A CASE STUDY UNDERTAKEN RECYCLING & REUSE OF GLASS FIBER REINFORCED THERMOSET POLYMER WASTES OF COMPOSITE MATERIALS INDUSTRY

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Abstract Glass fibre-reinforced plastics (GFRP) have been considered inherently difficult to recycle due to both: cross-linked nature of thermoset resins, which cannot be remoulded, and complex composition of the composite itself. Presently, most of the GFRP waste is landfilled leading to negative environmental impacts and supplementary added costs. With an increasing awareness of environmental matters and the subsequent desire to save resources, recycling would convert an expensive waste disposal into a profitable reusable material. In this study, efforts were made in order to recycle grinded GFRP waste, proceeding from pultrusion production scrap, into new and sustainable composite materials. For this purpose, GFRP waste recyclates, were incorporated into polyester based mortars as fine aggregate and filler replacements at different load contents and particle size distributions. Potential recycling solution was assessed by mechanical behaviour of resultant GFRP waste modified polymer mortars. Results revealed that GFRP waste filled polymer mortars present improved flexural and compressive behaviour over unmodified polyester based mortars, thus indicating the feasibility of the GFRP industrial waste reuse into concrete-polymer composite materials.

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1. INTRODUCTION AND RESEARCH SCOPE

Fibre reinforced polymer (FRP) is a composite material that can be produced with glass (GFRP), carbon (CFRP) or aramid (AFRP) fibres dispersed in an organic matrix, usually a thermoset polyester resin. FRP based products are appropriate for the design of structural elements demanding both high strength to weight ratio and high corrosion resistance. These composite materials are thus widely used in several fields, from building to furniture and from aerospace to military applications [1].

Among the several available techniques to produce and manufacture FRP composite materials, pultrusion process is the oldest continuous processing technique and, until now, is still the most cost-effective one [2]. In this process, typically, dry reinforcement fibres in the form of continuous strands (roving) or plys (mats, fabrics and veils), are pulled through a resin bath for impregnation and, after wetting process, the reinforcement pack is collimated into a performed shape before entering the heated die where it attains the final form and cures. Finally, outside the die, the composite part already polymerised is pulled by a continuous pulling system and then a cut-off saw cuts the part into a desired length. The final product of the pultrusion process can be a structural element or a simple flat sheet, a channel or an angle shaped element. FRP pultrusion profiles can be easily produced in any shape, along with the possibility of simple assembling operations and low maintenance costs [3].

Despite all the advantages associated to FRP composite materials, irrespective of their processing technique, they are responsible for a number of environmental and economic concerns related to waste disposal and recycling, disclosing a particular challenge in recycling processes since these thermoset based products can hardly be separated into their constituents [4-6]. In particular, the fibres are difficult to remove from the matrix, requiring complex thermo-chemical processes not economic viable, at least, for glass based FRP products [7]. In this context, GFRP wastes and end-of-life GFRP products are usually incinerated or landfilled, leading to additional costs and negative environmental impacts.

The perceived lack of recyclability of composite materials is now increasingly important and seen as a key barrier to the development or even continued used of these materials in some markets [4]. This increase awareness of environmental matters and seeking sustainable materials, stressed by the more restrictive directives of European Commission, have driven that several recycling techniques have been analyzed and proposed, mainly for GFRP and CFRP waste materials [4, 6]. Thermal and/or chemical recycling, with fiber recovering, have been proposed mostly for CFRP due to inherent economic value of carbon fiber reinforcement; whereas for GFRP materials, mechanical recycling by shredding and milling processes, with reduction to fibrous and/or powdered products, has been considered as a more viable recycling method. This approach, after material size reduction, generally requires specific sorting techniques into powdered and fibrous compounds as function of intended end-use application. Obtained recyclates could be applied either as filler extension or reinforcement into new composite materials [8-10]. Although mechanically recycled GFRP wastes have been considered as green and friendly materials, they remain however, somehow, as *'a little beautiful girl, well dressed, but no place to go',* hindered by the scarceness of feasible and cost-effective end-use applications.

The pressure on the development of new and economically viable markets for GFRP recyclates have led over the last 20 years a relative great amount of research work on potential added value applications. Several promising end-use applications have been investigated. The most extensive research work has been carried out on Portland cement concrete, in which grinded GFRP wastes have been incorporated either as reinforcement, aggregate or filler replacement [11-13]. Some added-values such as less shrinkage and improved durability were reported; however, slight to strong decreases on mechanical strength were also observed. Further, depending upon glass fibre nature, some incompatibility problems arisen from alkalis-silica reaction were also found [14]. This limitation, brought by cementitious nature of binder matrix, can be overpassed using as host material a cementless concrete, such as polymer concrete (PC).

PC materials are high performance resin based concretes, in which a polymer acts as binder matrix for the mineral aggregates. The main advantages of these materials rely on high mechanical strength and extreme resistance to chemical and frost attack [15-18], very fast curing process and great ability for incorporating recycled waste products, mainly due to hermetic nature of resin matrix [19-22]. Despite the considerable different approaches for testing recycled wastes admixed polymer based concretes, there is still a lack of research considering the incorporation of FRP recyclates into PCs [23]. Seeking filling this gap, the aim of the present work is to explore a potential waste management solution for GFRP waste (scrap, waste production and end-of-life products) as reinforcement, aggregate or filler replacement for polymer based concretes. For this purpose, different contents of mechanically recycled GFRP wastes, with distinct size grading, were incorporated into polyester based concretes as sand aggregates and filler replacements. Added value of recycling solution was assessed by means of flexural and compressive loading capacity of GFRP admixed PCs with regard to unmodified plain PCs. Applied waste material was proceeding from the shredding of the leftovers and non-conform products of GFRP pultrusion industry. Currently, these leftovers, jointly with unfinished products and scrap resulting from pultrusion manufacturing process (Fig.1a), are landfilled, with supplementary additional costs for the producers. Thus, besides the evident environmental benefits, a viable and feasible solution for these wastes would also conduct to significant economic advantages.

2. Experimental Program

2.1 Raw Materials and Trial Formulations

GFRP wastes applied in these experiments resulted from shredding leftovers derived of cutting and assembly processes of pultruded profiles at building sites. This scrap material was supplied by a Portuguese pultrusion manufacturing company (Alto, Perfis de Pultrusão, Lda.) that currently supports an annual cost of more than 4M € on landfill taxes of these leftovers and other scrap material resulting from manufacturing process.

GFRP wastes were mechanically recycled by milling on a heavy-duty cutting mill laboratory unit (SM2000, Retsch). Two different bottom sieves, with 2.5 mm square and 1.5 mm trapezoidal meshes, were used inside the grinding chamber in order to obtain two different grading sizes of milled GFRP waste. The final recycled products, illustrated in Fig. 1b) and 1c), consist of a mixture of particulate and fibrous material with different amounts of varying length of glass fibres, hereinafter designated by ground (Gpw) and fine (Fpw) pultrusion waste. Particle size distributions of both types of recycled waste revealed fineness modulus of 2.69 and 1.64 for Gpw and Fpw particulate systems, respectively. Burning tests carried out on five random samples of GFRP waste showed an average inorganic material content of 71% (w/w), corresponding to E- glass fibre and calcium carbonate fractions (20% of resin weight), and an average organic content of 29% (w/w), corresponding to polymer matrix**.**

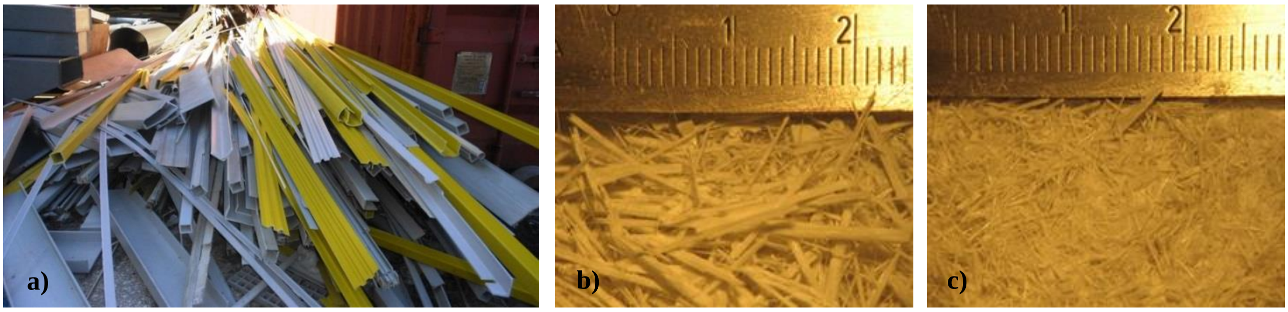

Fig. 1 Wastes generated by GFRP pultrusion industry (a) and obtained recyclates: Gpw (b) and Fpw (c)

An unsaturated polyester resin (Aropol FS3992, Ashland®), with a styrene content of 42%, was used as binder at 20% weight content of total mass. Polymerisation process was induced by cobalt octoate (0.5 phr), as promoter, and 50% methyl ethyl ketone peroxide solution (2.0 phr), as initiator. This resin system is the same applied in the GFRP manufacturing process and it was selected in order to prevent possible incompatibility problems with GFRP waste admixtures.

Siliceous foundry sand with rather uniform particle size, an average diameter (d_{50}) of 245 μm and a fineness modulus of 3.04, was used as aggregate.

GFRP waste admixed PC formulations were designed according to the Design of Experiments, considering a two-factor mix-level full factorial design. The type of GFRP recycled waste and its content (weight content of total mass) were chosen as factors, and each one was run at two (Gpw and Fpw) and four levels (0%, 4%, 8% and 12%), respectively. Resin to total aggregate (sand plus GFRP waste admixtures) weight ratio was kept constant to 1:4 in all formulations, thus, the GFRP waste recyclates played the role of sand aggregate replacement. Resultant mix design formulations, presented in Table 1, were evaluated with basis on four replicates for each different combination of factors and levels.

Table 1 Mix design of trial formulations: weight proportions of raw materials (w/w)

Trials	Fpw-0	Fpw-4	Fpw-8	Fpw-12	Gpw-0	Gpw-4	Gpw-8	Gpw-12
Resin (%)	20	20	20	20	20	20	20	20
Sand (%)	80	76	72	68	80	76	72	68
Fpw (%)	0	4	8	12	-	-	-	-
Gpw (%)	-	-	-	-	0	4	8	12

2.2 Manufacturing and Testing Methods

PC mixtures, with binder formulations and mix proportions specified in Table 1, were prepared in an automatic mixer and casted into standard prismatic moulds (40 x 40 x 160 mm^3) as per RILEM recommendation CPT PC-2:1995 [24]. For each trial formulation, 4 test specimens were casted. After hardening process (24h at 30ºC/50% RH), the moulds were stripped off and all test specimens were further cured for 3h at 80ºC.

Test specimens were firstly tested in three-point bending up to failure, over a span of 100 mm, at the loading rate of 1 mm.min-1 in accordance with RILEM CPT PCM-8:1995 recommendation [25]. 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, as per UNE 83821:1992 standard test [26].

3. Experimental Results and Discussion

3.1 Test Results and Statistical Analyses

Table 2 summarizes flexural and compressive test results obtained for all trial formulations, and Fig. 2 helps in the data description by resuming both the central tendency and dispersion measures.

Basic descriptors are supported by the results of factorial analyses of variance (two-way ANOVA), using the p-value approach to hypothesis testing (i.e., p-value ≤ significance level). Two-way ANOVA were performed to determine the main effect of contributions of each factor on target responses (flexural and compressive strengths), and to identify eventual interaction effects across them. For all performed analyses, factors effects with a significance level of 5% or lower (p-value ≤ 0.05) were considered statistically significant. The $2^1\ 4^1$ full factorial design leads to eight different formulations; however, both formulations Gpw-0 and Fpw-0 present, in fact, the same composition: 20% of resin, 80% of foundry sand and 0% of Gpw (or Fpw) admixture. Hence, for data treatment purpose, these mix design formulations, with equal composition, share the same replicates. ANOVA results for both analysed properties are presented in Table 3.

Table 2 Flexural and compressive test results: average mechanical strength of four replicates

Trials	Fpw admixed PM				Gpw admixed PM			
Formulations	Fpw-0	Fpw-4	Fpw-8	Fpw-12	Gpw-0	Gpw-4	Gpw-8	Gpw-12
Flexural Str. (MPa)	25.2	26.2	26.8	27.1	25.2	27.5	26.2	26.2
Compressive Str (MPa)	76.3	78.0	85.6	81.0	76.3	83.4	85.7	82.0

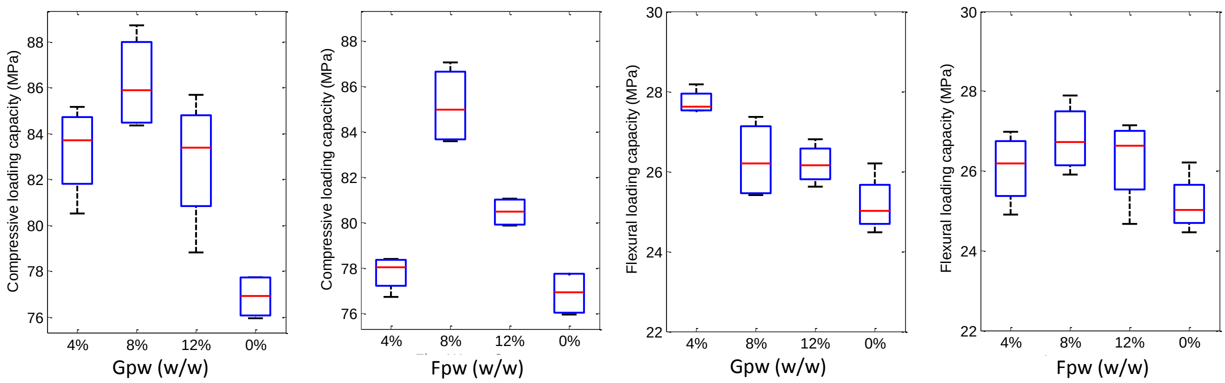

Fig. 2 Boxplots of compressive and flexural loading capacities of Gpw and Fpw trial formulations as function of GFRP waste content

Table 3 ANOVA test results: sum of squares (SS), degrees of freedom (df), mean of squares deviation (MSD), *p*-value and percent contributions to global variation (P)

	Compressive Strength Response					Flexural Strength Response				
Source	SS	df	MSD	*p*-value	P	SS	df	MSD	*p*-value	P
GFRP Waste Content	315.4	3	105.2	$\leq 5\text{x}10^{-5}$	67%	13.32	3	4.44	0.0018	31 %
GFRP Waste Type	39.1	1	39.1	0.0011	8%	0.57	1	0.57	0.3588	-
Interaction	33.8	3	11.3	0.0206	7%	5.62	3	1.87	0.0577	-
Error	68.8	24	2.9			15.70	24	0.64		
Total	457.2	31				35.21	31			

From the results provided by two-way ANOVA for compressive strength analysis, it is clear that both factors, 'GFRP Waste Content' (p-value ≤ 0.00005) and 'GFRP Waste Type' (p-value = 0.0011), as well as the two-factor interaction (p-value = 0.0206), have a significant influence on compressive strength response. The respective percent contributions to global variation (P), computed as the ratio of the pure sum of squares of the factor (or interaction) to the total sum of squares are, respectively, 67%, 8% and 7% (pure sum of squares, here understood as the sum of squares minus the degree of freedom times the error variance).

Regarding flexural strength analysis, although it is clear that 'GFRP Waste Content' has a significant effect on flexural strength (p-value = 0.0018), for the stipulated significance level of 5%, no evidences were found that 'GFRP Waste Type' lead to distinct flexural behaviours of modified mortars (p-value = 0.3588). The interaction between the two factors was also found not to have any significant influence on flexural strength (p-value = 0.0577).

3.2 Analysis of Results

In order to support the discussion of results and emphasize the effects of material factors on the mechanical strengths of PMs, the main effects of each factor are plotted and highlighted in response graphics displayed in Fig. 3 for compressive and flexural strength responses.

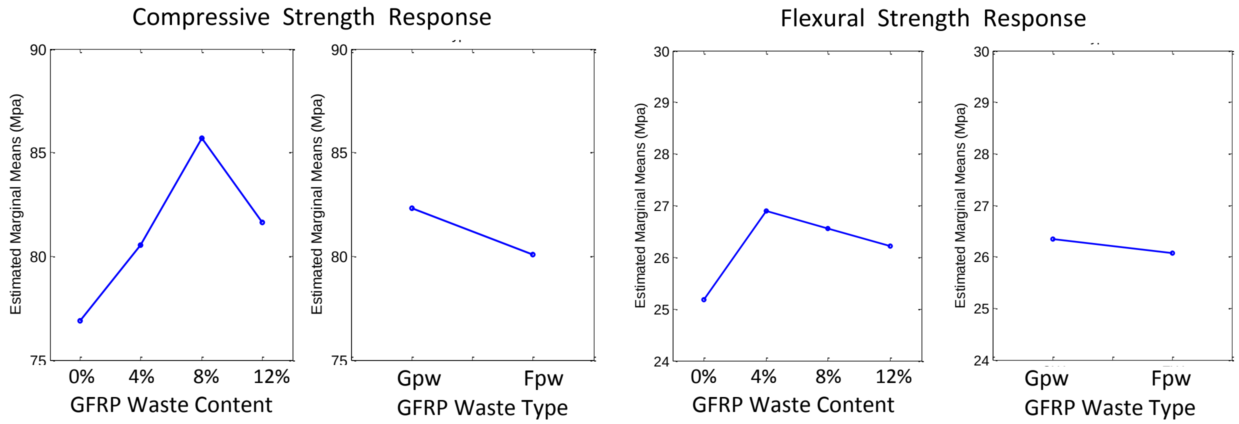

Fig. 3 Compressive and flexural strength responses: Main effects' plots of 'GFRP Waste Content' and 'GFRP Waste Type' factors

3.2.1 Effect of 'GFRP Waste Content'

As stressed by response graphs plotted in Fig. 3, the incorporation of GFRP waste into polyester PMs has an incremental effect on both compressive and flexural strengths regardless of the GFRP waste type. However, distinct trends were observed for the effect of waste admixture on mechanical performance according to the amount of waste addition and the mechanical response itself (in compression or in flexural).

Up to 8% content in sand replacement by GFRP waste, compressive strengths of PMs increase with increasing addition of GFRP recyclates. Average compressive strength increases of 4.7% and 11.5% corresponding to the addition of 4% and 8% in weight of GFRP waste, respectively, were observed with regard to unmodified PMs. The almost linear increase of compressive strength with GFRP waste content might be attributed to a more continuous particle size distribution of the mix sand/waste particles. The contribution of GFRP waste powder to filler fraction of sand aggregates, leading to an inferior void volume for dry-packed aggregate, has a relevant role in this feature. In flexural, this trend, the linear increase of loading capacity with increasing addition of GFRP waste, is not verified. Average increases on bending capacity of 6.8% and 5.5% were found for 4% and 8% in weight of GFRP waste additions, respectively. It was expected that fibrous fraction of GFRP recyclates would have a significant reinforcing effect, leading to a higher improvement on flexural behaviour. Although this expected flexural improvement did actually occur for Fpw test series, in which progressive increases of 3.5% and 6.6% on bending strength were noticed for Fpw-4 and

Fpw-8 trial formulations, respectively; slight decrease on flexural strength was observed for Gpw test series, when Gpw waste content was increased from 4% to 8%, and this tendency became even more marked for further addition amounts of coarse waste (Gpw-12). A possible explanation for observed behaviour might be advanced: in the mixing and casting process of Gpw modified mortar specimens, some tendency for the agglomeration of waste fibres was observed, hindering somehow a perfect homogenization of the mixture. This feature, more notorious as higher the Gpw content, led to a non-homogeneous distribution of GFRP waste, and might be the likely reason for observed decay in flexural properties. Another contributing factor might be the presence of larger particles on Gpw recyclates, which tend to be stress raisers, acting as failure initiation sites. This subject should be clarified in posterior study that will focus on microstructure analysis of mortar specimens.

Above 8% content in waste addition, slight decreases on both flexural and compressive strengths occur with regard to PMs with lower contents of GFRP waste. In fact, increasing waste content from 8% to 12% moves the estimated marginal mean of compressive strength response from up target downward. Regarding flexural strength response, the decay on loading capacity already observed from 4% to 8% in GFRP waste addition continues in a linear manner.

Nevertheless, mechanical strengths remain higher than those of plain mortars: for 12% content in waste addition, average increases of 4.2% and 6.2% were observed on flexural and compressive strengths, respectively, of modified mortars over waste-free PMs. As larger amounts of sand were replaced by GFRP waste throughout Gpw and Fpw test series, from 0% to 12%, overall specific surface area of aggregates was progressively increased, while resin content was kept constant to 20% in weight in all formulations. Thus, higher specific surface area of GFRP waste particles as regards to sand particles, requiring higher contents of binder matrix for a proper wettability and cohesive bonding, is for certain the main reason for observed turning point on materials' behaviour trend.

3.2.2 Effect of 'GFRP Waste Type'

In general, PMs modified with Gpw present improved mechanical behaviour over Fpw admixed PMs. For 4% addition of ground recyclates, higher increases in mechanical strength were observed: 10.2% and 8.3% increases in flexural and compression strengths, respectively, against 3.5% and 1.2% obtained for the finer waste. 8% and 12% additions of Gpw also lead to higher compressive strengths than the same contents of Fpw.

Gpw recyclates present a wide range of fibre lengths, varying between 25 mm and few micrometres. Maximum fibre length of Fpw is around 5 mm; thus, Gpw has a higher reinforcing effect than Fpw. This feature generally leads to improved mechanical behaviour of host material, providing that a good interface bonding is ensured. In general terms, taking into account the distinct geometric characteristics of Fpw and Gpw recyclates, it can be stated that whereas Fpw acts more like a filler extension for sand aggregates of modified mortar, leading to a less void-volume of resultant material; Gpw acts mainly as reinforcing material, conducting to improved mechanical strength of modified mortars. Nevertheless, at least on flexural behaviour concerning, no clear conclusions can be settled regarding the effect of 'GFRP Waste Type' on performance of modified PMs (as this factor, for a confidence level of 95%, was considered not influent or with little relative influence on global variance). Though, one issue must be stressed: once no real differences exist between Gpw-0 and Fpw-0 trial formulations, the effect of 'GFRP Waste Type' on global variance of mechanical responses was eventually weakened.

4. CONCLUSIONS

The feasibility of the incorporation of mechanically recycled GFRP wastes into polymer based mortars was investigated and assessed. With basis on the obtained test results the following conclusions may be drawn:

- The partial replacement of sand aggregates by GFRP waste recyclates has an overall incremental effect on both flexural and compressive strengths of resultant PMs, regardless the GFRP waste content (up to 12% w/w) and size grade (Gpw or Fpw).
- PMs modified with ground pultrusion waste (Gpw) show improved mechanical behaviour over fine pultrusion waste (Fpw) modified PCs, either in flexural or in compression. 4% and 8% in waste content constitute the turning points in the trend of the behaviour of PM materials for Fpw and Gpw based formulations, respectively.
- The observed dissimilar behaviour of trial formulations, according to both mechanical strength response (bending or compression) and size grade of GFRP recyclates, can be attributed to intrinsic differences between the geometric characteristics and specific surface areas of Fpw and Gpw admixtures; whereas Fpw acts more like a filler extension for sand aggregates of modified mortars, Gpw acts mainly as reinforcing material.

The outcomes of these experiments showed that a cost-effective and sustainable recycling solution for thermoset based GFRP wastes can be achieved, through mechanical recycling and the incorporation of the obtained recyclates into concrete-polymer composite materials as partial replacement of sand aggregates.

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