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María Rico[1]
Zelaya J.R.[1]
García, B[1]
Vanegas Daniela[1]

1. Institución Universitaria Colegio Mayor de Antioquia

Autor de correspondencia:

Zelaya, J.R.
Maestría en Construcción
Institución Universitaria Colegio Mayor de Antioquia
Correo electrónico: jose.zelaya@colmayor.edu.co

Abstract

Glass fiber-reinforced polymer (GFRP) composite materials are used extensively and increasingly in major industries including aerospace, marine, construction, electrical and automotive. These types of materials have advantages such as low densities, high mechanical properties in certain directions, ease of production and durability. However, its manufacture generates residues and at the end of its life cycle both the fiber and the resin cannot be easily decomposed or recycled. This article explored the influence of recycled GFRPs as reinforcement in limestone calcined clay cements mortar mixes with ratios of 2%,4%,6%, 8% and 10% replacing the fine aggregate weight in the flexural strength, compressive strength, and density. Results demonstrated flexural and compressive strengths in the range of 2.5 MPa-7MPa and 12-28 MPa respectively at the ages of 28 and 56 days.

Keywords: Limestone calcined clay cement; recycled glass fiber reinforced polymer; mortar; mechanical properties

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Propiedades mecánicas de polímeros reforzados con fibra de vidrio reciclado (rGFRP) en morteros de cemento de arcilla calcinada

Resumen

Los materiales compuestos de polímero reforzado con fibra de vidrio (GFRP) se utilizan ampliamente y cada vez más en las principales industrias, incluidas la aeroespacial, marina, de construcción, eléctrica y automotriz. Este tipo de materiales presentan ventajas como bajas densidades, altas propiedades mecánicas en determinadas direcciones, facilidad de producción y durabilidad. Sin embargo, su fabricación genera residuos y al final de su ciclo de vida tanto la fibra como la resina no pueden descomponerse ni reciclarse fácilmente. Este artículo exploró la influencia de los PRFV reciclados como refuerzo en mezclas de mortero de cemento de arcilla calcinada con proporciones de 2%, 4%, 6%, 8% y 10% reemplazando el peso del agregado fino en la resistencia a la flexión, la resistencia a la compresión y la densidad. Los resultados demostraron resistencias a la flexión y a la compresión en el rango de 2,5 MPa-7 MPa y 12-28 MPa respectivamente a las edades de 28 y 56 días.

Palabras clave: Cemento de arcilla calcinada de piedra caliza; polímero reforzado con fibra de vidrio reciclado; mortero; propiedades mecánicas

1. Introduction

Fiber-reinforced polymer (FRP) composite materials are used in many types of industries such as: aerospace, marine, construction, electrical, automotive, domestic appliances, furniture, and sports equipment, consisting of continuous or discrete fibers made of glass, carbon, or aramid encased in a thermosetting resin with concentrations in the range of 12%-60% by volume. The main advantages of composite materials (CM) over traditional construction materials (steel and concrete) are lower density, higher mechanical properties in certain directions, ease of production and installation, and greater durability in harsh chemical and aqueous environments(Patel et al. 2019; Yazdanbakhsh and Bank 2014). It is expected that within the next two decades this industry could grow between 10-15 (USD Billions) for different applications, especially those containing glass and carbon fibers (Clark et al. 2020) for the construction industry. Among them the global glass fiber reinforced polymer (GFRP) composites annual production is estimated at 8 million metric tons that generate 1.5 million metric tons of waste (Dehghan, Peterson, and Shvarzman 2017).

However, FRP composites have certain disadvantages such as lack of standard profiles and parts, design specifications and difficulty in recycling. These materials generate two different sources of waste; one is the scrap during the production process and the second is the end-of-life disposal (Yazdanbakhsh and Bank 2014). Since the resins used cannot be easily separated from the fiber, decomposed, or recycle, hence landfill and incineration are the most common methods for waste management. Recently, closed loop recycling has become an environmental-friendly approach, involving the collection of postconsumer waste in new applications, restricted by the quality of the raw materials(Kazmi, Williams, and Serati 2020) and an either chemical or mechanical processing. In this technique, waste of one system becomes the input of another, resulting in the elimination of waste disposal challenges and a reduction in the demand for new raw materials. GFRP requires a mechanical recycling to break down the composite material, into a reduced particle size through shredding, crushing, milling and turn them into resin and fibrous products (Yazdanbakhsh and Bank 2014).

Among the most used methods to use the mechanically process residue from GFRP wastes are either as fillers or reinforcements in Portland cement-based mortars and concretes. Portland cement based composite materials have a ceramic like type of cracking process that starts at a micro-scale under applied stresses due to formation and propagation in the interfacial transition zone (ITZ) between the cement paste and the aggregates, then it spreads through the paste until it grows into a macro-crack leaving the material with low tensile load bearing capacity. The use of dispersed fibers with different sizes generates a multiscale mechanism needed to control the different stages of crack growth to improve its durability, as well as different alternatives for reinforcement, and to make it lightweight (Shafei et al. 2021) (Song, Purnell, and Richardson 2015) (Khan et al. 2020) for a more sustainable industry.

Some studies have reported that fiber polymer matrix composites can be used as fillers in cement-based materials that can increase strength and durability for offshore applications because of the versatility and chemical stability under extreme conditions(Clark et al. 2020).

(García, Vegas, and Cacho 2014) explored different techniques such as shredding, milling and screening on four different types of GFRP composites that aim to obtain optimized fiber concentrate for use in cement-based materials. Substitution of 0%, 5% and 10% standard silica sand was made in which a reduction in both flexural and compressive strength at the age of 7 days in a range of 10 to 60% using a single grinding process. However, further mechanical recycling was applied in the composites and the process yielded improvements in mechanical flexural and compressive strengths of up to 16% and 22% with 1% of recycled GFRP in comparison to the reference mix.

Recycled GFRP composites has been obtained from end-of-life wind turbine blades as partial replacement of sand in ordinary Portland cement (OPC)-based mortars, using four different types of sizes of aggregate (large, medium, small and powder) evaluating compressive strength, flexural strength, and toughness index among its main properties. Results demonstrated that large particles increased compressive strength by 2% using 3% of reinforcement, and negligible changes and reduction was reported for the other specimens, increased for flexural strength by 23%, and toughness index values higher than 1 compared to other sizes (Rodin et al. 2018). While others used three different sources of GFRP recyclates via mechanical routes, compared to plain OPC mortar 7 and 28-days compressive strengths presented different extents of strength increase attributed to the bonding strength of one of the fibers to the mortar matrix (Zhou et al. 2021).

Another major strategy to reduce carbon emissions is through the use of supplementary cementitious materials (SCMs) with OPC, which helps prevent the virgin resource consumption and enhances the microstructure of cement paste(Amer, Rangaraju, and Rashidian-Dezfouli 2021; Vali et al. 2020). Today more than 80% of SCMs are either fly ash, limestone, or slag, but calcined clays in combination with limestone (LC3 technology) as partial replacement of clinker, could become a more viable alternative since they are widely available in equatorial to subtropical parts of the world. It needs to be heated to temperatures close to 700-850 °C to form metakaolin, an amorphous alumino-silicate that can react with calcium hydroxide to give C-(A)-S-H and aluminate hydrates that contribute to the strength, durability and decrement alkalinity in the binder(Baghban and Mahjoub 2020; Scrivener, Martirena, et al. 2018) making it an alternative and compatible solution for different sources of fiber in which traditional OPC can easily degrade.

Kaolinitic clays, are available in very large quantities, have excellent reactivity after calcination making a substantial contribution to reducing carbon dioxide emission associated with the production of cementitious materials. The clays used do not need high kaolinite content, in some cases above around 40% can perform as well. Limestone is highly available and low energy consumption during its grinding enabling its use for adjustment of particle size distribution of cementitious components to enhance the workability and early-age strength (Avet and Scrivener 2020; Scrivener, Avet, et al. 2018).

The present investigation explores the use of recycled GFRP composites as a discrete reinforcement in limestone calcined clay cement based mortars. This study analyses its influence on the mechanical properties (compressive and flexural strength) and density of the material.

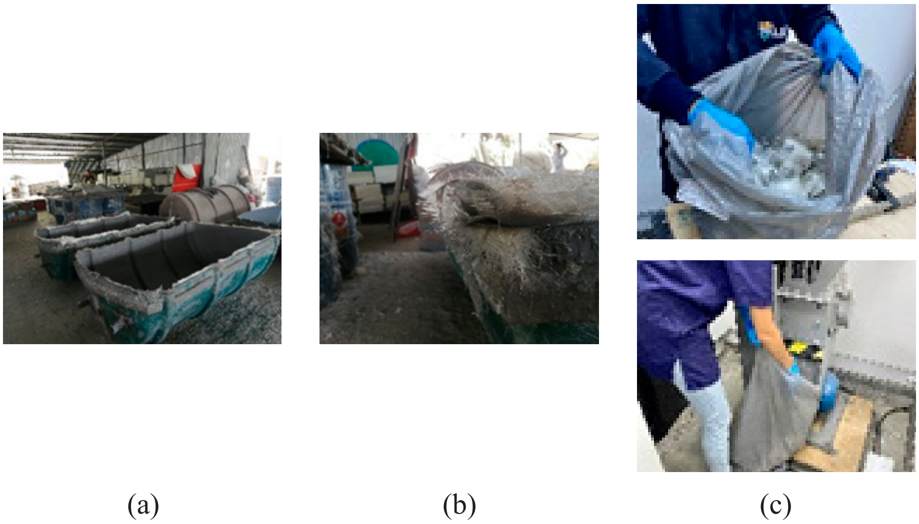
2. Materials and methods

Recycled GFRP

The reinforcing materials used in this study to produce limestone calcined clay cement mortar are from residues generated during the elaboration of septic tanks located in la Estrella Medellin, Antioquia.

The mechanical recycling process starts with cutting some of the larger pieces into smaller ones before being introduce into a shredder Figure 1.

Figure 1. *a) Septic tanks b) Glass fiber reinforced polymer residue c) Shredding process*



(a) (b) (c)

The result of the shredding process is a mixture of fines, fibers, clusters, and flake-like materials as can be seen in Figure 2a. The materials were then separated through a physical process separating coarse and fine particles with a series of square mesh sieves. Figure 2b shows a magnified view of the recycled fibers measured with a ZEISS Stemi 508 stereo microscope. Different sizes of clusters and agglomerated glass fibers can be observed. The results of the sieve analysis performed are provided in figure 2c. The shredded material can be categorized into powder (passed 0.3mm), fiber cluster (passed 9.5mm and remained on 0.3mm sieve) flake (remained on 9.5mm sieve) and some tiny fibers were conglomerated into balls (Zhou et al. 2021). The rGFRP fiber cluster and fibers were used as reinforcements in mortars herein.

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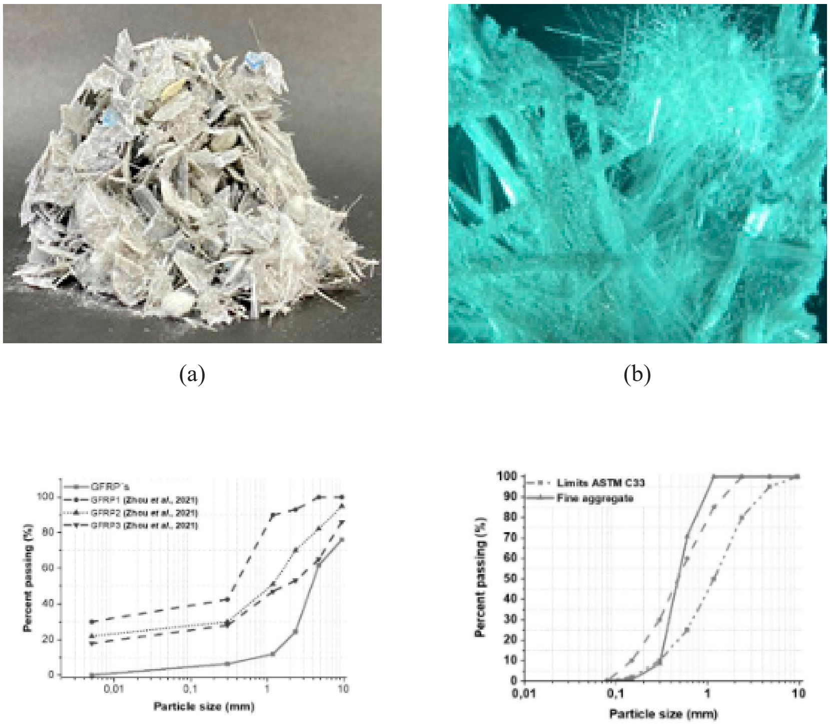
Figure 2. *a) Recycled glass fiber reinforced polymer (rGFRP) b) Microphotograph of rGFRP with magnification of 0.63x c) Particle size distribution of rGFRP d) Particle size distribution of quartz sand.*



(a) (b)

(c) (d)

The physical properties evaluated at the fibers were its relative density and water absorbency tested, using the criteria of what states the ASTM C128 being 1.71 and 36% respectively. According to (Zhou et al. 2021) some glass fibers possessed superhydrophobic, hydrophobic and hydrophilic surface.

Cement and sand

Limestone calcined clay cement was obtained from Argos Colombia also known as “cemento verde” and quartz sand from Indusilika S.A.S. with specific gravities of 2.87 and 2.50 respectively. The water absorption of the sand was 0.41%, the fineness moduli of 2.20. Figure 2d shows the particle size distribution of the quartz sand.

Additive

A superplasticizer, Plastol 6000 manufactured by Toxement was used following the recommended compositions and instructions for use.

Sample preparation and testing

The rGFRP fiber clusters were mixed into the cement mortars at 0 wt%,2 wt%,4 wt%, 6 wt%, 8 wt% and 10 wt%, based on wt% replacement of aggregate, it was also brought to a saturated surface dry state prior to mixing and study its influence on the mechanical and physical properties of cement mortar. A fixed water to cementitious ratio of 0.50, a mass ratio of cement to sand of 1:2. Water corrections were made on the mixture based on the absorption of the aggregate and the used of superplasticizer of 1.0% in all mixes.

Specimens were mixed according to ASTM C305 using a bench top mixer. The mixture was poured into cubic molds (50x50x50 mm) as stated by ASTM C109 to evaluate its compressive strength. Flexure prisms were cast in 40x40x160 mm to evaluate its flexural strength according to ASTM C348. All specimens were hit on the sides with a rubber asphalt mallet to remove entrapped air and then finished with a trowel on the surface. Compressive and flexural strength specimens were demolded after 24 hours and cured at 25 °C and 100% relative humidity for 28 and 56 days.

The hardened density of each specimen was calculated by dividing the dry mass and the volume of each specimen. Its volume was determined by calculating the dimensions of each side of the specimen using an average of two caliper measurements.

Optical Microscopy

Stereo microscope is a device designed for low magnification observation visualization of the sample. Photomicrography of the fracture surface of the limestone calcined clay reinforced mortars was obtained by using ZEISS Stemi 508 with ZEISS Axiocam 208 color and lens 5 Apo 1.5x FWD 53 mm.

Statistical design analysis

A one-way analysis of variance using a general linear model and Fisher’s least significant difference (SD) test ($P<0.05$) were used to separate means. We assessed the normality of the data for ANOVA using the Shapiro Wilk test. (Walpole et al. 2012). Here the factor corresponds to the percentage of rGFRP and five number of levels (2, 4, 6, 8, 10%) representing the amount of material used for sand substitution. Three replications for each level were carried out. ANOVA was adopted for testing the significance of the main effect and interaction on the compressive and flexural strength of the material. All statistical analyses were conducted using RStudio software (version 2023.09.1 Build 494).

3. Results and discussions

Mechanical properties

Figure 3a illustrates the compressive strength (CS) of the mixtures prepared with 0%, 2%, 4%, 6%, 8% and 10% of sand substituted for rGFRP. Compared to plain mortar, 28 day and 56 days strengths were reduced in ranges between 7.6%-50.91% and 22.2% and 50.88% respectively as the percentage of rGFRP increased. These reduction might be due to the following reasons: a) strength reductions in these types of composites may be caused by the fact that small particles having a higher the surface area requires a greater binder content to allow complete bonding throughout the matrix, while large particles could have added a weak linkage causing a disrupt in the continuity of the cement matrix hence its load paths (Rodin et al. 2018) and b) insufficient dispersion of the agglomerated small fibers during the mixing process throughout the matrix(Odera, Onukwuli, and Aigbodion 2018).

It can be observed that among every mixture there is an increased in strength of 12.25%- 41.77% comparing 28- and 56-days, suggesting that the alkalinity of the calcined clay cement mortar has little impact on the degradation of the rGFRP reinforcement, further testing is needed.

Figure 3b presents the flexural strength of the mixtures as a function of the rGFRP content, presenting a similar behavior with a decreased up to 50.82% compared to control mixture with 10% of fibers. The influence of the rGFRP on the mortars might be caused by the different dimensions of the fibers, if too short they had less contribution while too high could be detrimental specially those particles that contain resin coating which may provide an extra slipping resistance on the fiber-cement paste interfaces (Zhou et al. 2021).

The ANOVA of the 28 days and 56 days for both CS and FS results were conducted and F-values along with p-values are presented in Table 1, Table 2, Table 3 and Table 4. The higher value of F-value and p-value less than 0.05 establishes that there is a significant effect on the amount of rGFRP for at least two percentages at different ages. The Shapiro Wilk normality test P-value for CS and FS for both ages were 0.98, 0.30, 0.36 and 0.48 respectively. Table 5 presents the results of Fisher's least significant difference test analysis, the addition of rGFRP in the cement matrix with different percentages showed variability in the results.

Table 1. *ANOVA of 28-days CS*

Source	DF	SS	MS	F	P
%rGFRP	5	294.22	58.84	61.2	$4.03x10^{-8}$
Residuals	12	11.54	0.96		

DF=Degrees of freedom, SS=Sum of squares, MS=Mean Square, F=F value, P=P value

Table 2. *ANOVA of 28-days FS*

Source	DF	SS	MS	F	P
%rGFRP	5	12.64	2.53	22.49	1.04×10^{-5}
Residuals	12	1.35	0.11		

DF=Degrees of freedom, SS=Sum of squares, MS=Mean Square, F=F value, P=P value

Table 3. *ANOVA of 56-days CS*

Source	DF	SS	MS	F	P
%rGFRP	5	401.2	80.24	110.2	1.34×10^{-9}
Residuals	12	8.7	0.73		

DF=Degrees of freedom, SS=Sum of squares, MS=Mean Square, F=F value, P=P value

Table 4. *ANOVA of 56-days FS*

Source	DF	SS	MS	F	P
%rGFRP	5	21.81	4.36	10.48	0.000473
Residuals	12	4.99	0.42		

DF=Degrees of freedom, SS=Sum of squares, MS=Mean Square, F=F value, P=P value

Table 5. Fisher's Least Significant difference analysis

	28-days CS		28-day FS		56-day CS		56-day CS	
Treatment	MPa	Groups	MPa	Groups	MPa	Groups	MPa	Groups
0% rGFRP	21.78	A	4.72	A	29.02	A	7.0	A
2% rGFRP	20.12	A	4.01	B	22.57	B	5.38	B
4% rGFRP	16.53	B	2.97	C	20.96	C	4.96	BC
6% rGFRP	13.12	C	2.86	C	18.73	D	4.72	BC
8% rGFRP	12.60	C	2.55	C	16.62	E	4.20	CD
10% rGFRP	10.69	D	2.40	C	14.29	F	3.44	D

Numbers followed by the same letter within a column are not significantly different according to Fisher´s least significant difference (LSD) test (alpha=0.05)

The fiber´s content, length, distribution, and orientation are main variables that can affect the overall mechanical properties of the mortar (Cihan and Avşar 2022). Particle size analysis showed in figure 2a and 2c presents the different shapes, and sizes of the GFRP recyclates, results revealed that almost 70% of the particles are between 0.3 and 9.5mm and 30% less than 0.3mm considered clusters and powders respectively. Clusters introduces stress concentrations and pores in the mortars which affects negatively the overall mechanical performance of the composite (Zhou et al. 2021). Other studies (Asokan, Osmani, and Price 2009)(Asokan, Osmani, and Price 2010) have demonstrated that the use of recycled waste powder can improved the CS of mortars in contents lower than 15% of substitution of sand in the mixture in oven cured specimens at 50 °C by creating a polymeric film which intermingled with cements hydrates. For this study the higher contents of clusters negatively affected the mechanical performance.

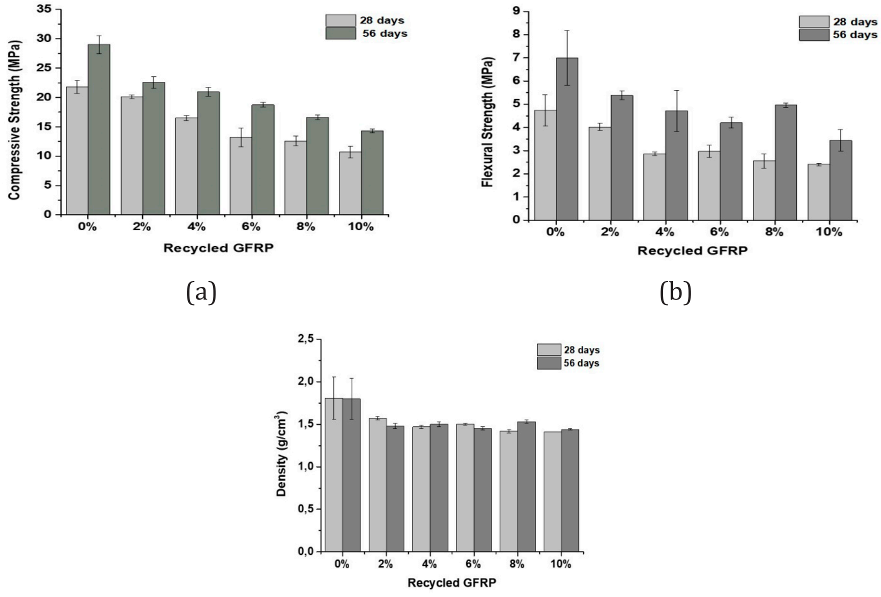
Furthermore, both compressive and flexural strengths results are consistent with other studies. For instance, the reyclates were able to increase both properties using low (0.5-2.0%) of fiber content with lengths between 10-30mm of recycled fibers from GFRP sheets with 0.11 mm thickness (Mastali et al. 2018), while the use of a thermoset composite (thermostable resin and glass fiber) from a pyrolysis process where the proportions of the mixtures included 5%, 10% and 15% wt% had a negative effect on the overall mechanical performance, the higher the content the lower the compressive and flexural strength (Criado et al. 2014).

The negative results showed there is a need to improve the bond quality of the recycled GFRP, studies like (Xiong et al. 2006), have suggested the use of a silane coupling agent with an appropriate concentration can modify the microstructure of the interfacial transition zone and therefore significantly increase the bond strength with the cement matrix, but further testing is required.

Density

Density is a property that establishes a relationship between the dry mass and the volume of a solid material. However, it can be considered an indirect method to relate the number of voids, the higher the density gives a denser microstructure with fewer voids(Zaid et al. 2021). Figure 3 shows the density of the rGFRP mortar with different percentage contents. A reduction of 13% compared to the control sample was achieved by using the different dosages. This slight decrease is expected when substituting sand in mortars due to the lower specific gravity of the rGFRP recyclates of 1.70 as to the sand of 2.50(Kazmi et al. 2020).

Figure. 3. *Mechanical and physical properties a) compressive and b) flexural strength (c) Density*



(a) (b)

(c)

The ANOVA of the 28 days and 56 days for both CS and FS results were conducted and F-values along with p-values are presented in Table 6 and Table 7. The higher value of F-value and p-value less than 0.05 establishes that there is a significant effect on the amount of rGFRP for at least two percentages affecting the density of the mortar. The Shapiro Wilk normality test P-value for the different densities at the age of 28 and 56 days are 0.43 and 0.38 respectively. Table 8 presents the results of Fisher's least significant difference test analysis, the addition of rGFRP in the cement matrix with different percentages showed an effect in its density.

Table 6. *ANOVA 28 days density*

Source	DF	SS	MS	F	P
%rGFRP	5	0.13	0.03	62	$3.74\text{x}10^{-8}$
Residuals	12	0.005	0.00042		

DF=Degrees of freedom, SS=Sum of squares, MS=Mean Square, F=F value, P=P value

Table 7. *ANOVA 56 days density*

Source	DF	SS	MS	F	P
%rGFRP	5	0.14	0.028	82.86	$7.03x10^{-9}$
Residuals	12	0.004	0.00033		

DF=Degrees of freedom, SS=Sum of squares, MS=Mean Square, F=F value, P=P value

Table 8. *Fisher's Least Significant difference analysis*

	28-days		56-day	
Treatment	Density (g/cm3)	Groups	Density (g/cm3)	Groups
0% rGFRP	2.06	A	2.08	A
2% rGFRP	1.98	B	1.91	B
4% rGFRP	1.91	C	1.88	B
6% rGFRP	1.86	D	1.88	BC
8% rGFRP	1.86	D	1.85	C
10% rGFRP	1.81	E	1.81	D

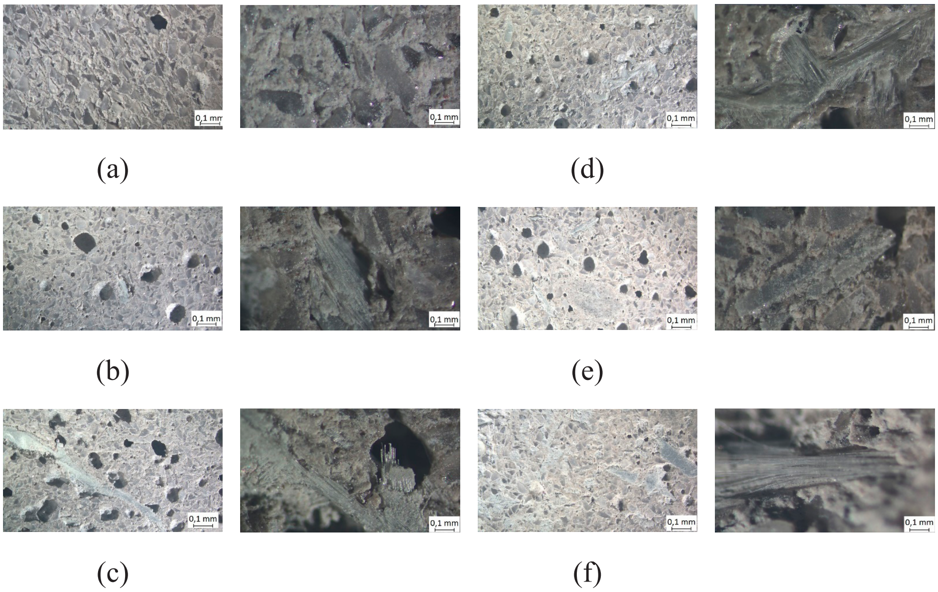
Numbers followed by the same letter within a column are not significantly different according to Fisher´s least significant difference (LSD) test (alpha=0.05)

Optical Microstructure

A stereo microscope was used to analyze the microstructure of the mortar samples at the age of 56 days with different contents of rGFRP. The samples were inspected without any cover. Figure 4(a)-(f) indicates details of the fracture surfaces. Figure 4a shows a detail of the control sample where a very dense and homogeneous matrix can be observed. The effect of the different additions of reinforcement in Figure 4 (b) and(f) respectively. After the milling and sieving process, a part of the recyclates is composed of glass fibers only that can be seen in figure 4 (c) and (d) which have better linkage with the matrix while the composite (glass fiber with remains of polymeric resin) appears to have low adherence according to figures 4 (b) and (f).

Figure 4 presents the variation in the porosity of the mortar due to an increase in the rGFRP content which affected the compressive and flexural strength, as previously discussed and at the same time the overall density of the mortar, which has good agreement with studies already reported (Singh et al. 2022)(Rodin et al. 2018).

Figure 4. *Microphotographs of rGFRP´s mortar with reinforcement at 56 days a) 0% control sample, b) 2%, c) 4%, d) 6%, e) 8%, f) 10%. Magnifications at 0.63x and 3.2x*



4. Conclusions

Recycle GFRP was milled and sieved to use as a reinforcement on limestone calcined clay cement mortars to evaluate its effect on mechanical and physical properties. The following conclusions were drawn:

1. The shredding and sieving process of the GFRP composite resulted in a series of clusters, agglomerated glass fibers and glass powder that negatively affected both compressive and flexural strength of the composite material up to 50% due to a probable weak linkage of the cluster with the cement paste and a nonuniform dispersion of the glass fibers.

2. The use of rGFRP in limestone calcined clay cement mortars was able to reduce the density of mixture up to13%.
3. Compressive and flexural strength improve after 28 and 56 days of curing in a range of 12-40% and 33-93% respectively, suggesting that the alkalinity of the calcined clay cement mortar has little impact on the degradation of the rGFRP.

5. Declaration of interest

The author (s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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ORCID iD

https://orcid.org/0000-0003-0323-9634

Data availability statement

Data reported in this paper are available upon request to the corresponding author.

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