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Influence of Alkali-resistant Glass Fibers Distribution on Properties of Cementitious Composites

Branka Mrduljaš[1], Alenka Mauko Pranjić[2], Mateja Štefančić[2], Katarina Didulica[1] and Ana Baričević[1]

[1] Department of Materials, Faculty of Civil Engineering University of Zagreb, Republic of Croatia, bmrduljas@grad.hr (Branka Mrduljaš), kdidulica@grad.hr (Katarina Didulica), abaricevic@grad.hr (Ana Baričević)

[2] Slovenian National Building and Civil Engineering Institute - ZAG, The Department of Materials, Slovenia, alenka.mauko@zag.si (Alenka Mauko Pranjić), mateja.stefancic@zag.si (Mateja Štefančić)

Abstract *The waste hierarchy establishes a prioritized framework for effective waste management, where higher levels such as prevention, re-use, and recovery are associated with the conservation of primary resources and the retrieval of secondary resources. This approach aligns with the objectives of the construction industry, which aims to promote the efficient utilization of resources by treating raw materials in an environmentally responsible manner. In this context, utilizing waste fibers to reinforce cementitious composites becomes more logical than producing new fibers with similar properties. These waste fibers typically originate from the production of high-performance technical textiles used for structural repair and rehabilitation. By reusing these waste materials, the construction industry contributes a circular economy in its own sector and fosters cross-sectoral industrial symbiosis. Although the potential benefits and positive environmental impact of utilizing such materials are recognized, their influence on the properties of composites requires further investigation. This study examines the impact of production waste glass fibers on the properties of the mortar in both the fresh and hardened states. The study focusses on properties such as compressive strength and toughness. To gain a better understanding of the fibers' contribution to the matrix properties, an investigation was conducted using µCT. The study focuses on investigating the effects of waste fibers with lengths of 5 and 10 mm and different dosages (0.2, 0.6 vol.%) on the properties of high-strength mortar, comparing them with factory fibers.*

Keywords: *Waste fibers, X-ray microcomputed tomography (X-ray µCT), fibre distribution.*

1 Introduction

The construction industry is increasingly seeking opportunities for incorporating "green" materials. Synthetic fibers, also known as man-made fibers, serve specific purpose and require the utilization of raw materials and energy. Additionally, they pose challenges as non-biodegradable waste and difficult to recycle. Simultaneously, the production of high-performance technical textiles used to reinforce cement or polymer matrices generates substantial waste, commonly referred to as clean wastes. Although some degradation may occur during the production process and subsequent cutting, most fibre properties are retained. However, the full potential of the fibers in the cement matrix can only be achieved through their uniform dispersion. The extent of fiber dispersion significantly impacts the functional properties of fiber-reinforced composites. According to Chung (Chung 2005), the difficulty of fiber dispersion increases as fiber diameter decreases. Similarly, shorter fiber lengths facilitate fiber dispersion. Fiber volume, distribution, and orientation affect the microstructure of FRM, which influences the properties in the hardened state. Micro computed tomography (µCT) is a non-destructive technique that provides insight into the microstructure of the sample. It is commonly used to asses steel fiber distribution due to the excellent contrast between steel, cement matrix, and voids. However, when it comes to synthetic fibers, the use of µCT to quantify the number of fibers and their spatial distribution is more complex. Synthetic fibers have a density similar as the cement matrix or air voids, and they lack magnetic or electrical properties (Chung et al. 2019). Additionally, the shape and dimensions of synthetic fibers pose a challenge in µCT analysis since they tend to bend during casting, making them difficult to identify accurately during data analysis (Bordelon and Roesler 2014). As reported by Hong et al. (Hong et al. 2021), synthetic fibers can be observed using scanning µCT, but analysing them with a grayscale thresholding method, without the use of deep learning technology, is not possible.

This paper examines the impact of production waste glass fibers on the properties of the mortar in both the fresh and hardened states. The study focusses on properties such as compressive strength and toughness. To gain a better understanding of the fibers' contribution to the matrix properties, an investigation was conducted using µCT, and the results were analysed using Avizo ThermoFischer Scientific software.

2 Materials and Methods

The alkali-resistant glass fibers (wGF) used in this study are waste derived from the manufacturing of high-quality textiles for the construction industry. These fibers were obtained locally by Kelteks Ltd. and were initially supplied as roving for the production of meshes, producing waste in the form shown in the Figure 1. Subsequently, they were cut into lengths of 5 and 10 mm, with an average diameter of 20.4 ± 2.4 µm (Figure 2 b & 2c). For comparison, factory-produced alkali-resistant glass fiber (fGF) from Schwarzwaelder Textil-Werke were used as a reference, with lengths of 6 and 12 mm and a diameter of 14 mm (Figure 2d & 2e).

Figure 1 As received alkali resistant waste fibers

Fiber-reinforced mortar (FRM) was prepared by combining cement (CEM I 52.5 N), aggregates (including limestone filler 0.005/0.125 mm, quartz 0/1.0 mm, dolomite 0.1/0.6 mm, 0.6/1.25 mm, and 1.25/2.0 mm), water, and polycarboxylic ether polymer superplasticizer. The mass ratio of cement to water to aggregate was 1: 0.39: 1.64.

A total of five mixtures were tested, including: a) the reference mixture, b) two mixtures with 0.2% and 0.6% wGF, c) two mixtures with 0.2% and 0.6% fGF. The mixing process began with dry mixing. The fibers were dispersed using a professional shaker, subjected to high-frequency vibrations for up to 5 minutes. The total mixing time was 4.5 minutes, starting with the addition of water and superplasticizer to the mixer, followed by the inclusion of the dry mixture. The resulting wet mixture was then poured into prismatic molds (40 x 40 x 160 mm) and stored under laboratory conditions for 24 hours before demolding. After demolding, the specimens were stored in a chamber at 20 ± 2 °C and relative humidity at least 95%, where they were stored until testing at 28 days of age.

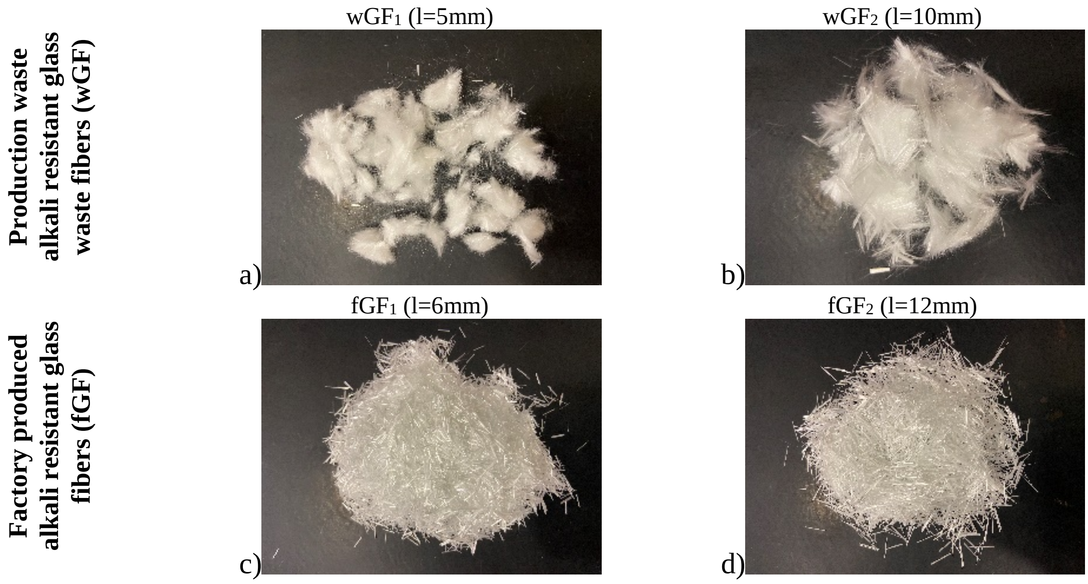

Figure 2 Alkali resistant fibre: a) & b) wGF, l = 5mm & l =10 mm, c) & d) fGF, l =6 mm & l =12 mm.

Mortar properties were assessed in both the fresh (density - EN 12350-6:2009, flow table test - EN 12350-5:2009) and hardened state (compressive strength –EN 1015-11:2019, modulus of elasticity - EN 12390-13:2021, toughness). To measure toughness, specimens were supported on steel rollers with a diameter of 10 mm and a span of 100 mm. The Zwick press, with a capacity of 600 kN, was used for the test. The load was applied at a rate of 0.05 mm/min until a deflection of 0.2 mm was reached, followed by a rate of 0.1 mm/min until the completion of the test. This test speed was selected to obtain a stable portion of the curve after reaching the peak force and to generate a localized crack in the middle of the span. Each hardened state property was tested on a minimum of three specimens.

The samples used for µCT were cylindrical in shape with a diameter of 8 mm. This shape was specifically selected because it allows the sample to be fully projected onto the detector from all angles, resulting in more accurate X-ray projection in the tomographic images (Brisard et al. 2020). At the age of 28 days, the hydration process was stopped by immersing the specimens in isopropanol solvent. They were submerged in the solvent for seven days and then subjected to vacuum conditions for an additional seven days. Following preparation, the specimens were stored in a nitrogen chamber at a temperature of 20 ± 2 °C and a relative humidity of 35% to prevent potential carbonation.

Tomography imaging was conducted using microXCT-400 (Xradia, Zeiss) at the Slovenian National Building and Civil Engineering Institute. The cylindrical specimen was affixed to a turntable and rotated from 0° to 360° with a step angle of 0.199°. The X-ray source was configured with a voltage of 80 kV and a power of 10 W. An exposure time of 10 seconds was employed. Data was obtained with a pixel size of 7.95 µm, and the average scan time per sample was approximately 6 hours. The optimal scan parameters were determined through a trial and error process. Two-dimensional (2D) 16-bit grayscale images were generated for each scanned sample, enabling subsequent image analysis.

3 Results and Discussion

The results of the FRM properties tested are shown in Table 3.

Table 1 Results of testing properties of mortar reinforced with GF fibers in fresh and hardened state.

Property	REF	fGF_1	wGF_1	fGF_2	wGF_2
Fresh state properties					
Density (g/dm^3)	2199	2209	2137	2189	2025
Consistency (mm)	185/190	198/200	186/186	185/185	188/188
Superplasticizer (%m_c)	0.40	0.42	0.46	0.42	0.62
Hardened state properties					
Compressive strength (MPa)	58.37±2.40	71.16±1.34	64.37±1.58	76.61±1.96	65.95±2.19
Spec. energy absorption capacity (N/m)	213.90±92.09	207.87±13.64	243.23±29.30	504.87±33.31	158.07±8.80

The amount of superplasticizer was adjusted in the fibre-reinforced mortars compared to the reference mix to achieve the same workability (Table 1). To attain the desired workability, an increase of 5% in superplasticizer content was necessary for fGF_1 and fGF_2, whereas an 15% increase was required for wGF_1 and 55% increase for wGF_2. Glass fibers, being rigid and non-ductile, impede the flow of the mixture, resulting in reduced consistency (Ahmad et al. 2022). However, all the mixes fall within the same consistency class, namely plastic consistency, with values ranging from 140 to 200 mm.

Further tests were performed on hardened samples treated and prepared as previously described. The age of 28 days was chosen because no major changes in the structure are expected after. By employing an image segmentation technique based on thresholding, it is possible to effectively filter out air voids from µCT images. Parameters such as average surface area, equivalent diameter, volume, and number of pores were then extracted from the designated region of interest (ROI). The findings from the µCT analysis validate that dosage and length of the fibers have an impact on the distribution and quantity of air voids within the FRM. The results clearly demonstrated a significant increase in the number of air voids with the addition of both types of fibers. However, there is a decrease in the surface area and volume of the pores, indicating that the fibers contribute to the intersection of the air void system (Table 2). Obtained results are in accordance with previously published paper (Chen et al. 2021).

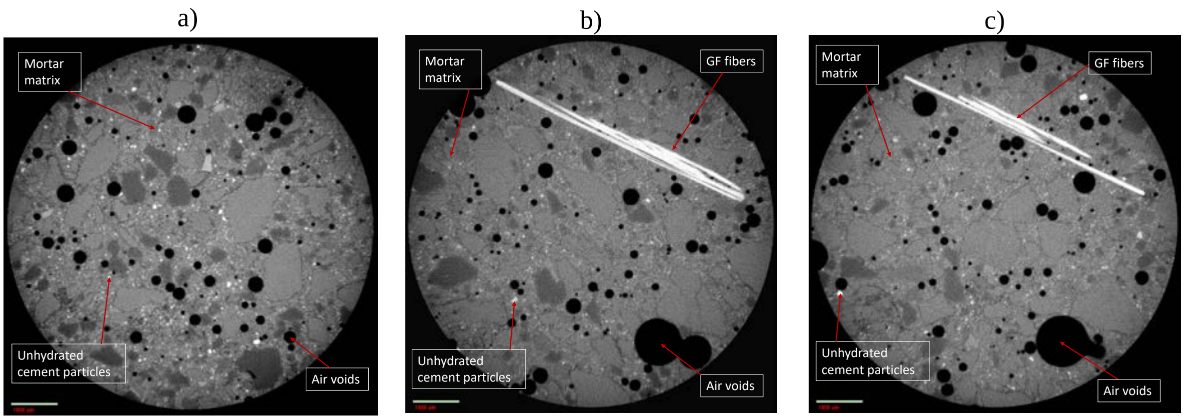

Figure 3 µCt images of FRM reinforced with a) reference mixture (REF); b) waste (wGF_2) and b) factory (fGF_2) glass fibers.

Table 2 µCT characterization of pore structure in FRM

Mixture ID	*Fiber dosage (% V_f)*	*Average surface area (µm²)*	*Equivalent diameter (µm)*	*Average Volume (µm³)*	*Number*
Ref	0	68871.98	95.04	4278761.00	1778
fGF_1	0.2	37983.92	79.67	1454795.80	2674
wGF_1		46542.84	87.85	1811450.60	4921
fGF_2	0.6	41414.22	87.39	1326117.50	3200
wGF_2		46557.35	87.46	1731395.30	3201

Simultaneously, the fibers contributed to an increase in compressive strength. An increase of 22% and 31% was observed for fGF, while an average increase of 10% was observed for wGF. This enhancement can be attributed to interfacial transition bond between fGF and the cement matrix (Yuan and Jia 2021). It is worth mentioning that the majority of the literature reports a decrease in compressive strength when glass fibers are added (Brazão Farinha et al. 2021, Fenu et al. 2016), whereas a smaller number of publications corroborate the findings of this research (Blazy et al. 2022). The optimal amount of fibers plays a crucial role in optimizing the internal pore structure and increasing the density of the concrete microstructure. The presence of uniformly dispersed fibers forms a dense network structure, thereby enhancing the distribution of internal stress and the mechanical properties of concrete (Jiao et al. 2022). Also, as it was showed in the work of Salem et al., a higher amount of SP contributed to an increase in strength after 28 days (Salem et al. 2016).

Glass fibers are commonly incorporated into cement composites to enhance either volume deformation or toughness (Blazy et al. 2022). In this study, the focus was on examining the mechanical properties, and thus, the force-deflection curves are presented in Figure 4. These curves, along with the specific energy absorption capacity indicated in Table 1, clearly validate the substantial impact of the 12 mm fGF. A remarkable increase of 136% compared to the reference mixtures is observed, which is consistent with previously published papers (Yuan and Jia 2021). Conversely, no significant contributions were observed from other fibre reinforced mixture in the post-crack region.

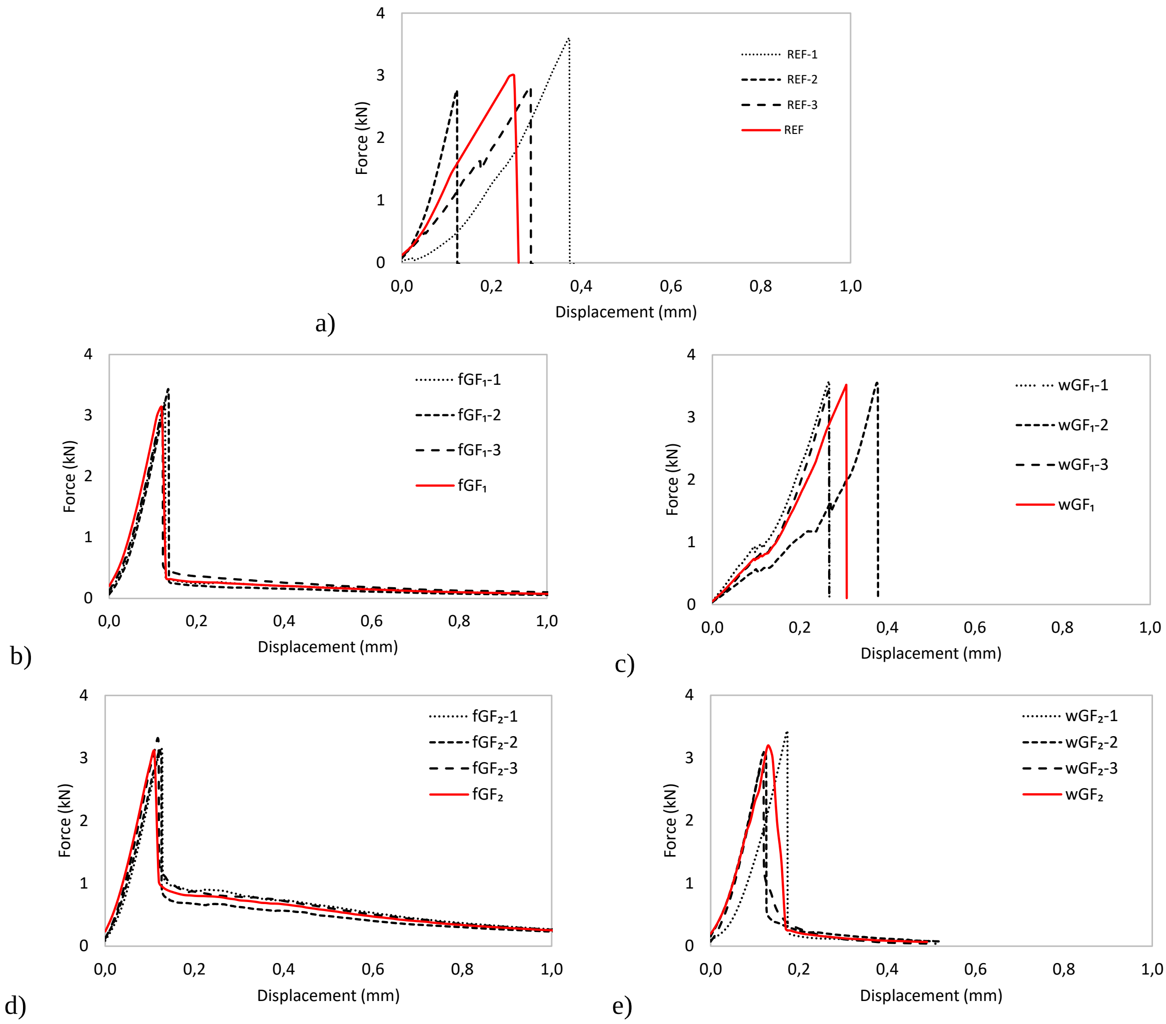

Figure 4 Load-deflection curves of FRM: a) reference mixture (REF); b) mixture with 0.2%V of fGF_1; c) mixture with 0.2%V of wGF_1; d) mixture with 0.6%V fGF_2; e) mixture with 0.6%V wGF_2.

As mentioned earlier, achieving the full potential of the fibers in the cement matrix necessitates their dispersion within the matrix (D. D. Chung, 2005). However, due to the similar density values of glass fibers and non-hydrated cement particles, distinguishing between them solely based on their grey level threshold becomes challenging, thereby posing difficulties in 3D reconstruction and segmentation (Saha et al., 2020). In order to elucidate the findings of the toughness test, µCT analysis was employed. Specifically, it was anticipated that fGF_2 and wGF_2 would exhibit similar post-cracking behaviour considering their fibre length and content in the mixture. However, the µCT analysis revealed that the wGF were not adequately distributed across the cross-section. Despite having the same fibre quantity in the mixture, the visualisation in Figure 5 demonstrates that the fGF fibers were more effectively distributed within the selected cross section. In contrast, the wGF remained clustered together, resulting in a lower fibre count within the cross-section. All fibre mixtures exhibited the same fiber, namely perpendicular to the loading direction. The observed disparity in fiber dispersion clearly elucidates the underlying reasons for the non-uniform post-cracking behaviour in fibre-reinforced mortars. These findings indicate the necessity for further enhancements in the dispersion of wGF to fully harness their potential.

a) b)

Figure 5. 3D visualization of FRM with a) fGF2, b) wGF2

4 Conclusion

The distribution of fibers in cementitious composites significantly affects the properties of the composite in both the fresh and hardened states. The following conclusion have been drawn:

- The inclusion of wGF leads to a decrease in the density of the mixtures due to the higher entrapped air during mixing compared to the mixtures with fGF. However, the mortar reinforced with both wGF and fGF exhibit pores with smaller surface area and pore diameter compared to the reference mix. This analysis clearly demonstrates that the addition of fibers has a strong influence on the pore structure.
- Both wGF and fGF show a trend towards increasing compressive strength, but effect is more pronounced for factory fibers.
- Only fGF with a length of 12 mm contribute significantly to the toughness of the composites.
- Detecting the distribution of synthetic fibers using µCT is challenging without the utilization of deep learning technology to automatic segmentation of glass fibers in reinforced mortar.

Based on the preliminary testing, it can be concluded that waste fibers hold promise as a valuable resource in the construction industry.

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ORCID:

Branka Mrduljaš: https://orcid.org/0000-0002-6550-6091
Alenka Mauko Pranjić: https://orcid.org/0000-0002-9822-7806
Mateja Štefančić: https://orcid.org/0000-0002-7031-9426
Katarina Didulica: https://orcid.org/0000-0003-1115-055X

Ana Baričević: https://orcid.org/0000-0002-5779-9939

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