which reinforcing fibres have a relative low economic value such as GFRPs. This last process shows significant environmental and economic advantages when compared to the previous ones. In fact, mechanical size reduction does not produce atmospheric pollution by gas emission or water pollution by chemical solvents effluents, and does not require such



Fig. 1. Samples of production wastes and non-conform products of the pultrusion industry (courtesy of Alto, Perfis Pultrudidos Lda.).

sophisticated, and expectably expensive, equipment like the ones that are required in the other processes. As drawbacks, safety issues (risk of ignition during shredding process due to the presence of catalyst plus promoter, eventually not consumed during polymerisation process), and the lower value of the final product (a mix of powdered and fibrous material), can be argued. Nevertheless, GFRP products obtained by pultrusion process do not contain promoter, only initiator, as polymerisation reaction is induced by temperature; hence, risk of fire during mechanical recycling process of these materials is avoided. Guaranteeing that viable markets outlets exist for the recyclates, mechanical recycling could be considered as the most cost-effective recycling technique, at least for relatively low-cost and clean GFRP waste materials proceeding from promoter-free manufacturing processes (Palmer et al., 2009; Pickering, 2006).

Mechanically recycled GFRP wastes remain, however, mired by the scarceness of cost-effective end-use applications and clearly developed recycling routes (logistics, infrastructures and recycling facilities) between waste producers and potential consumers for the recyclates. Presently, in order to solve this issue, new end-markets with added value for the GFRP recyclates are required.

Regarding this subject, over the last 20 years several end-use applications were envisioned and investigated for mechanically recycled thermoset GFRP wastes or recovered glass fibre wastes:

i) filler material for artificial wood (Demura et al., 1995), high density polyethylene plastic lumber (George and Dillman, 2000), rubber pavement blocks (Itoh and Kaneko, 2002), dense bitumen macadam (Woodside et al., 2003), and bulk or sheet (BMC/SMC) moulding compounds (DeRosa et al., 2005), ii) reinforcement for wood particleboard (Reynolds et al., 2004) and soils (Ahmad et al., 2012; Mujah et al., 2013); and iii) core material for textile sandwich structures (Adolphs and Branca, 2001). Most of them have not succeeded for one or both of the following reasons: a) tendency of the recyclate addition to negatively affect the mechanical properties of final composite; and b) negative cost balance, where mechanical recycling and sorting operational costs outweighed the market value of the virgin product (chopped glass fibres and calcium carbonate) (Halliwell, 2006; Palmer et al., 2009).

The most extensive research work in this field has been carried out on Portland cement concrete in which mechanically recycled GFRP waste, and more rarely CFRP waste, have been incorporated either as reinforcement, aggregate or filler replacement (e.g., Asokan et al., 2009, 2010; Correia et al., 2011b; Osmani and Pappu, 2010; Tittarelli and Moriconi, 2010; Tittarelli and Shah, 2013). In addition to the environmental benefits, and as function of specific mix design formulation, reported added values include slight to strong decreases of permeability with subsequent improved durability (Asokan et al., 2010; Correia et al., 2011b; Tittarelli and Moriconi, 2010; Tittarelli and Shah, 2013), less drying shrinkage (Asokan et al., 2010; Tittarelli and Moriconi, 2010), improved workability and reduced risk of cracking induced by restrained shrinkage (Tittarelli and Shah, 2013), and a global cost reduction of raw materials. In some particular cases, for lower sand replacement ratios, slender increases on compressive, splitting tensile and/or flexural strengths were observed (Asokan et al., 2010). However, most of the times some undesirable features were noticed such as significant losses in the mechanical properties (mainly due to high water-cement ratio required to achieve the desirable workability) (Asokan et al., 2009; Correia et al., 2011b; Tittarelli and Moriconi, 2010; Tittarelli and Shah, 2013), higher wear loss (Correia et al., 2011b) and weak adhesion at recyclate-binder interface. Moreover, depending upon glass fibre nature, some incompatibility issues derived from alkali-silica reaction may even occur (Tittarelli and Moriconi, 2010).

These limitations, by and large resultant from the use of a cementitious binder as matrix, might be avoided using a cement-less concrete as host material like polymer-based concrete (PC) materials.

PC materials have gained an increasing research interest due to their wide range of possible applications in civil construction (Bhutta and Ohama, 2010; Fowler, 2007). In this class of materials, a thermoset resin is used as binder of natural or artificial aggregates, replacing the paste of Portland cement/water of conventional hydraulic concretes (ACI, 2009). The initial applications of PC during the late 1950's were the production of building cladding and cultured marble products. However, the excellent properties exhibited by these materials rapidly promoted the spread of its end-use applications. Its fast curing, excellent bond to concrete and steel reinforcement, high strength to weight ratio, good damping properties and high resistance to chemical, frost and weathering agents attack (Fowler, 2007; Ribeiro et al., 2002, 2004, 2009), made it a very attractive material for overlays and industrial floorings, precast industry and for repair purposes (Bhutta and Ohama, 2010). Though, currently, the main asset of PC materials over conventional concretes is their great ability for incorporating recycled waste products, mainly due to the hermetic nature of resin matrix.

Recycling and waste encapsulation constitute nowadays an emerging branch market for PCs. Most of the successful applications reported involve either industrial by-products or recyclates derived from end-of-life products. Industrial wastes, such as fly ash (Rebeiz et al., 2004), slag, wood shaves, cork powder and cork granulates (Nóvoa et al., 2004), tire rubber (Bignozzi et al., 2000), marble rejects (Barrera et al., 2013), contaminated foundry sands (Reis and Jurumenha, 2011), plastic chips and plastic granulates proceeding from milled waste electrical cables (Bignozzi et al., 2000), as well as crushed end-of-life PC products (Yeon et al., 2011), have been successfully used for replacing or partially replacing the filler and mineral aggregate components in PC materials.

1.2. Research significance

Despite the relative large amount of research work undertaken on recycled wastes in polymer based concretes, so far and not taking into account the on-going research of the present research team, no studies have been focused on the incorporation of FRP recyclates into polymer concrete (PC) materials. This approach seems to be very promising towards a cost-effective end-use application for mechanically recycled GFRP wastes and will be followed in the present study.

Hence, the integrated recycling approach here proposed encloses the mechanical recycling of the GFRP pultrusion wastes and their incorporation as fine aggregate and filler replacement into polymer based concrete materials, more specifically into polyester polymer mortars (PM). In order to meet the criteria of cost-effectiveness, the new application for the recyclates must create a higher value; hence, in the mix design process of the new GFRP waste based PM the effect of several material factors (e.g., recyclate content, recyclate morphology or size grade and the addition of an adhesion promoter) are taken into account in an attempt to optimize the mechanical responses of the final composite. Optimization process is achieved by means of a Computational Intelligence method, the Fuzzy Boolean Networks (FBN), which are universal approximators with excellent generalisation capabilities that are able to predict the response of parameter-dependent systems.

Computational Intelligence methods other than FBN have been extensively used, especially in the last decade, to analyse, model and predict mechanical properties such as the compressive strength or the elastic modulus for different formulations of

composite and cementitious based materials. Techniques such as artificial neural networks (Kim et al., 2004; Lee, 2003), evolutionary algorithms (Jayarama et al., 2009; Nazari, 2013; Tsai and Lin, 2011), fuzzy sets and systems (Bohlooli et al., 2012; Demir, 2005), hybrid systems (Akkurt et al., 2004; Reza et al., 2013) are just a few examples among others.

The aforementioned techniques are not adequate for the present study due to the nature and sparseness of the available trial data (see Section 3.1). Therefore, the FBN, which have been shown to provide good results in similar cases (e.g., Carvalho and Tomé, 2007; Meira-Castro et al., 2011, 2012), were chosen in the present analysis to fine-tune mix design formulation of GFRP waste admixed polymer mortars.

2. Materials and methods

GFRP waste admixed PM specimens were prepared by mixing an unsaturated polyester resin (20% w/w) with different sand aggregates/GFRP waste ratios. Two differently processed GFRP wastes, with distinct size grades, were used as partial substitute for sand aggregates within a range from 0% to 15% in weight of total aggregates. Plain mortar specimens were also casted and tested for comparison purposes.

One of the main common problems reported in several research studies focused on the feasibility of FRP waste incorporation into new composite materials arises from the weak adhesion at recycle-binder interface (DeRosa et al., 2005; Palmer et al., 2009; Wong et al., 2012). In order to prevent this undesirable feature the effect of the incorporation of an adhesion promoter, between resin matrix and aggregates/recyclates mix, was investigated and also considered as a material factor. Hence, a second series of experiments was carried out in which 1% of active silane coupling agent by weight of resin matrix was added to all formulations in analysis. For each series, added value of the recycling solution was assessed by means of flexural and compressive loading capacities of GFRP admixed mortars with regard to unmodified PMs.

2.1. Characterization of raw materials

GFRP waste material was obtained from the shredding of the leftovers resultant from the cutting and assembly processes of GFRP pultrusion profiles during building sites and it was supplied by local pultrusion manufacturing company (Alto-Perfis Pultrudidos, Lda) with headquarters in Maia, Portugal. Currently, these leftovers as well as non-conform profiles and scrap resulting from pultrusion manufacturing process (Fig. 1), which constitute around 7% of total annual production of 40 ton, are landfilled with an estimated cost for the company of 4000V per year. The applied GFRP waste

material was comprised essentially of an unsaturated polyester resin loaded (Aropol® FS3992) with calcium carbonate and reinforced with E-glass roving (4800 Tex), continuous filament mat (25 Tex) and surfacing veils.

Shredded GFRP waste further processed by milling using a Retsch SM2000 Cutting Mill laboratory unit (Fig. 2). Two size grades of ground GFRP waste were obtained using bottom sieves inside the grinding chamber with differently-sized meshes: 2.5 mm square mesh and 1.5 mm trapezoidal mesh. Obtained recycled products, hereinafter designated by coarse (CW) and fine (FW) pultrusion waste, consist of a mix of powdered and fibrous particulate materials with different quantities of varying length of glass fibres as shown in Fig. 3.

GFRP recyclates were characterized in terms of the organic and inorganic fraction contents and particle size distribution. Burning tests carried out on five random samples according to procedure described in Volkswagen AG TL 523 42 technical specification (2003) revealed an average inorganic material content of 71% (w/w), corresponding to glass fibres (55% w/w) and calcium carbonate (16% w/w), and an average resin content of 29% (w/w). Particle size distribution of both types of recyclates, obtained by sieving and laser diffraction techniques, revealed an average diameter of 390 µm or 950 µm, and a fineness modulus of 1.64 or 2.69 for FW or CW admixtures, respectively (Figs. 4 and 5). The fineness modulus was computed as the sum of the total percentages of GFRP wastes retained in the ASTM sieves n° 100 and upper (sieves n° 50, n° 30, n° 16, n° 8 and n° 4 of ASTM principal series) divided by 100. Sieving process was conducted as per EN 933-1:2012 standard and laser diffraction analyses of filler fractions (<74 µm) were performed on a Particle Size Analyser Laboratory Unit (Malvern Mastersizer 2000 G) using an aqueous solution as dispersion media.

It is worth pointing out that both grades of recyclates were proceeding from the same type of GFRP profiles, have the same proportion of glass fibre, calcium carbonate and organic resin and only differ with regard to average particle size and average fibre length. Siliceous foundry sand (SP55, Sibelco Lda), with rather uniform particle size, an average diameter of 245 µm and a fineness modulus of 3.04, was used as fine aggregate (Fig. 4). Foundry sand is a generic term to denote sand with a high-grade of silica (>99.0%) and detailed characterization of the specific foundry sand applied in this study can be found elsewhere (Ribeiro et al., 2003).

Commercially available unsaturated polyester resin (Aropol® FS3992), with a styrene content of 42%, was used as binder. The resin system is the same applied as matrix in the manufacturing process of GFRP pultrusion profiles produced by Alto. Its application in this study as binder matrix was justified in order to prevent possible incompatibility problems with GFRP waste admixtures. The polymerization process of resin system was induced by cobalt

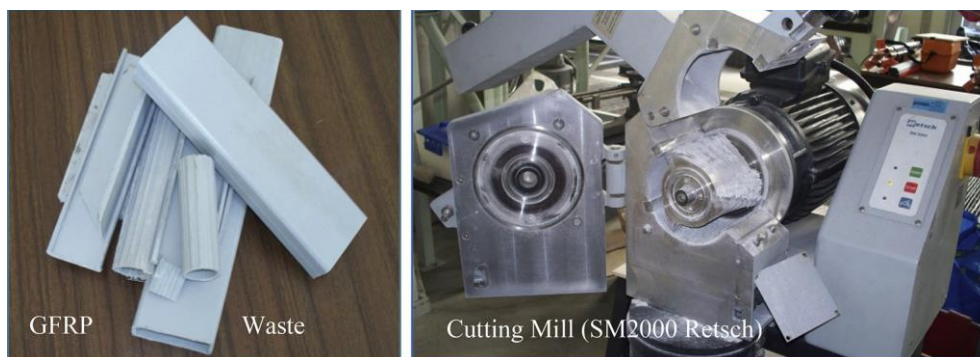


Fig. 2. Sample of GFRP wastes before being processed and Cutting Mill laboratory unit used in the grinding and milling process.



Fig. 3. Samples of CW and FW recyclates.

octoate (0.5 phr), as promoter, and 50% methyl ethyl ketone peroxide solution (2 phr), as initiator. Physical and mechanical properties of the resin binder, as supplied by the manufacturer, are presented in Table 1.

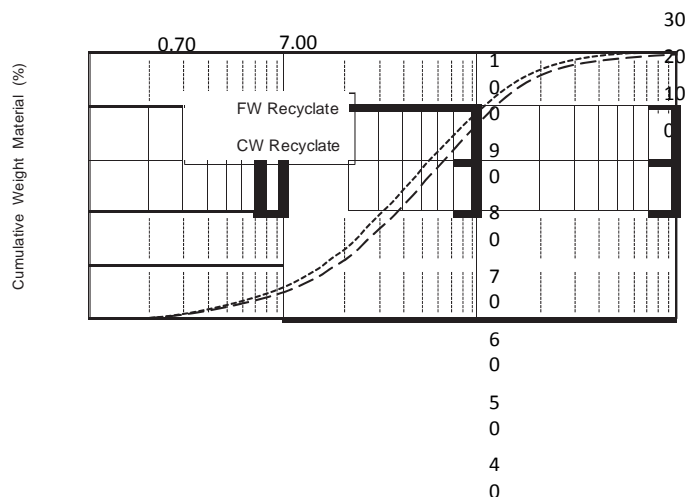
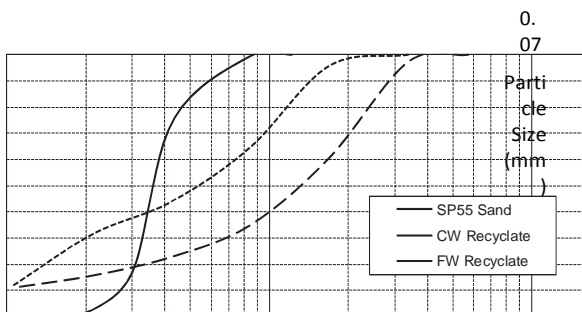
An organofunctional silane chemical solution (Dow Corning® Z-6032), with 40% of active silane in methanol, was applied as adhesion promoter of resin binder to the inorganic aggregates and GFRP recyclates. Z-6032 silane contains a vinylbenzyl and amine organic groups and a trimethoxysilyl inorganic group. As a coupling agent, it can be used either as an additive to a polymer or as a pre-treatment on inorganic surfaces. In this study, Z-6032 silane solution was applied as an additive to the polyester resin binder, in the proportion of 1% of active silane by weight of resin content.

2.2. Mix design and testing procedures

The mix design of the reference PM formulation was in accordance with previous studies carried out by Ribeiro et al. (2003), in which a polyester resin binder with similar viscosity was applied. With a basis on the reference mix design, 4 main test series of PM formulations were prepared by mixing the resin binder (modified or unmodified with silane coupling agent additive), with the sand aggregates/GFRP wastes mixtures (FW or CW grades). For each

main test series, 4 different weight percentages of sand aggregate replacement were considered (0%, 5%, 10% and 15%). Analysed trial formulations correspond to a three-factor full factorial design ($2^2 \cdot 4^1$), in which 'Silane Content', 'Waste Type' and 'Waste Content' were considered as material factors and each one was run, respectively, at 2 (0% and 1% in weight of resin mass), 2 (CW and FW grades) and 4 (0%, 5%, 10% and 15% in weight of aggregates mass) variation levels. The resin to total aggregate (sand plus recyclates) weight ratio was kept constant at 1:4 in all formulations; therefore, the GFRP recyclates played the role of sand aggregate replacement. Resultant mix design formulations were evaluated on the basis of six specimens (replicates) and the following notation was adopted: the letter 'S' (or its absence) denotes the modification (or not) of resin binder with silane coupling agent, 'CW' or 'FW' accounts for the type/grade of GFRP recyclates, and the sequent number for the weight percentage of sand aggregates replacement.

The $2^2 \cdot 4^1$ full factorial design leads to sixteen different formulations; however, both the pairs of formulations CW-0/FW-0 and SCW-0/SFW-0 are in fact of the same composition: 20% of resin (modified or not with silane coupling agent), 80% of foundry sand and 0% of CW (or FW) admixture. Hence, for data treatment purposes, these pairs of mix design formulations, with equal composition, share the same replicates.



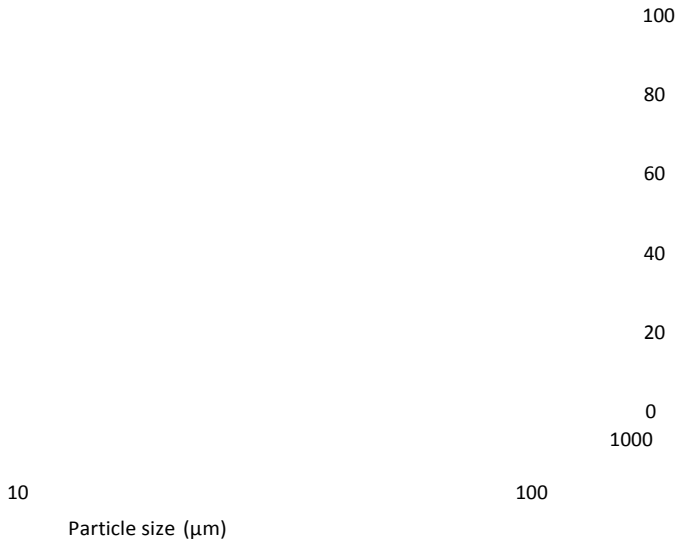


Fig. 4. Particle size distribution obtained by sieving process of sand aggregates and GFRP recyclates.

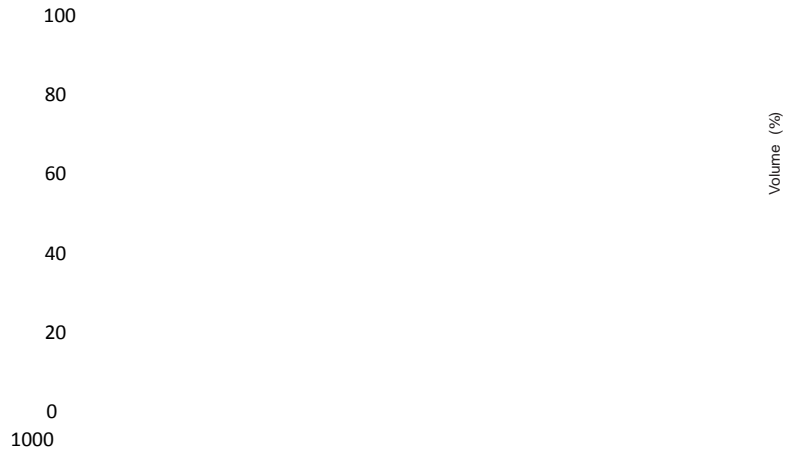


Fig. 5. Particle size distribution obtained by laser diffraction technique of filler fraction of GFRP recyclates.

Table 1
Physical and mechanical properties of cured resin (Aropol FS3992).

Resin properties	Method	Value
Heat defl. temp. (°C)	ASTM D-648	95
Barkoll hardness	ASTM D-2583	45
Tensile strength (MPa)	ASTM D-638	60
Flexural strength (MPa)	ASTM D-790	110
Elongation at break (%)	ASTM D-638	3.2

PM mixtures were prepared in an automatic mixer and casted into standard prismatic moulds (40 x 40 x 160 mm³) as per RILEM recommendation CPT PC-2:1995. After hardening process (24 h at 30°C/50% RH) the moulds were stripped off and all the test specimens were further cured for 3 h at 80 °C prior to being tested in bending and compression at the same age, after a minimum conditioning period of 24 h at room temperature (Fig. 6).

Prismatic PM specimens were tested in three-point bending up to failure at the loading rate of 1 mm min⁻¹ over a span length of 100 mm, as specified by RILEM CPT PCM-8:1995 test method. One of the two leftover parts of each broken specimen in bending was tested afterwards in compression at the loading rate of 1.25 mm.min⁻¹, in compliance with UNE 83821:1992 test standard. Applied test operating methods were similar to those specified in EN 206-1:2005, the test standard commonly used in the determination of the strength of cement mortars.

2.3. Fuzzy Boolean Nets methodology and complementary analyses

The FBN were used to analyse the trial data obtained for the main test formulations as function of the weight percentages of sand aggregate replacement by GFRP wastes. The goal was to detect what is the best formulation in what concerns compressive and flexural strengths, and what is the optimal percentage of sand replacement by GFRP waste within the tested range (0% up to 15%). The need to use FBN arose from the fact that the trial data only contained four different values of GFRP waste percentage, and there was a need to generalize the results for the whole interval.

Two 1-antecedent/1-consequent FBN were created. The input of both FBNs consists in the GFRP waste percentage. The output of FBN-1 is compressive strength and the output of FBN-2 is flexural strength. Both FBN used 128 neuron per area. Each neuron contained $n \approx 25$ inputs. Maximum granularity was used. These parameters were chosen empirically and are known to provide good approximation and generalization results for similar data sets (Carvalho and Tomé, 2007).

Training phase: Each FBN was trained using the compressive and flexural trial data obtained for each main formulation and GFRP

waste percentage. Each input/output pair was presented to the FBN for $r \approx 100$ times. After training phase completion, the FBN is able to estimate compressive and flexural strengths (and associated estimation error) of each formulation for any desired GFRP waste percentage. Overall training time for each FBN was negligible (less than 1 min) using a laptop with an Intel 1.8 Ghz i7 dual-core processor and 4 Gb of RAM.

Inference phase: Each FBN was tested for all formulations, and for GFRP waste percentages ranging from 0% to 15%. Due to the FBN probabilistic nature, each input value was run 100 times, and the results were averaged. Once again inference phase time is negligible (less than 2 min) when compared to the experimental test procedures described in Section 2.2.

In order to complement FBN analyses, data results were also submitted to non-parametric Kruskal-Wallis analyses of variance (ANOVA). Initially, parametric analyses of variance were considered. However, the analyses of residues previously performed according to Shapiro-Wilk's and Levene's tests showed that ANOVA's assumptions related to the normality and homoscedasticity were not met (Lix et al., 1996). Therefore, the nonparametric Kruskal-Wallis ANOVAs were used to test the null hypothesis (i.e., to verify if each factor independently considered has significant influence on flexural and compressive strength responses, to determine the main contributions of each factor to global variance, and to identify any eventual interaction effect across them). A data rank transformation was made considering the entire set of observations from smallest to largest, and the usual parametric procedure was then applied to the ranks of the data instead of to the data themselves as described in Conover and Iman (1981).

3. Results and discussion

3.1. Experimental and theoretical results

Compressive and flexural test results obtained according to the experimental methodologies described in Section 2.2 in terms of average mechanical strengths and correspondent standard deviations are summarized in Tables 2 and 3, respectively. The discrete results obtained for each specimen of each trial formulation are also presented in these tables.

Tables 4 and 5 show the FBN estimation of the compressive and flexural strength responses, and respective error intervals, for the 4 main test formulations as function of different GFRP waste contents within the tested range. In order to allow a better visualisation of FBN outputs, these data are also graphically displayed in Figs. 7 and 8.

The non-parametric Kruskal-Wallis ANOVA test results are presented in Tables 6 and 7 for compressive and flexural strength

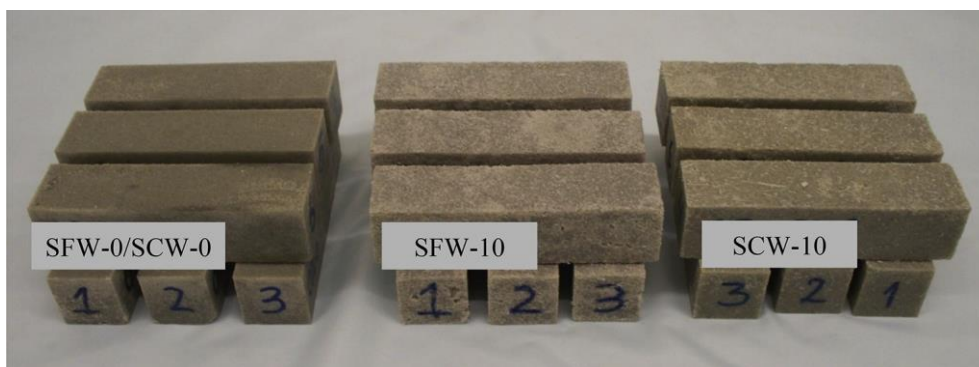


Fig. 6. Some examples of PM test specimens before being tested in bending (SCW-0/SFW-0, SFW-10 and SCW-10 trial formulations).

Table 2

Experimental compressive test results obtained for each trial PM formulation: discrete values, average values and correspondent standard deviations.

Compressive strength (MPa)	CW formulations GFRP waste (%)				FW formulations GFRP waste (%)			
	0%	5%	10%	15%	0%	5%	10%	15%
0% Silane	77.71	80.36	88.71	77.12	77.71	76.73	90.13	80.98
	70.30	83.11	84.37	82.83	70.30	78.40	83.61	75.01
	75.96	86.92	84.56	83.91	75.96	78.34	83.79	81.07
	79.89	80.52	82.68	85.68	79.89	77.75	82.68	82.83
	77.76	84.30	89.50	90.26	77.76	74.40	87.09	79.86
	76.14	85.17	87.25	78.83	76.14	82.66	86.23	79.99
Average	76.29	83.39	86.18	83.10	76.29	78.05	85.59	79.96
St. dev	3.26	2.60	2.71	4.75	3.26	2.71	2.79	2.65
1% Silane	82.07	97.16	104.45	83.23	82.07	85.43	81.89	58.39
	80.45	97.47	105.12	65.31	80.45	83.28	68.87	63.90
	80.10	96.54	101.63	56.28	80.10	86.18	85.73	76.90
	81.74	102.24	103.88	78.94	81.74	89.10	75.62	74.11
	80.92	98.91	101.47	84.54	80.92	89.10	70.98	74.42
	83.79	100.60	105.32	82.97	83.79	84.30	86.62	83.38
Average	81.51	98.82	103.64	75.21	81.51	86.23	78.29	71.85
St. dev.	1.34	2.22	1.70	11.68	1.34	2.43	7.58	9.11

responses, respectively. In both performed analyses, factors effects with a significance level of 5% or lower (p -value < 0.05) were considered statistically significant.

3.2. Discussion

From the FBN results presented in Table 4 and Fig. 7 regarding compressive strength response, it is clear that the formulation modified with silane coupling agent and with the coarser waste (SCW) gives the best overall results, with the optimum GFRP waste content varying between 8.25% and 11.25%. In order to use as most GFRP waste as possible in this formulation, it is possible to apply CW contents up to 12% as there is not significant loss on compressive strengths when compared to the optimal value of around 10% (estimated loss less than 2.4%, and within the estimated

Table 3
Fuzzy Boolean Nets outputs for the estimated compressive strength response of PM formulations as function of GFRP waste content.

GFRP waste

Table 4

Experimental flexural test results obtained for each trial PM formulation: discrete values, average values and correspondent standard deviations.

Flexural strength (MPa)	CW formulations GFRP waste (%)				FW formulations GFRP waste (%)			
	0%	5%	10%	15%	0%	5%	10%	15%
0% Silane	25.11	30.71	24.25	25.61	25.11	25.84	29.70	29.32
	24.91	27.52	25.41	27.79	24.91	26.97	26.36	26.39
	26.95	27.54	26.91	25.98	26.95	24.53	29.59	26.86
	23.37	26.72	28.65	26.80	23.37	24.89	27.89	27.84
	26.20	28.19	25.48	24.67	26.20	28.65	27.11	22.25
	24.47	27.70	27.36	26.34	24.47	26.53	25.91	25.30
Average	25.17	28.06	26.34	26.20	25.17	26.24	27.76	26.33
St. dev	1.27	1.38	1.59	1.06	1.27	1.50	1.61	2.42
1% Silane	34.79	40.87	43.10	38.96	34.79	36.73	34.95	25.57
	38.29	34.53	42.42	27.24	38.29	40.11	29.76	27.38
	37.64	39.28	39.05	26.69	37.64	36.61	37.40	29.59
	35.60	39.91	42.22	37.89	35.60	40.97	36.55	32.39
	35.83	44.14	37.49	41.29	35.83	41.63	29.05	31.18
	35.80	41.33	35.83	31.27	35.80	38.89	33.23	32.93
Average	36.32	40.01	40.02	33.89	36.32	39.16	33.49	29.84
St. dev	1.34	3.17	3.00	6.31	1.34	2.13	3.48	2.90

FBN error). A predicted added value correspondent to more than 20% increase in compressive strength is achieved with this trial formulation (SCW-10) with regard to analogous waste-free formulation (SCW-0). This predicted value is also validated by experimental test results (see Table 2).

Regarding SFW formulation, the higher compressive strengths were predicted for lower contents of GFRP waste, between 3% and 5.25%. A slight increase of less more than 3% in compressive strength is estimated for the optimum content of GFRP waste content (4.5%); as such, no significant higher value is achieved within this formulation through aggregates replacement by FWrecyclates.

Without binder modification with silane coupling agent, the formulation with coarse waste (CW) also provides the best compressive test results when compared to the formulation with fine waste (FW). For both formulations, the best results were found for GFRP waste contents in the range of 9e12%, with the optimal points at 11.25% and 9.75% for, respectively, CW and FW based formulations. Predicted compressive strength increases of around 10% and 12% were found for these formulations when compared to

FBN output: Average compressive strength and estimated error (MPa)

	CW	FW	SCW	SFW
0.0%	79.29 ± 0.00	74.76 ± 0.00	86.08 ± 0.00	84.57 ± 0.00
0.75%	81.73 ± 1.75	78.15 ± 1.65	95.58 ± 2.50	83.41 ± 1.45
1.50%	82.80 ± 1.79	78.80 ± 1.60	98.56 ± 1.47	84.07 ± 1.81
2.25%	82.74 ± 1.68	78.82 ± 1.77	99.48 ± 1.13	85.03 ± 2.01
3.00%	83.09 ± 1.87	79.29 ± 1.73	99.49 ± 1.08	86.54 ± 1.75
3.75%	84.58 ± 1.27	81.11 ± 1.19	99.49 ± 1.08	87.14 ± 1.96
4.50%	84.58 ± 1.27	81.11 ± 1.19	99.49 ± 1.08	87.14 ± 1.96
5.00%	85.24 ± 1.47	80.01 ± 1.95	100.13 ± 1.13	86.70 ± 1.76
5.25%	85.32 ± 2.05	80.15 ± 1.89	100.09 ± 1.22	86.78 ± 1.81
5.50%	85.58 ± 2.25	81.05 ± 2.19	100.94 ± 1.34	85.72 ± 2.01
5.75%	85.91 ± 2.95	81.96 ± 2.06	101.95 ± 1.09	84.14 ± 2.27
6.00%	85.88 ± 1.69	83.44 ± 1.68	102.67 ± 0.94	82.40 ± 2.20
6.25%	86.41 ± 1.79	83.90 ± 1.98	103.41 ± 0.77	81.89 ± 2.09
6.50%	86.30 ± 2.19	84.04 ± 2.11	103.63 ± 0.69	81.57 ± 1.90
6.75%	86.30 ± 2.19	84.04 ± 2.11	103.63 ± 0.69	81.78 ± 1.79
7.00%	86.30 ± 2.19	84.04 ± 2.11	103.63 ± 0.69	81.99 ± 1.88
7.25%	86.30 ± 2.19	84.04 ± 2.11	103.63 ± 0.69	82.05 ± 1.83
7.50%	86.90 ± 1.68	83.90 ± 1.92	102.79 ± 1.18	82.68 ± 1.96
7.75%	86.45 ± 2.12	83.59 ± 1.81	101.20 ± 2.26	82.79 ± 1.99
8.00%	84.44 ± 2.25	83.40 ± 1.91	96.86 ± 3.62	83.21 ± 1.68
8.25%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	84.06 ± 1.52
8.50%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
8.75%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
9.00%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
9.25%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
9.50%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
9.75%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
10.00%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
10.25%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
10.50%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
10.75%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
11.00%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
11.25%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
11.50%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
11.75%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
12.00%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
12.25%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
12.50%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
12.75%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
13.00%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
13.25%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
13.50%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
13.75%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
14.00%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
14.25%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
14.50%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
14.75%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
15.00%	82.25 ± 2.38	82.25 ± 2.38	96.86 ± 3.62	85.08 ± 0.94
0.00	78.54 ± 0.00	80.42 ± 0.00	80.80 ± 0.00	85.33 ± 0.00

interaction effects results obtained by Kruskal-Wallis test as this analysis may be unable to properly identify interaction effects when multiple factors are involved (Wobbrock et al., 2011).

Regarding flexural strength response, FBN outputs presented in Table 5 and Fig. 8 also show that the SCW formulation exhibits the best flexural performance among the 4 main test formulations in analysis, but with the optimum GFRP waste contents varying between lower values than those found for compressive strength response, between 3% and 6%. The optimum value of CW content is estimated to be around to 3.75%, which leads to a predicted improvement on flexural strength of 14% with respect to plain

formulation (SCW-0). For the homologous formulation with fine

waste incorporation (SFW), the best results are also achieved for

Table 5

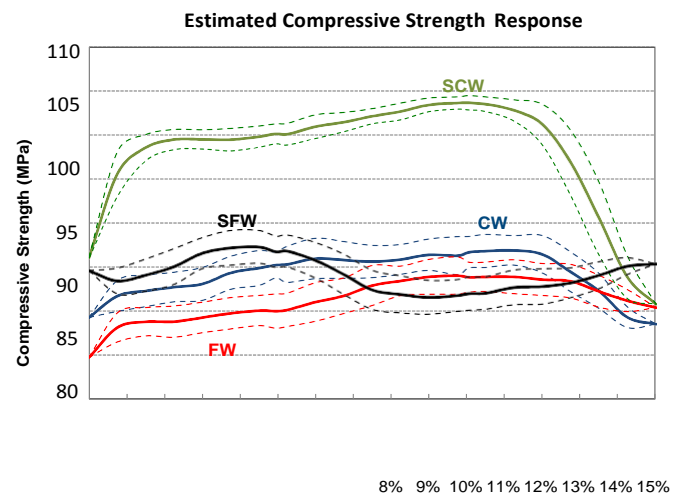
Fuzzy Boolean Nets outputs for the estimated flexural strength response of PM formulations as function of GFRP waste content.

GFRP waste	FBN output: Average flexural strength and estimated error (MPa)			
	CW	FW	SCW	SFW
0.0%	24.44 ± 0.00	24.95 ± 0.00	36.23 ± 0.00	36.56 ± 0.00
0.75%	26.85 ± 0.77	25.55 ± 0.55	38.93 ± 0.85	38.37 ± 0.75
1.50%	27.28 ± 0.62	25.81 ± 0.68	39.89 ± 0.74	38.88 ± 0.71
2.25%	27.20 ± 0.66	26.09 ± 0.59	40.42 ± 0.77	39.21 ± 0.64
3.00%	27.03 ± 0.70	26.05 ± 0.60	40.92 ± 0.67	39.22 ± 0.71
3.75%	27.28 ± 0.77	25.95 ± 0.57	41.34 ± 0.58	39.30 ± 0.70
4.50%	27.36 ± 0.75	25.94 ± 0.61	41.32 ± 0.62	39.25 ± 0.67
5.00%	27.35 ± 0.73	26.07 ± 0.65	41.24 ± 0.61	39.16 ± 0.77
5.25%	27.48 ± 0.71	26.15 ± 0.64	41.01 ± 0.74	38.92 ± 0.79
6.00%	27.60 ± 0.74	26.42 ± 0.65	40.67 ± 0.68	38.44 ± 0.86
6.75%	27.72 ± 0.62	26.82 ± 0.88	40.36 ± 0.68	37.72 ± 1.01
7.50%	27.68 ± 0.70	27.22 ± 0.79	39.88 ± 0.68	36.90 ± 1.13
8.25%	27.72 ± 0.64	27.50 ± 0.75	39.69 ± 0.68	35.82 ± 0.96
9.00%	27.64 ± 0.70	27.75 ± 0.71	39.44 ± 0.68	35.19 ± 1.07
9.75%	27.51 ± 0.76	27.60 ± 0.72	39.34 ± 0.67	34.34 ± 1.05
10.00%	27.25 ± 0.70	27.48 ± 0.79	39.21 ± 0.71	33.95 ± 0.92
10.50%	27.08 ± 0.79	27.50 ± 0.75	39.07 ± 0.74	33.63 ± 0.89
11.25%	26.71 ± 0.68	27.03 ± 0.74	38.89 ± 0.79	33.17 ± 0.82
12.00%	26.22 ± 0.74	26.82 ± 0.74	38.31 ± 0.97	33.25 ± 0.74
12.75%	25.91 ± 0.68	26.62 ± 0.71	37.09 ± 1.18	33.21 ± 0.84
13.50%	25.80 ± 0.49	26.22 ± 0.52	35.14 ± 1.43	33.93 ± 0.76
14.25%	26.00 ± 0.31	25.89 ± 0.31	32.72 ± 0.93	34.69 ± 0.46
15.00%	26.12 ± 0.00	25.79 ± 0.00	31.68 ± 0.00	35.05 ± 0.00

15.00% 26.12 ± 0.00 25.79 ± 0.00 31.68 ± 0.00
 35.05 ± 0.00

the same GFRP waste content (3.75%), but the estimated increases on flexural strength are quite lower (around 7.5%). In both formulations modified with silane, sand replacement weight contents up to 6% are feasible without significant losses on flexural strengths when compared to the response values achieved for the estimated optimum contents.

As stressed by Fig. 8, no significant differences were found on flexural strength behaviour between both the formulations without binder modification (CW and FW). The highest estimated values of flexural strength attained in CW and FW based formulations are almost equal (27.72 MPa and 27.75 MPa, respectively), the relative increases with respect to plain formulations are very similar (13% and 11%, respectively), and the estimated variations as function of GFRP waste content are quite close within the tested range. Though, whereas the optimum CW content is estimated to be close to 7.5%, that of the FW content is a little higher, around 9%. Nevertheless,



Estimated Flexural Strength Response

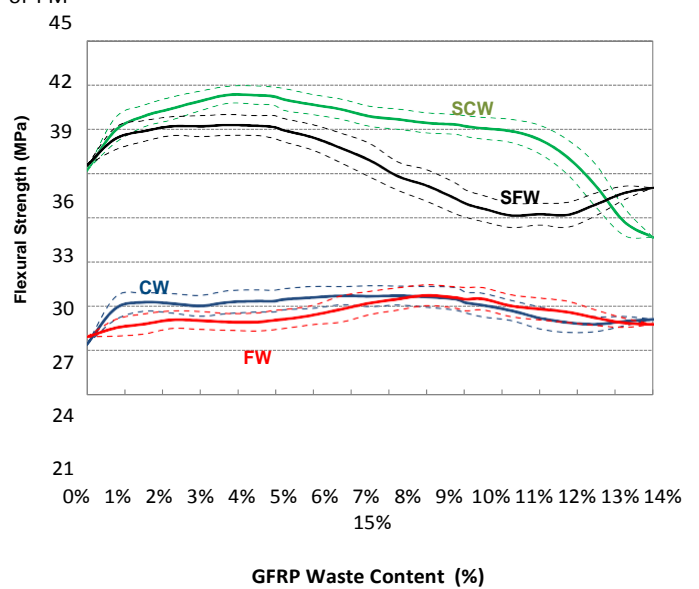


Fig. 8. FBN estimation of the flexural strength responses of the main PM formulations as function of GFRP waste content and respective error intervals (dashed lines).

this result may well indicate that the type, or size grade, of GFRP recyclates applied in this study has no significant influence on flexural strength behaviour of PM formulations without silane coupling agent addition.

This feature is confirmed by ANOVA test results regarding flexural strength response. According to these results presented in Table 7, for a significance level of 5%, the null hypothesis is not rejected for the material factor 'Waste type' (p -value > 0.05) denoting the weak influence of this variable. On the other hand, 'Silane content' has a strong influence on flexural strength response contributing with more than 63% to global variation.

The beneficial effect of silane coupling agent addition on both compressive and flexural strengths of modified mortars, regardless of the GFRP waste content and type, is mainly due to the adhesion improvement effect at binder-overall aggregates interface. This fact was confirmed by Scanning Electron Microscopy (SEM) analyses performed on samples of the fracture surface of PM specimens. As shown in Fig. 9, illustrating some examples, the test series modified with silane coupling agent present in general good adhesion at binder-sand aggregate or at binder-GFRP waste interfaces. On the other hand, signs of slipping or pull-out of GFRP waste fibres and zones denoting weak adhesion between the matrix binder and overall aggregates (sand plus GFRP wastes) were found on the samples proceeding from silane-free PM specimens.

As synopsis of experimental and FBN test results it can be stated that the formulation modified with silane coupling agent and with sand replacement by coarse waste (SCW) gives the best overall results for both compressive and flexural strengths, even if for

	0%	1%	2%	3%	4%	5%	6%	7%
Waste content	22463.9	3	7488.0	28.2	<0.00005	29.4		
Waste type	7686.3	1	7686.3	29.0	<0.00005	10.1		
Silane content	2542.0	1	2542.0	9.6	0.0027	3.1		
'Waste cont. * waste type'	2994.1	3	998.0	3.8	0.0140	3.0		
'Waste cont. * silane cont.'	12172.4	3	4057.5	15.3	<0.00005	15.4		
'Waste type * silane cont.'	605.0	1	605.0	2.3	0.13500	NS		
3-Factors interaction	4016.9	3	1339.0	5.1	0.0030	4.4		

Fig. 7. FBN estimation of the compressive strength responses of the main PM formulations as function of GFRP waste content and correspondent error intervals (dashed lines).

Table 6
Kruskal-Wallis ANOVA test results for compressive strength response.

Source	Sum Sq.	df	Mean Sq.	F	p-value	P (%)
Error	21231.4	80	265.4			
Total	73712.0	95				

NS e Not statistically significant for a confidence level of 95%.

P(%) e Percent contribution to global variation (computed as the ratio of the pure sum of squares of the factor or interaction to the total sum of squares).

Table 7
Kruskal-Wallis ANOVA test results for flexural strength response.

Source	Sum Sq.	df	Mean Sq.	F	p-value	P (%)
Waste content	5325.6	3	1775.2	9.6	<0.00005	6.5
Silane content	47126.1	1	47126.3	253.1	<0.0000	63.7
Waste cont. * waste	294.3	3	98.3	0.5	0.6635	NS
Waste cont. * silane	2555.3	3	851.7	4.6	0.0051	2.7
Waste type * silane	570.1	1	570.4	3.1	0.0835	NS
3-Factors interaction	2338.3	3	779.5	4.2	0.0082	2.4
Error	14855.80	80	185.7			
Total	73712	95				

NS e Not statistically significant for a confidence level of 95%.
P(%) e Percent contribution to global variation (computed as the ratio of the pure sum of squares of the factor or interaction to the total sum of squares).

different optimal ranges of GFRP waste percentage. A compromise value of 6% may be proposed for the CW weight content. This formulation ensures a very good performance in both criteria,

providing an increase of over 17% and 12% in, respectively, compressive and flexural strength responses, when compared to the waste-free reference formulation (CWS-0), while losing little more than 2% over the best estimated value in each criteria.

4. Conclusions

An integrated cost-effective recycling approach for GFRP waste proceeding from pultrusion manufacturing industry, involving mechanical recycling of industrial rejects and their incorporation as aggregate and filler replacement into concrete-polymer

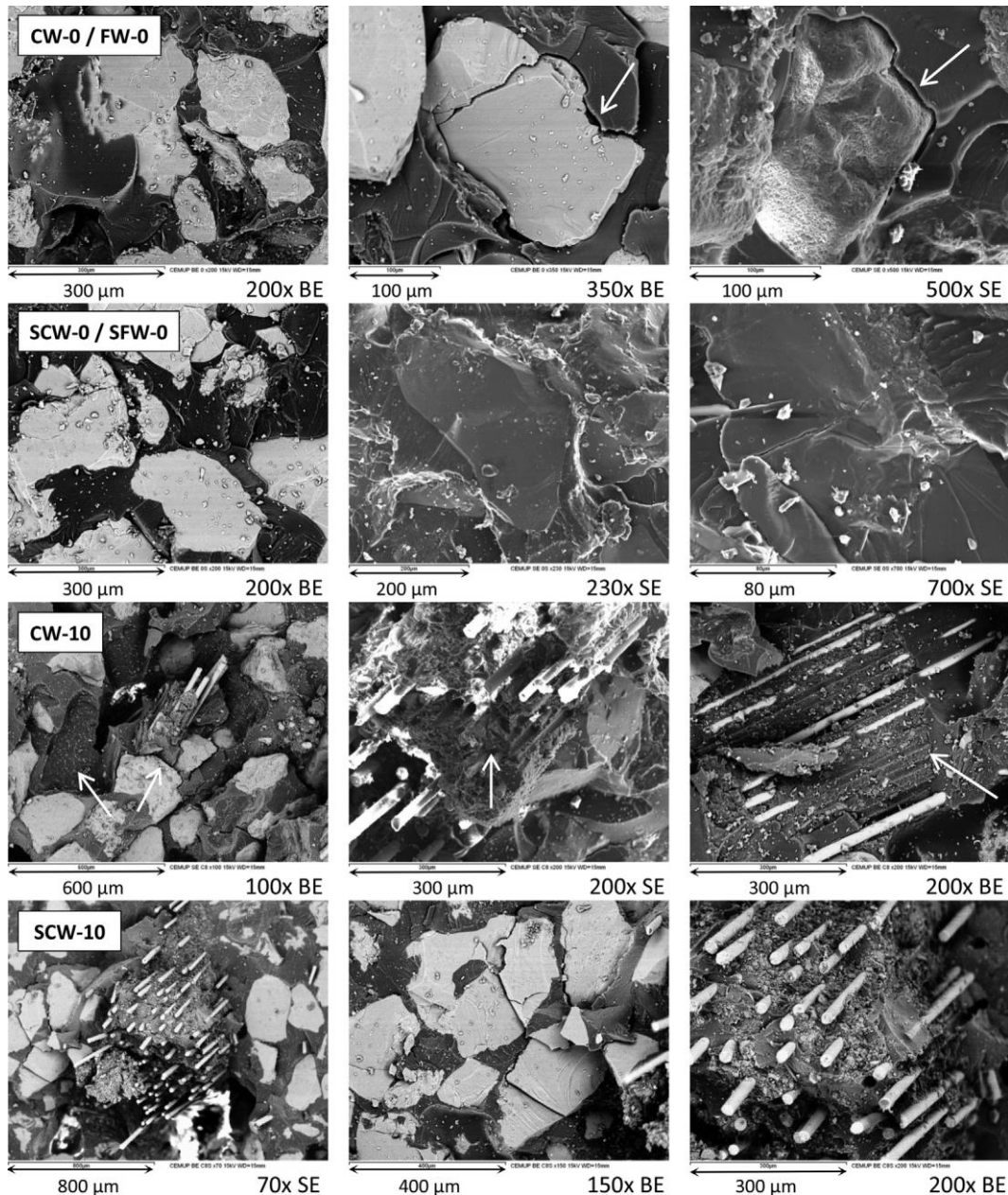


Fig. 9. SEM images of bending fracture surface of PM specimens of FW-0/CW-0, SFW-0/SCW-0, CW-10 and SCW-10 trial formulations; White arrows: signs of weak bond at matrix- aggregate interface or slippage of waste fibres (Accelerating voltage ¼ 15 kV; Wave Length ¼ 15 mm; Variable magnifications).

composites, was analysed and validated. The outputs of the present study can be summarized as follows:

- Under the present framework on recycling technologies for thermoset based composite materials, mechanical recycling can be considered as the most viable recycling method for GFRP pultrusion wastes, at least until new technological developments on current thermal and/or chemical recycling techniques lead to more cost-effective and less expensive recycling processes;
- Mechanically recycled GFRP pultrusion wastes can be cost-effectively used as aggregate and filler replacement into PM materials without special upstream and downstream sorting operations; though, cautions should be taken in order to provide proper sieves inside the grinding chambers, with suitable size meshes according to the intended end-use for the recyclates;
- Within the variation levels of material factors applied in the experimental part of this study, the partial replacement of sand aggregates by GFRP recyclates up to 12% in weight content has, in general, an overall incremental effect on both flexural and compressive strengths of resultant polymer mortars, regardless of the GFRP waste content, size grade and silane coupling agent addition;
- FBN methodology allows determining the best mix design formulation and sand replacement ratio that optimize the final mechanical strengths of GFRP waste admixed PMs. The mix design formulation modified with silane coupling agent and with partial sand replacement by coarse waste (SCW) gives the best overall results for both compressive and flexural strengths. A compromise value of 6% may be proposed for the CW weight content, which ensures a very good performance in both criteria and provides an increase of over 17% and 12% in, respectively, compressive and flexural strength responses when compared to the waste-free reference formulation.

The integrated recycling approach here proposed for GFRP industrial rejects and GFRP end-of-life products can be generalised and applied, with the required adjustments and studies, to other GFRP wastes derived from other manufacturing processes of composite materials than pultrusion. This will lead to a global waste management solution for GFRP based products and to a more sustainable composite materials industry.

Acknowledgements

This study was supported by national funds through 'ADI e Agência de Inovação', under 'QREN- Quadro de Referência de Estratégia Nacional, ON.2 Program', and 'FCT e Fundação para a Ciência e a Tecnologia', under project PEst-OE/EEI/LA0021/201. The technical support of 'Alto-Perfis Pultrudidos, Lda.', is also gratefully acknowledged.

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