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Article

Insights into the Effect of Recycled Glass Fiber Reinforced Polymer on the Mechanical Strengths of Cement Mortar

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Abstract: The incorporation of recycled glass fiber reinforced polymer (rGFRP) in cementitious materials is an interesting recycling and valorization method. However, this incorporation generally results in a significant loss of workability, often compensated by an adjustment of the water to cement ratio, which can affect mechanical performance, particularly compressive strength. The aim of this paper is to examine the effect of different size fractions of rGFRP (0.063 mm, 0.16 mm, 0.63 mm, 1.25 mm and 2 mm) on the mechanical strengths of cement mortars with a mixing method that is likely to maintain the workability of the mix without adjusting the water to cement ratio. For this, a substitution rate of 10% (in volume of sand by rGFRP), supposed to induce workability loss, is chosen. A pre-mixing of rGFRP with water before adding cement and sand is performed and allows for the workability to be maintained without increasing the water content. The results show that compressive and flexural strengths are almost maintained compared with reference mortar for two rGFRP size fractions (2 mm and 0/2 mm). For the 2 mm fraction, a slight improvement (3%) in flexural strength after 7 months of curing and a 5% reduction in compressive strength are observed. After 7 months of curing, fibers or clusters of rGFRP are still observed, although they are not alkali-resistant.

Keywords: cement mortar; recycled GFRP; workability; water demand; strength



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1. Introduction

Glass fiber reinforced polymers (GFRPs), which consist of glass fibers dispersed in an organic resin (epoxy or polyester resin), are widely used in multiple applications, such as automotive, aerospace, shipyards and wind turbine blades [1]. GFRP dominates the composite market (over 95% market share) [1–3]. In recent decades, worldwide production and consumption of GFRP have increased. The consequence of this high level of production is a large flow of waste, raising the challenge of disposal method [2]. For instance, an estimated 100,000 tons per year of wind turbine blades constructed with GFRPs will be cumulated by 2030 [4]. Landfilling and incineration were the two commonly used disposal methods, but these solutions tend to be limited due to increasing environmental awareness [5–7] (European Union legislation (1999/31/CE) prohibits the landfilling of large composite parts).

In fact, the recyclability of GFRP is a great challenge due to the difficulty to recover fibers from resin. Different recycling methods are used including thermal (incineration with energy recovery, fluidized bed process, pyrolysis), chemical (nitric acid, solvolysis) and mechanical methods [1,4]. Chemical and thermal recycling methods, which display some limitations [1,4], are mainly used to reclaim carbon fiber from carbon fiber reinforced polymer (CFRP) due to the higher price of virgin carbon fibers [8,9]. The fibers reclaimed via gasification process have shown significant mechanical property reduction; for instance, a

33% decrease in tensile strength was reported [10]. Similar to pyrolysis, the fibers reclaimed via fluidized bed are fluffy with unstable dimensions, which makes it difficult to reuse and remanufacture recycled composite [4]. Unlike carbon composite, in the case of GFRP recycling strategies, low-cost recycling methods are preferred due to the lower price of virgin glass fiber [1,8]. This is why the mechanical recycling that consists of breaking down GFRP into smaller pieces for reuse [11,12] seems to be a more suitable alternative approach. In fact, a viable option to use mechanically recycled GFRP composites is to incorporate them as powder, fiber or aggregate in cementitious materials [13,14]. Recycled GFRP (rGFRP) elements are mixed in concrete as a partial replacement of fine and coarse aggregate or as a reinforcing element [1,15–17]. This alternative repurposing approach has the potential to be a long-term and sustainable solution for managing the growing GFRP wastes by reducing the environmental impacts of waste composites [1]. This solution offers several perspectives, notably in the construction sector, being able to absorb large amounts of GFRP waste. In addition, several studies have shown that the incorporation of GFRP waste into cementitious materials as a replacement for aggregates and fillers can improve mechanical properties [13,16–21]. It should be noted that this process is cost-effective compared with the thermal and chemical recycling [4]. However, this solution must be studied further in order to define a practical and viable methodology to avoid some problems [17,18].

The effect of the incorporation of recycled GFRP on the properties of cementitious materials depends on their form (powder or fibers) and their dosage [12,17,18,22,23]. In general, the incorporation of rGFRP results in a loss of workability often compensated by an increase in the water to cement ratio (or by the addition of superplasticizer) [6,16,18,24]. Oliveira et al. [24] found that partial replacement of sand with recycled GFRP decreases the mechanical strengths of cement mortar. The reduction in compressive strength is always greater than that of flexural strength. The replacement of 15% (by mass) of sand with recycled GFRP resulted in a reduction of 37% in flexural strength and 54% in compressive strength [18]. The reduction in strength can be attributed primarily to the adjustment of the water to cement ratio and to the lower compressive strength of the glass composite particles compared with sand. This results in a weaker interface between particles and the cement matrix, increased voids and an uneven distribution of glass composite waste [12,24]. The impact of rGFRP on the mechanical strength of concrete is related to the content of fiber-rich or resin-rich waste and to physical properties (shape, density, size, etc.) [1].

Other studies showed improved mechanical properties of concrete when rGFRP is used as reinforcement (recycled glass fibers). Patel et al. [6] observed an improvement in the mechanical strength of concrete containing 0.3% (by volume) recycled glass fibers [6]. Xu et al. [25] studied the incorporation of recycled fibers from wind turbine blades into concrete. They found a reduction of 14% in compressive strength with 2.5% (by volume) of recycled fibers, an increase of 38% in flexural strength and a significant increase in flexural toughness of 36.8 times.

Baturkin et al. [7] studied the valorization of rGFRP in the form of powder, aggregate or fibers as a substitute for cement and coarse aggregate. They found that mixes containing 10% rGFRP powder (as a replacement for cement) had compressive strength comparable with reference mixes after 90 days of curing. For concretes reinforced by rGFRP fibers, the flexural strength increased by 15% without a significant reduction in compressive strength.

Furthermore, several studies showed that the incorporation of recycled GFRP (rGFRP) into cementitious materials affects their workability [16,18,24,26]. Tittarelli and Moriconi [16] replaced 15% and 20% of the aggregate volume with GFRP industrial by-product powder and observed a significant reduction in mortar workability. The addition of superplasticizer improved the workability without additional water. Mastali et al. [26,27] reported that partial replacement of fine aggregates with GFRP powder has an impact on the workability of the mix, depending on the replacement rate. Indeed, they observed that replacing more than 5% of fine aggregates with GFRP powder resulted in a significant loss of workability. For substitution rates below 5%, an improvement in workability is observed. The decrease in workability is attributed to particle agglomeration, which increases wa-

ter demand [1]. Glass composite particles also have a high specific surface area, which increases the water demand [14]. The size, morphology and amount of rGFRP are the main influencing factors [1,13].

Tittarelli and Shah [22] found that the partial replacement of sand with rGFRP dust (5–10% by volume) reduced the viscosity and yield stress of cement paste, resulting in an increase of mortar workability. Farinha et al. [13] observed that for the same w/c ratio, replacing less than 20% of sand weight by rGFRP (smaller than 63 μm) increases workability. This is due to the fact that rGFRP absorb less water than fine mineral aggregates, helping to reduce water demand [13]. However, Oliveira et al. [24] noted that the progressive increase in the substitution rate of sand by rGFRP leads to a progressive increase in water demand. Correia et al. [17] reported that higher substitution rates of sand by GFRP (>5%) caused a significant increase in water demand and, consequently, a decrease in mechanical strengths, decreasing the compressive strength by about 22% and 47% with a 10% and 20% replacement rate (by volume), respectively. El Bitouri et al. [18] found that adjusting the water to cement ratio of mortar containing recycled GFRP can result in a significant loss of compressive strength, reaching 37% with 10% replacement (by volume).

All of these studies show that the incorporation of rGFRP in cementitious materials is an interesting avenue of valorization. This incorporation, particularly at a high replacement rate, generally leads to a loss of workability. In fact, rGFRP increases water demand [16–18,24,28], which in turn reduces workability. To maintain workability, the water to cement ratio (w/c) is generally adjusted. However, this compensation can affect the mechanical performance [17,18,24]. The aim of this study is to examine the effect of each fraction obtained after mechanical grinding of GFRP parts from end-of-life boats with a mixing method likely to reduce water demand. For this, a pre-mixing of rGFRP with water before adding cement and sand is performed at a high replacement rate of sand by rGFRP (10%vol). At such a replacement rate, it is often reported that there is a loss of workability with a significant decrease in mechanical strengths [1,13,18,20]. This study will help to define the suitable method to recover each fraction after mechanical recycling (substitution or reinforcement).

2. Materials and Methods

2.1. Materials

The recycled GFRP (rGFRP) used in this research was collected from end-of-life boat parts. Large pieces of GFRP were shredded in an Alpine[®] grinder, and then ground with a Retsch[®] SM300 grinder (Haan, Germany) at 1500 rpm using a 4 mm grid. Different size categories were selected after sieving using a set of sieves stacked vertically with mesh sizes typically ranging from 4 mm to 0.063 mm. Each selected fraction corresponds to the reject in the corresponding sieve (e.g., 1.25 mm fraction corresponds to the reject in 1.25 mm sieve, i.e., the fraction 1.25 mm/2 mm) (Figure 1). It should be noted that rGFRPs are mainly composed of a mixture of E-glass fiber and polyester resin, and the density varies from one fraction to another (Table 1). The calcination method was used to determine the resin content in each fraction according to the ISO 1172 standard [29]. The results are shown in Table 1.

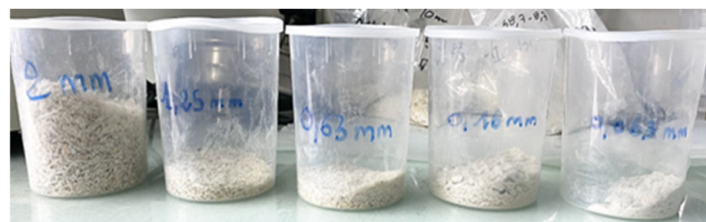


Figure 1. Different GFRP waste size categories.

Type III Portland cement (CEM III 42.5 N PM-ES) and standard sand [30] were used. The absolute density of raw materials was measured with a pycnometer (Micromeritics AccuPyc 1330, Micromeritics Instrument Corporation, Norcross, GA, USA). These measurements were performed in triplicate (Table 1).

Table 1. Density of raw materials and fiber/resin content in rGFRP.

Materials	Cement	Sand	rGFRP				
			2 mm	1.25 mm	0.63 mm	0.16 mm	0.063 mm
Measured density (g/cm ³)	3.2	2.7	2.1	1.9	1.7	1.6	1.6
Fiber/Resin content (%)	-	-	74/26	54/46	55/45	34/66	14/86

2.2. Mortar Preparation

The mixture procedure was carried out according to the EN 196-1 standard [30] with a slight adaptation to pre-wet and deagglomerate the rGFRP to avoid water entrapment. It was carried out at room temperature (approximately 20 °C) in an automatic mixer. Water and rGFRP were first placed in the mixer bowl and mixed (140 ± 5 rpm) for 30 s, then the cement was added and mixed for 30 s. The sand was added for 30 s, and the mixing speed (285 ± 10 rpm) was then increased for 30 s. The mixer was stopped for 90 s. During the first 30 s, the walls were scraped to homogenize the mixture. Finally, the mixer was started for 60 s at high speed (285 ± 10 rpm).

Samples were cast into prismatic molds ($40 \times 40 \times 160$ mm³) for 24 h at 20 °C and 100% RH (relative humidity). Then, they were demoulded and kept in water at 20 °C. Samples were weighed to determine their density.

Different formulations were produced: a reference mortar without rGFRP and six other mortars containing rGFRP with a replacement rate of 10% (by volume). Given the difference in density Table 1, the mass of rGFRP to be added differs from one fraction to another for a volume substitution rate that was kept constant (10%vol). For instance, the mixture E corresponds to mortar with 10% vol of rGFRP, in which the corresponding mass of rGFRP consists of different fractions, as shown in Table 2.

Table 2. Composition of mortars.

Samples	Cement (g)	Water (g)	Sand (g)	rGFRP (%)				
				2 mm	1.25 mm	0.63 mm	0.16 mm	0.063 mm
				Ref	450	225	1350	
A	450	225	1215					100
B	450	225	1215		50		50	
C	450	225	1215			100		
D	450	225	1215		33.3	33.3	33.3	
E	450	225	1215	33.3	16.5	16.5	16.5	16.5
F	450	225	1215	100				

3. Test Methods

3.1. Flow Test

A flow table test was performed to characterize the consistence of mortars according to the NF EN 1015-3 standard [31]. The fresh mortar has been placed on a flow table disc by means of a truncated conical mold (60 mm in height and with an internal diameter of 100 mm at the bottom and 70 mm at the top), and it was given a number of vertical impacts by raising the flow table and allowing it to fall freely through a given height. The spread diameter was measured by the mean diameter in two directions at right angles to one another using calipers. Three samples have been tested.

3.2. Mechanical Test

The flexural and compressive strengths were determined according to the NF EN 196-1 standard [30]. The flexural strength was determined on $40 \times 40 \times 160 \text{ mm}^3$ prisms using a three-point bending configuration (Figure 2a). It was carried out with a 3R RP40-40kN loading machine (Montauban, France). The compressive strength was measured on the two sample halves obtained after the prism had broken during bending test using 3R RP400-425kN (Montauban, France) with a loading speed of 2.4 kN/s (Figure 2b). Mechanical tests were performed at 7 days and 7 months.

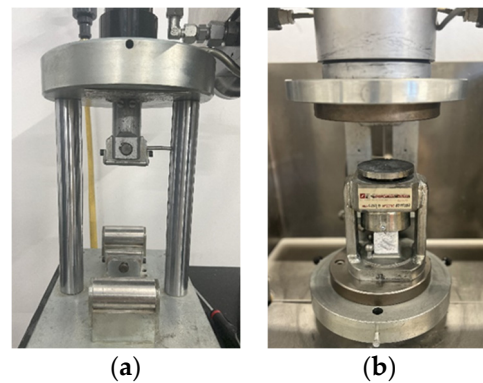


Figure 2. Compressive test (a) and three-point bending test (b).

3.3. Porosity Measurement

Porosity measurements were carried out according to the NF P 18-459 standard at $20^\circ \pm 2$ (except for the drying step) after 7 months of curing. This porosity characterizes the water accessibility and is determined as the ratio between the total volume of water-accessible voids and the volume of sample, using the following Equation (1).

$$\emptyset = \frac{M_s - M_d}{M_s - M_h} \times 100 \quad (1)$$

where: M_s is the mass of the saturated specimen, M_d is the mass of the dry specimen and M_h is the mass of the specimen immersed in water.

A prismatic sample of each mortar was placed in a vacuum container for at least 4 h. A water imbibition was performed in the pressure container for 45 h. The sample was then placed on a hydrostatic balance and weighed immersed (M_h). It was extracted from water and wiped quickly and carefully using a damp sponge in order to eliminate the surface water without removing the water from the pores, and it was weighed in air (M_s). Finally, the sample was dried at $105 \pm 5^\circ \text{C}$ until the first measurable constant mass was obtained (M_d). These measurements were carried out in triplicate.

3.4. Microscope Observations

Optical microscope observations were also performed on the mortar samples using a KEYENCE VHX-S650E (KEYENCE FRANCE SAS, Bois-Colombes, France) digital microscope. The observations were carried out on the samples from the mechanical tests (at 7 months) after rupture without prior preparation.

4. Results and Discussion

The incorporation of rGFRP has a deleterious effect on workability [16,18,28]. In this study, the mixing process has been modified to reduce the negative effect of rGFRP on workability. Thus, by mixing water and rGFRP first in the mixing process without adjusting the water to cement ratio, only a slight drop in workability is observed, as shown in Figure 3. The most significant decrease is observed for Samples A and F containing, respectively, fine particles (0.063 mm) and 2 mm particles. However, all formulations remain workable.

The use of this mixing procedure enabled sufficient workability to be achieved without the need for additional water (Figure 3). In fact, pre-mixing rGFRP with water allows for the deagglomeration of fibers (especially the smallest ones) under the action of shear and the release of water that may be trapped between these agglomerates. This improves workability without adding additional water to compensate for the water trapped by rGFRP. It must be kept in mind that other factors can affect the workability, such as the size of rGFRP, as observed for samples A and F. In fact, for sample A, it is likely that the mixing method could not release all the water trapped between the agglomerated particles (0.063 mm), while for sample F, the size of the particles (rather fibers > 2 mm) can affect the flow test.

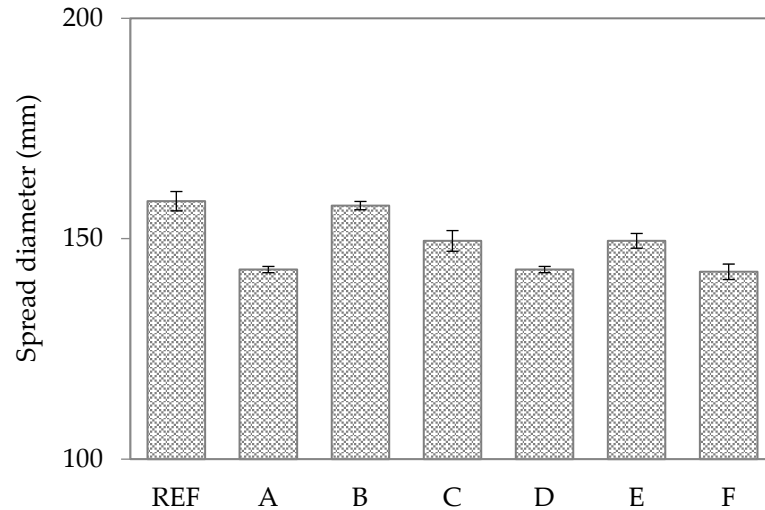


Figure 3. Spread diameter of fresh mortars obtained by flow table.

Figure 4 shows the loss of density of rGFRP mortars compared with the reference mortar (without rGFRP). The incorporation of rGFRP results in a decrease in mortar density. A significant loss of density was observed for mortars containing more fine particles (Sample A). Furthermore, the density loss is lower when the particle size distribution is fairly homogeneous (sample E). The loss of density can be attributed to the low density of rGFRP particles compared with sand aggregates. The higher density loss with fine particles can be explained by their lower density (1.6), as shown in Table 1 (resin content). In addition, a homogeneous size distribution of rGFRP particles can improve the compactness (filling effect).

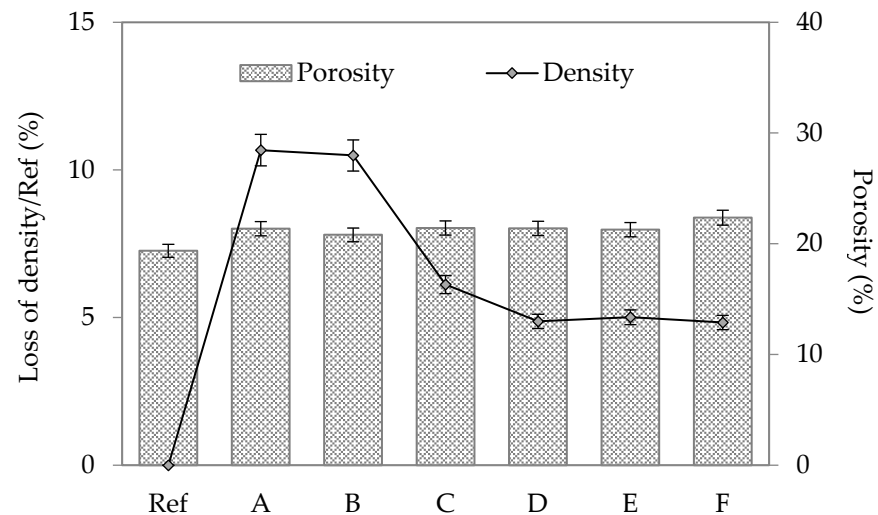


Figure 4. Loss of density (compared with density of reference mortar 2.25) and porosity of tested mortars.

Water accessible porosity was measured after 7 months of curing. The results show a slight increase in porosity with the incorporation of rGFRP (Figure 4). It should be kept in mind that these results only characterize the porosity accessible to water and cannot explain the decrease in density (and mechanical strength). Indeed, other factors can explain these decreases (entrained air, difference in density, etc.).

Compressive strength was measured in samples cured for 7 days and 7 months with different particle sizes of rGFRP. Figure 5a presents the obtained results. All samples showed increased compressive strength as curing time increased due to cement hydration. For each sample, after 7 months of curing, compressive strength reached at least 40 MPa. Furthermore, samples with lower density reduction showed better compressive strength. Figure 5b shows the percentage change in the various compressive strengths of the different samples containing rGFRP compared with the reference mortar. A reduction in compressive strength for all specimens containing rGFRP can be observed. However, this reduction was less significant for samples E and F. Sample A containing only fine particles displays a significant reduction in compressive strength, reaching 31%. Samples E and F exhibit the lowest reduction in compressive strength compared with the reference mortar (<10%).

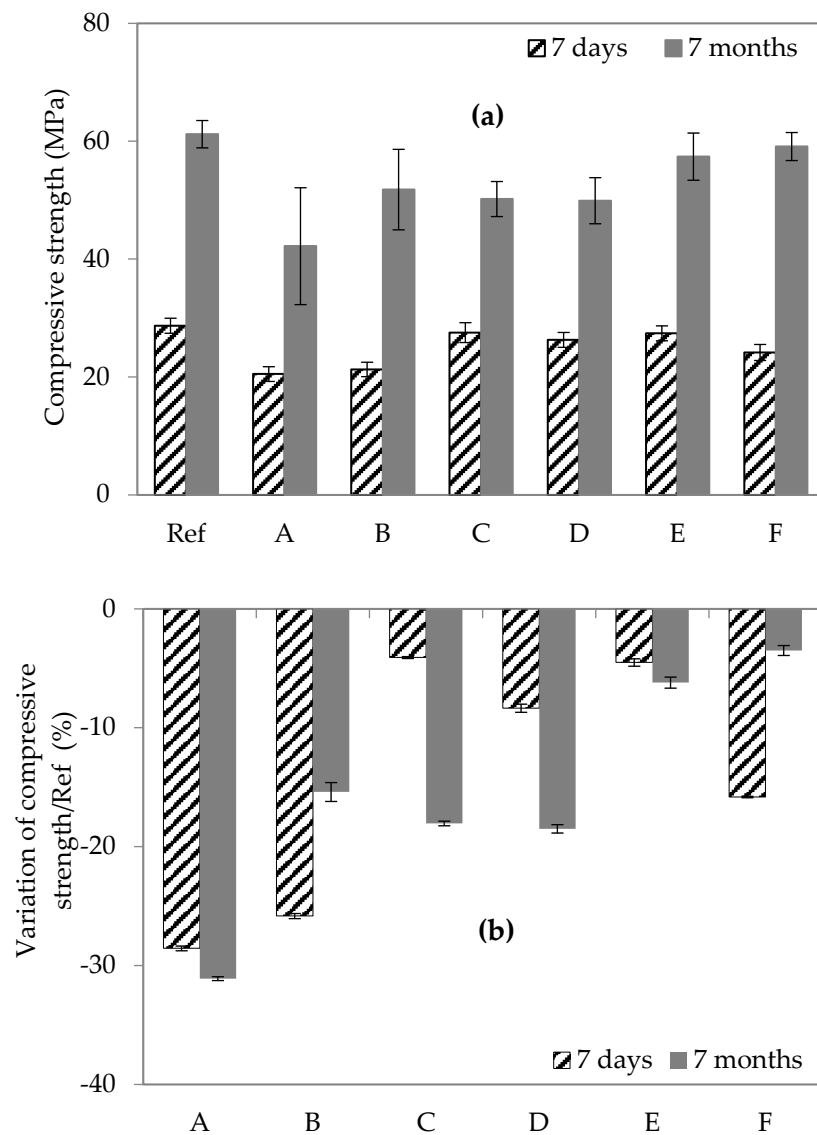


Figure 5. (a) Compressive strength of mortar at 7 days and 7 months; (b) variation of compressive strength (%) compared with reference mortar.

These results seem promising as in previous studies, when considering the adjustment of the w/c ratio to compensate for the loss of workability, a significant reduction in compressive strength was often observed [16–18]. The mixing procedure performed in this study allowed for the workability to be maintained and limited the loss of compressive strength, which does not exceed 10% in samples E and F. In fact, El Bitouri et al. [18] used a size distribution close to that of sample E with an adjusted water/cement ratio to maintain workability. In their study, the compressive strength decreased by 37% and the flexural strength by 17%.

Figure 6a shows the flexural strength of different samples. It can be noted that the flexural strength increases with curing time from about 5 to 6 MPa at 7 days to about 10 MPa at 7 months. After 7 months of curing, no significant difference between the flexural strength of the reference mortar and that of mortar containing various sizes of rGFRP is observed. Figure 6b shows the variation of flexural strength of mortar with rGFRP compared with the reference mortar. A slight improvement of about 2.7% is observed in sample F at 7 months.

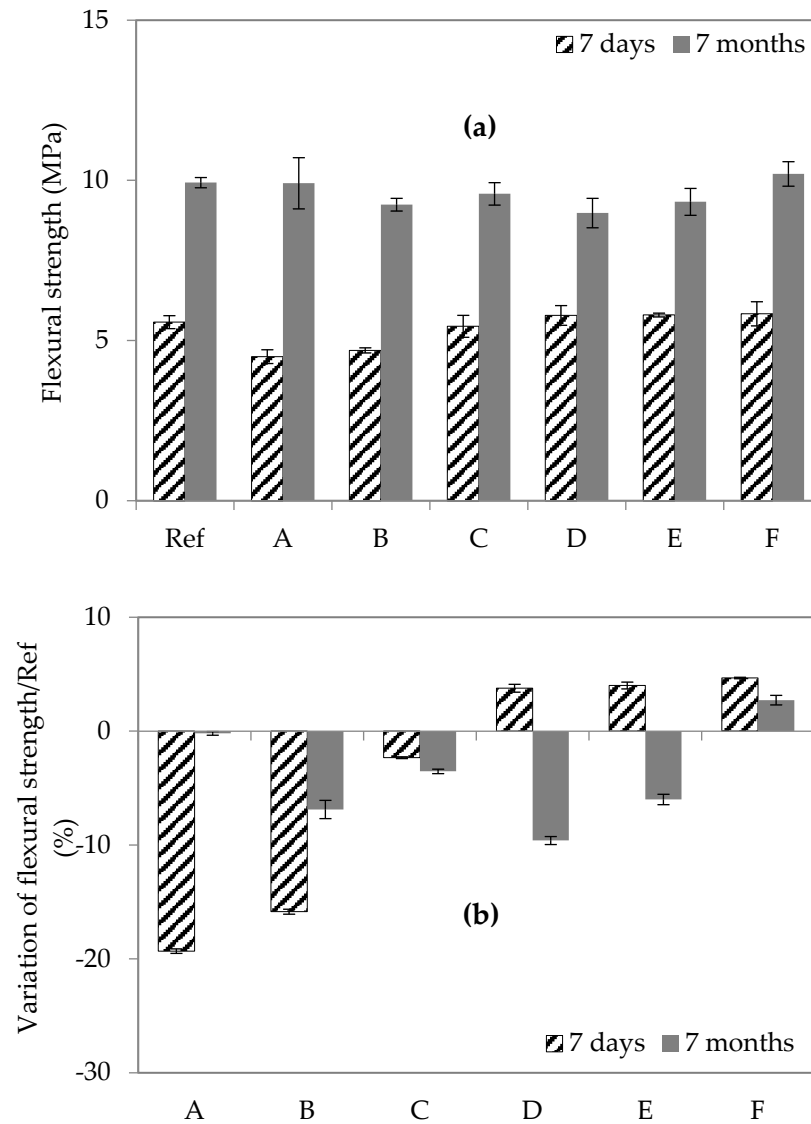


Figure 6. (a) Flexural strength of mortar at 7 days and 7 months; (b) variation of flexural strength (%) compared with the reference mortar.

It is worth noting that the differences in mechanical strength between samples at 7 days are not always maintained after 7 months of curing. Some samples show an improvement

in flexural strength at 7 days and a reduction at 7 months (samples D and E). The loss in mechanical strength observed at 7 days tends to decrease at 7 months in some samples (samples A and B). In addition, it seems that compressive strength is more affected by the replacement of sand by rGFRP than flexural strength, as observed previously by other authors [16–18].

Figure 7 shows examples of the failure mode of the samples during the bending test. Samples without fibers exhibit clear brittle fracture, with the specimens splitting into two halves after rupture. Conversely, specimens with rGFRP show crack formation during rupture but do not split into two halves, indicating that brittle behavior is attenuated. This has already been observed in a previous study [18] with a significant improvement in flexural toughness. It is interesting to note that the improvement in flexural toughness strongly depends on the dimension of rGFRP as the sample containing mainly 2 mm fibers (sample F) exhibits the least brittle fracture with slower crack propagation.

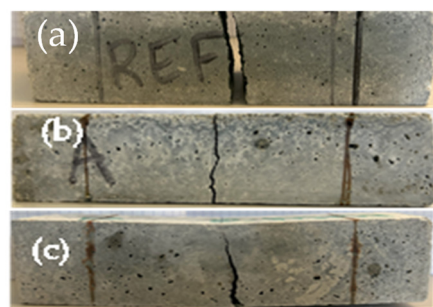


Figure 7. Cracking mode of mortar samples after flexural testing (at 7 months): (a) reference mortar; (b) sample A; (c) sample F.

Microscopic observations after mechanical tests were carried out to observe the presence of rGFRP in the cement matrix after 7 months of curing. Figure 8a shows the surface mapping of a mortar sample containing only rGFRP powder (0.063 mm), with no significant particle agglomeration being observed. It also shows a distribution of small clusters of particles (rGFRP powder) scattered over the mortar surface. Figure 8 shows the surface of sample E (particle size from 0.063 mm to 2 mm), where glass fiber bundles are observed to separate into filaments and disperse randomly in the matrix, which could improve resistance to crack propagation. When the rGFRP incorporated are only 2 mm size (sample F), as shown in Figure 8f, a few glass fiber filaments are observed on the surface with an uneven distribution, but most fibers are still bundled with a resin shell. Microscopic observation shows that after 7 months of curing, the rGFRP particles are still present in the matrix and show no deterioration, despite the fact that the glass fibers are not alkali-resistant.

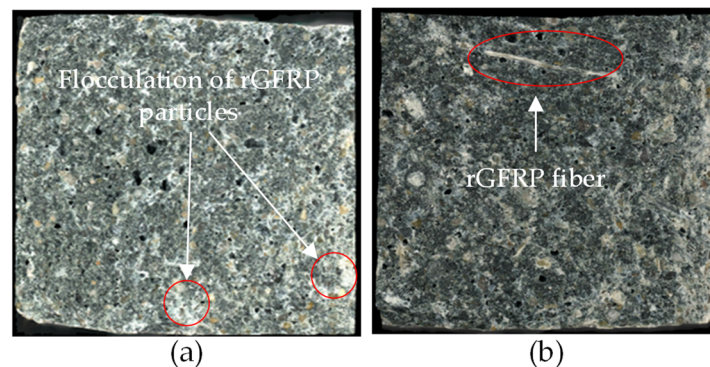


Figure 8. Cont.

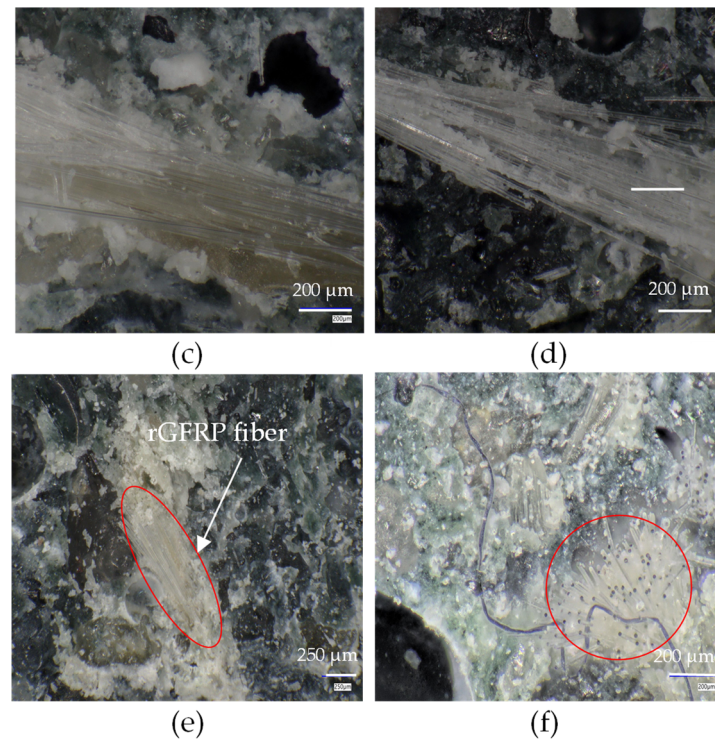


Figure 8. Microscopic images of mortars containing rGFRP. (a) Sample A; (b–d) sample E; (e) and (f) sample F.

5. Conclusions

This study aimed to examine the effect of different rGFRP fractions obtained by sieving after grinding large pieces of end-of-life boats on the mechanical properties of mortar. The mixing procedure was slightly adapted to limit the loss of workability. In fact, it is often reported in the literature that the incorporation of rGFRP in cement mortar at a high substitution rate (>5%) results in a significant loss of workability, leading to an adjustment of the water to cement ratio. This results in a significant reduction in the mechanical performance, particularly the compressive strength.

The main conclusions of this study are:

- The pre-mixing of water and rGFRP before adding cement and sand allowed the workability of all studied mixes to be maintained without adjusting the water to cement ratio.
- The incorporation of rGFRP results in a decrease in mortar density. A significant loss of density was observed for mortars containing more fine particles due to the resin content.
- A slight increase in water accessible porosity with the incorporation of rGFRP was observed.
- No significant reduction in compressive strength for sample E (containing 0/2 mm) was observed, while sample F containing mainly glass fibers (>2 mm) exhibited a slight improvement in flexural strength.
- No significant reduction was observed in compressive strength for sample F containing mainly fibers >2 mm.
- Sample E seems the most suitable for a substitution of a part of sand, whereas for a reinforcement approach, particularly for crack propagation control, sample F containing mainly glass fibers (>2 mm) seems the most relevant.
- rGFRP particles are still present in the cement matrix after 7 months of curing, despite the fact that the glass fibers are not alkali-resistant.

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