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Electron Microscopy Observations on Glass Fiber Reinforced Concrete (GFRC) Materials

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Abstract

Doping concrete structure with glass fibers gives rise to superior mechanical and chemical properties in macro-scale constructional applications. This type of materials is also currently called a generic name: Glass Fiber Reinforced Concrete (GFRC). Despite the fact that numerous studies have been conducted to shed light on the structure-property relationship in GFRC materials, focusing on the microscopic features to gain a better understanding of fibers role in the concrete matrix is still a challenging task. The goal of present research is, therefore, to reveal the micro-scale behavior of commercially available glass fibers in resulting concrete structures through a scanning electron microscopy (SEM) technique. For this purpose, the fracture surfaces of GFRC samples were characterized using a high-resolution field emission scanning electron microscope (FESEM, Carl Zeiss Supra 50VP) equipped with an energy dispersive X-ray spectrometer (EDXS, Oxford Instruments 7430), operating at 30 kV in variable pressure (VP) mode. The back scattered electron (BSE) showed the general microstructure of GFRC material. As a result of this, the random distribution of Zr-rich glass fibers in mainly calcium-based silicate concrete matrix phases was clearly disclosed, which can be also confirmed by the EDX analysis. In addition, loose structure between the glass fiber and matrix phases was also observed. In fact, this unexpected micro-structural evolution can be considered as an apparent evidence of hydrophobic tendency of very thin polymer coating existing on the fibers' surface determined with non-microscopic techniques, e.g., simultaneous differential thermal analysis (DTA)/thermal gravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR).

Key words
GFRC;
Investigation;
Structure;
SEM

Cam Fiber Katkılı Beton (GFRC) Malzemeleri Üzerine Elektron Mikroskopi Gözlemleri

Özet

Cam fiberler ile beton yapısının katkılanması makro ölçekteki yapısal uygulamalar için üstün mekanik ve kimyasal özelliklerin elde edilmesini sağlamaktadır. Bu tür malzemeler aynı zamanda günümüzde "Cam Fiber Katkılı Beton (CFKB)" genel ismiyle adlandırılmaktadırlar. CFKB malzemelerinde yapı-özellik ilişkilerini aydınlatmak üzerine birçok çalışma gerçekleştirilmiş olmasına rağmen, beton matrisi içerisinde fiberlerin rolünü daha iyi anlamak için mikroskobik özellikler üzerine odaklanma günümüzde halen zor bir hedeftir. Dolayısıyla, bu çalışmanın amacı bir taramalı elektron mikroskobu tekniği yardımıyla, elde edilen beton yapıları içerisinde ticari cam fiberin mikro ölçekteki davranışını açığa çıkartmaktır. Bu amaç için CFKB numunelerinin kırık yüzeyleri, değişken basınç (VP) yöntemiyle 30 kV'da çalıştırılan bir enerji saçınımlı X-ışını spektrometresi (EDXS, Oxford Instruments 7430) içeren yüksek-ayırma güçlü alan yayınım taramalı elektron mikroskobu (FESEM, Carl Zeiss Supra 50VP) kullanılarak karakterize edilmişlerdir. Geri yansıyan elektron (BSE) CFKB malzemesinin genel mikro-yapısını göstermiştir. Sonuçta, başlıca kalsiyum-esaslı silikat beton matris fazları içerisinde Zr'ca zengin cam fiberlerin düzensiz dağılımı açıkça tespit edilmiş ve durum EDX analiziyle de doğrulanmıştır. İlave olarak, cam fiber ve matris fazlar arasındaki zayıf bağlanma gözlemlenmiştir. Aslında, bu beklenmedik mikro-yapısal gelişim mikroskobik olmayan teknikler, örneğin; eşzamanlı diferansiyel ısısal analiz (DTA)/ısısal gravimetrik analiz (TGA) ve Fourier dönüşüm kızılötesi spektroskopisi (FTIR) ile fiber yüzeylerinde belirlenen mevcut çok ince polimer kaplamasının su sevmezlik (hidrofobik) eğiliminin görünür bir delili olarak değerlendirilebilmektedir.

Anahtar kelimeler
CFKB;
İnceleme;
Yapı;
SEM

1. Introduction

Glass fiber reinforced concrete (Glass Fiber Reinforced Concrete-GFRC) or glass fiber reinforced composite (Glass Fiber Reinforced Composite), calling with more general name, is composed of high strength glass-fibers that embedded in a cement-based matrix. In such a composite structure, the physical and chemical properties of both fiber and matrix remain unchanged. Additionally, the most important advantage of resulting composite is obtaining a considerably more improved structure with respect to both glass and concrete's properties. The fibers are generally defined as the load-carrying elements, while the matrix structure existing in their surroundings is known as load-conducting environment that also makes a stable of fibers' motions inside it and further protects the fibers from environmental effects. Moreover, the fibers enable a more flexible property to concretes without any cracking in the case of coated with a material that usually has polymeric content [1].

The first of glass used with the aim of fiber production is specified as soda-lime or A-glass. These types of glasses are not durable against alkaline solutions. Therefore, E-glasses are designed instead. The E-glasses generally contain a low amount of alkaline (< 2 %) and are well-known as borosilicate glass [2]. The most of fiber glass production in the world still comprise of E-glasses.

In present research, the detailed micro-structural observations on the glass-fiber reinforced concrete (GFRC) structures were employed with SEM and energy dispersive X-ray spectroscopy (EDXS) combination. Here, considered as a first novelty on the study, a commercial glass fiber, known as E-glass, was incorporated into concrete structure. Following, as a second vital point covering the research, an additive called as ADVA Flow 501 in the market was also employed to give a considerable amount of fluidity and durability for concrete structure. Thus, in the case of using this additive during the concrete production process, the required water content can be remarkably decreased. ADVA Flow 501 is a modified synthetic-based polymer carboxylate and is produced under controlled conditions to obtain a stable product. By this unique property, high workability concretes with enhanced mechanical properties can be designed by joining of ADVA Flow 501 and E-glass within concrete structures [3].

This research, therefore, shortly aims to clarify the structure-property relationship in resulting GFRC advanced-materials produced herein.

2. Experimental Procedure

A commercial E-glass fiber was used as a reinforcing material to produce GFRC structures. The detailed chemical and morphological characterization of E-glass fibers was performed by using SEM-EDX analysis.

To obtain high performance GFRC advanced-structures, the compositional designing studies were carried out based on the combined use of chemical (CA) and commercial glass fiber (CGF) additives. Here, the ADVA Flow 501 was chosen as CA. The composite concrete samples were fabricated by separately doping with CA and CGF in the compositions of 450 g cement + 1350 g SiO_2 sand + 6.75 g ADVA Flow 501 (1.5 wt. %of cement) and 0.3, 0.5, 0.8, 1 and 2 wt. % of total weight, respectively. As a preference, CA was simultaneously added to concrete structure with water. Direct doping of CA in cement leads to a variety of undesired problems. Please also note that we do not use any CA in the system different than ADVA Flow 501. Moreover, un-mixing of CA with the other components of concrete during batch preparation protects the chemical stability of CA and further improves its function in the mixture.

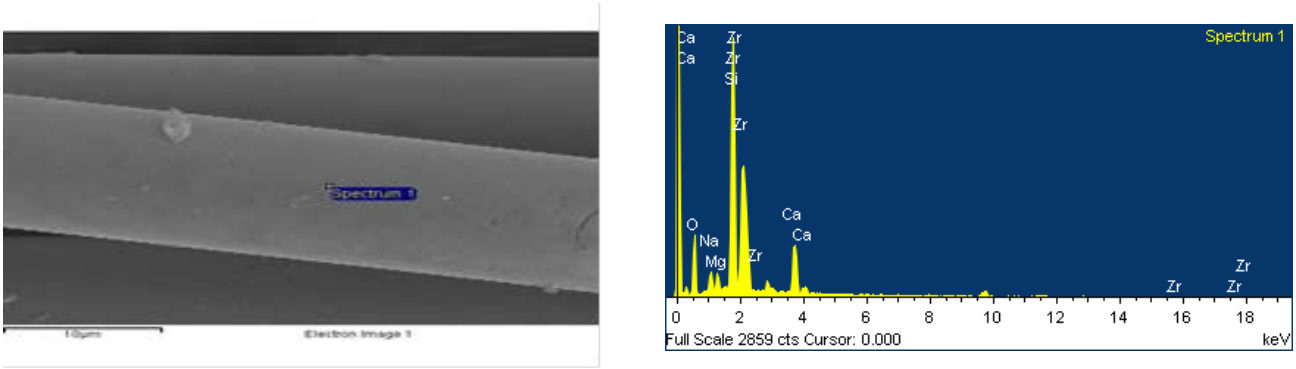
The micro-structural examinations of GFRC samples produced with addition of CFG, which includes a polymeric-based coating material, silane, on the surface as well as its removed form through applying a heat treatment process and combination of CA to CFG were performed by using high-resolution field emission gun (FEG) SEM (Carl Zeiss Supra 50VP) attached with EDX (Oxford Instruments 7430) spectrometer operated at 30 kV in variable pressure (VP) mode in Anadolu

University, Department of Materials Science and Engineering, Electron Microscopy Laboratories.

Three point bending test and compressive experiments of the concrete structures with CFG doping in different weight percentages and without adding any CFG at the end of 2, 7 and 28 days cure were carried out in Afyon Kocatepe University, Department of Civil Engineering Laboratories on the basis of TS EN 196-1 standards.

3. Results and Discussion

The SEM-EDX analysis results performed with the aim of determination glass fibers' chemical composition were given in Figs. 1 (a-b) and Table 1.



(a) **(b)**

Figure 1. (a) The secondary electron (SE) SEM image indicating a place where EDX point analysis on glass fiber was acquired from, (b) The EDX spectrum data coming from marked region in Fig. 1 (a).

Based upon the SEM-EDX findings presented in Figs. 1 (a-b), it was determined that the chemical composition of commercial glass fiber consisted of O, Na, Mg, Ca, Si, and Zr elements. According to the EDX analysis data in Fig. 1 (b), the quantitative results of related elements are presented in Table 1.

Table 1. The percentage of concentration values driven from EDX analysis in Figs. 1 (a-b)

Element-X-ray Line	Weight %	Atomic %	Compound %	Formula
Na-K	2.73	3.12	3.68	Na_2O
Mg-K	1.82	1.96	3.01	MgO
Si-K	19.79	18.50	42.34	SiO_2
Ca-K	8.07	5.28	11.29	CaO
Zr-L	29.37	8.45	39.68	ZrO_2
O-K	38.22	62.69	-	-
Total	100.00	100.00	100.00	-

With Table 1, it is deduced that the commercial glass fiber is based on silica (SiO_2). Also, it is thought that the main purpose of zirconia (ZrO_2)'s usage herein is to increase the chemical resistance of glass fiber. Additionally, it is estimated that the presence of alkali (Na_2O) and earth alkali oxides (MgO+CaO) is for making the fiber draw easier by means of reducing the viscosity and working temperature of glass. The commercial glass fibers, as a reinforced-material, coated with a polymeric component (silane) on their surface were incorporated into the concrete structure in proportions of 0.5, 0.8 and 1 wt. %. As a result, the GFRC materials were successfully fabricated. The three point bending test and compressive experiments on the concrete structures with CFG doping and without adding any CFG, namely as-received samples at the end of 2, 7 and 28 days cure were comparatively presented in Table 2.

Table 2. The three point bending and compressive test results obtained from the GFRC samples produced with the commercial glass fibers addition in the proportion of 0.5, 0.8 and 1 wt. %

Fiber Additive	2 Days		7 Days		28 Days	
	Three Point Bending Test	Compressive Test	Three Point Bending Test	Compressive Test	Three Point Bending Test	Compressive Test
Standard	4.76	20.25	6.0	32.3	7	43
0.5 %	4.4	18.9	5.7	28.1	6.8	36
0.8 %	4.5	22.95	5.3	30.1	6.4	30.95
1 %	4.3	21.5	5.9	22.1	5.4	22.35

Based on the data of Table 2, as a result of glass fibers' incorporation into the concrete structures, it is clearly observed that the mechanical properties of GFRC samples were negatively affected when compared to those of as-received sample. Therefore, it was thought that the reasons of this negativity could be related to the polymeric coating material's properties on the fiber surface, dispersion and bonding characteristics with matrix phases of commercial glass fibers within the concrete structure. At this point, for the sake of the clarity why the mechanical properties were affected in a negative way in the case of fiber doping into the concrete structure, the micro-structural and analytical observations taken from GFRC sample resulting with the addition of 0.5 wt. % commercial glass fiber after 7 days cure were conducted with SEM-EDX combination under VP conditions without any coating and the obtained examinations' results were presented in Figs. 2 (a-d).

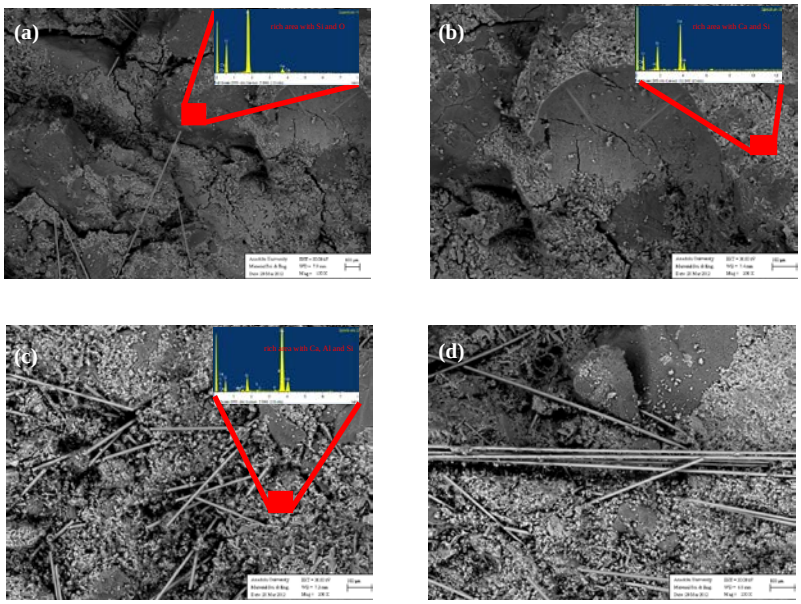


Figure 2. (a) The SEM image acquired from GFRC sample containing 0.5 wt. % commercial glass fiber addition. Please note that the insert EDX spectrum taken from a region marked with red lines shows the quartz-based aggregate, (b-c) The other SEM images obtained from the different regions of GFRC sample containing 0.5 wt. % commercial glass fiber addition. Please note that the insert EDX spectra taken from regions labeled with red lines reveals the calcium silicate- and calcium aluminum silicate-based matrix phases, respectively, (d) The SEM image from GFRC sample containing 0.5 wt. % commercial glass fiber addition indicating the positions of fibers within the matrix.

Considering the SEM-EDX imaging and chemical analysis results given in Figs. 2 (a-d) together, it was clearly observed that the polymer-coated commercial glass fibers used as reinforcement material were not homogenously dispersed in matrix and aggregate structures of GFRC. Furthermore, it was determined that the fibers did not properly contact with the matrix and aggregate components. Therefore, this result implies that the polymeric–based coating material existing on the

fiber's surface might be in hydrophobic character. As a result of this fiber nature, it can be said that glass fibers do not entirely hold on the matrix and aggregate components.

In the light of such results, the silane coating on the fibers' surface was cleaned by means of a chemical reaction occurring between acetone and fiber. Afterwards, different GFRC samples were produced by using the addition of 0.5, 0.7, and 0.8 wt. % uncoated (no silane on the fibers' surface) short fibers and 0.8 wt. % long ones. In Figs. 3 (a-d), the SEM images of GFRC samples that reinforced with uncoated short and long glass fibers in different proportions (maximum 0.8 wt. %) were given.

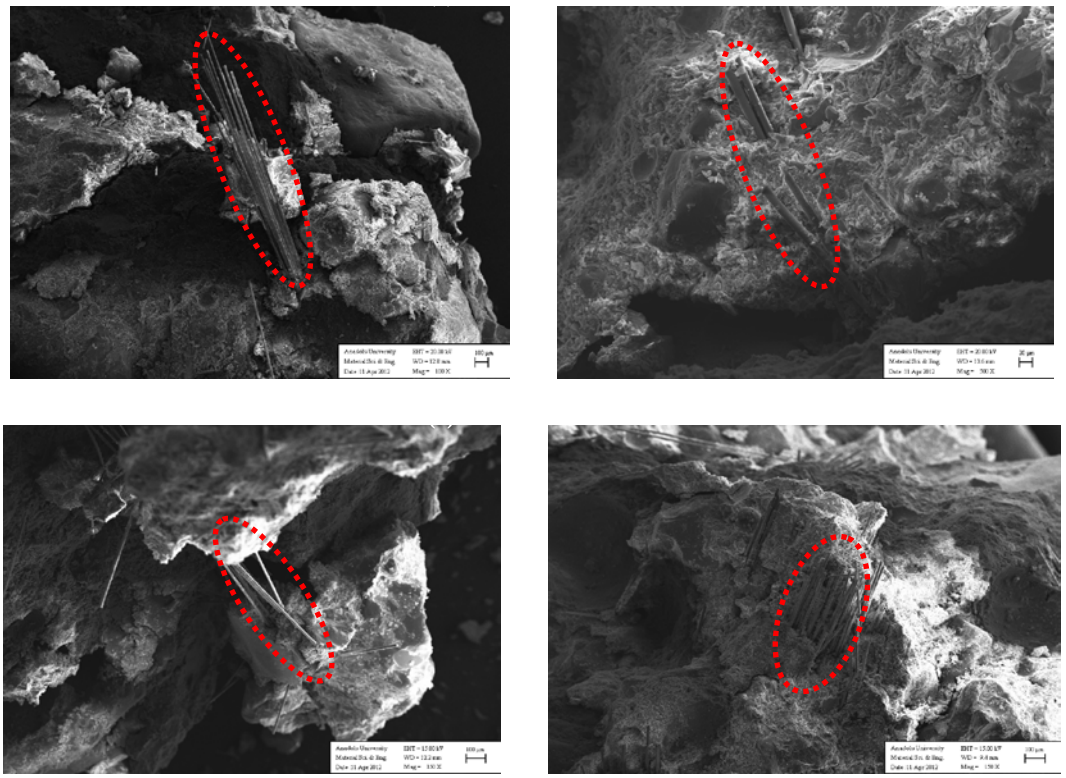


Figure 3. (a-b) The SEM images of GFRC samples with the addition of uncoated short commercial glass fibers in 0.5 and 0.7 wt. %, respectively, (c-d) The SEM images of GFRC samples with the addition of uncoated short and long commercial glass fibers in 0.8 wt. %

Considering the findings observed in Figs. 3 (a-d), it was seen that the commercial glass fibers, surfaces of which were treated with acetone were not homogenously distributed in GFRC matrix structure. Furthermore, the agglomerated groups can be clearly discerned in the special regions that labeled with dashed red lines. The acquired results clearly reveal that not only controlling the fibers' surface characteristics is sufficient, but also additional chemical doping agents are required to homogenously disperse the commercial glass fibers and to prevent the clusters' formation in concrete matrix structure during the GFRC process. Therefore, it was decided to use a chemical additive to obtain the homogeneity in the distribution of glass fibers within GFRC.

Here, new GFRC samples produced by mixing of ADVA Flow 501, used as a CA, and commercial glass fibers in the proportions of 0.3, 0.5, 0.8 and 1 wt. % are given. Thus, the values of three point bending and compressive tests of resulting new GFRC samples taking from the end of the 2, 7 and 28 days cure are comparatively shown in Table 3.

Table 3. The three point bending and compressive tests' results of GFRC samples consisted of no including chemical additive (CA) and commercial glass fiber (CGF), called as standard concrete samples, as well as those containing CA and CFG in proportions to 0.3, 0.5, 0.8, 1 and 2 wt. % together

Amounts (% Weight)	2 Days		7 Days		28 Days	
	Three Point Bending Test	Compressive Test	Three Point Bending Test	Compressive Test	Three Point Bending Test	Compressive Test
Standard*	4.76	20.25	6.0	32.3	7	43
Standard**	5.2	33.3	9.6	56.6	8.0	57.75
0.3 % (CA+CFG)	4.4	25.1	6.9	41.4	7.2	47.3
0.5 % (CA+CFG)	5.50	27.25	7.2	42.8	7.2	52.5
0.8 % (CA+CFG)	2.8	13.35	4.5	24.9	5.7	28.4
1 % (CA+CFG)	3.9	17.5	5.7	27.3	5.9	29.8
2 % (CA+CFG)	-	-	5.1	43.2	-	-

* CA and CFG (Only concrete and no doping) do not exist.
**CA exists, but not CFG (Only chemical doping).

According to Table 3, the significant data for the improvement of mechanical properties by means of glass fiber incorporation and chemical additive usage within the GFRC structures were obtained from the recipe with CA+CFG as 0.5 wt. %. Here, it can be observed that three point bending and compressive strengths of resulting GFRC advanced-materials at the end of 2, 7 and 28 days cure considerably increase when compared to both standard samples shown in Fig. 3. However, in the cases of the recipe with CA+CFG over 0.5 wt. %, it was observed that the strength values reduced due to the possible formation of voids in the structure.

The micro-structural evolutions of GFRC samples acquired from the recipe with CA+CFG as 0.5 wt. % at the end of 7 and 28 days cure were presented in the Figs. 4 (a-b) and 5 (a-b).

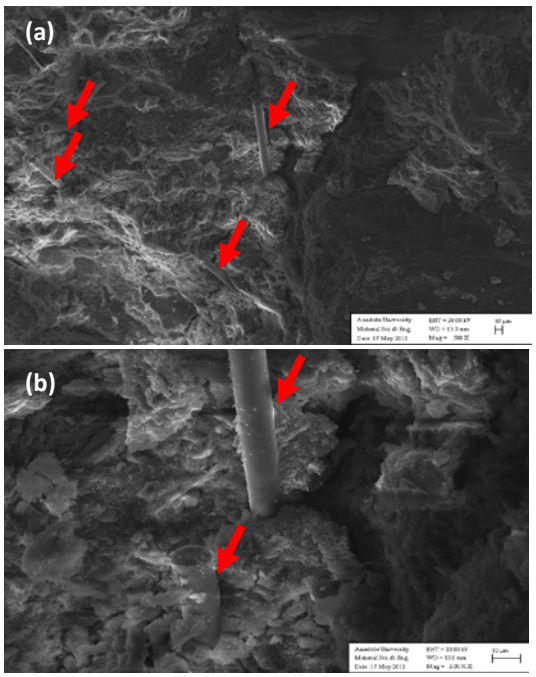


Figure 4. (a-b) The SEM images in different magnification taken from the GFRC sample with CA+CFG as 0.5 wt. at the end of 7 days cure.

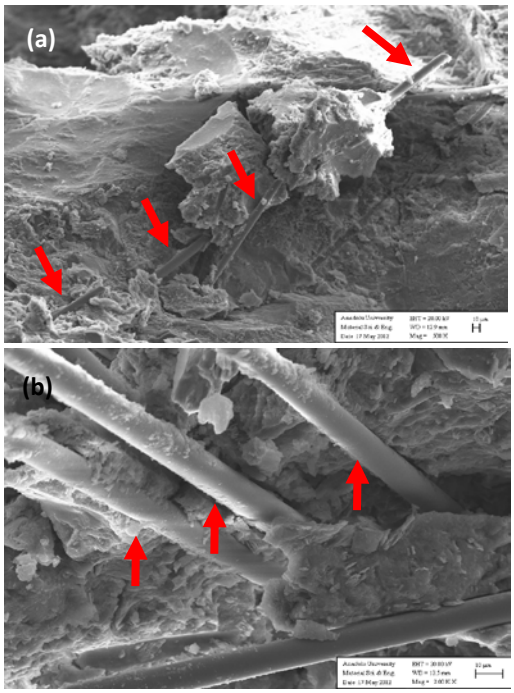


Figure 5. (a-b) The SEM images in different values taken from the GFRC sample with CA+CFG as 0.5 wt. % at the end of 28 days cure.

Looking at the Figs. 4 (a-b) and 5 (a-b), it could easily be noticed that the commercial glass fibers were homogeneously dispersed in GFRC structure wherein especially special regions marked with red arrows. Additionally, no cluster-type formation was detected in GFRC material. Furthermore, it was concluded that glass fibers showed good bonding behavior with matrix phases, which also means that they play a role as load-carrying constituents in GFRC matrix. Moreover, based on the evolved micro-structural designs in Figs. 4 (a-b) and 5 (a-b), it can be explained that the addition of ADVA Flow 501 makes the homogenous distribution of glass fibers in GFRC structures easier and hence leads to the enhancement of the mechanical properties of resulting composites.

4. Conclusion

In the present study, the commercial glass fibers' incorporation into concrete structures for making a new type of advanced composite material generally called as glass fiber reinforced concrete/composite (GFRC) is reported. Firstly, the effects of polymeric-based coating material-silane-were investigated on the resulting GFRC structure. Here, using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) spectroscopy facilities, the cluster-type formation of glass fibers in GFRC matrix further revealed the inhomogeneous distributions of fibers in GFRC was clearly observed. In the light of such an observation, secondly, the surfaces of commercial glass fibers were heat-treated to remove the polymer coating. However, even if heat-treated commercial fibers were added into GFRC structure, the formation of fiber clusters was not unfortunately prevented. Therefore, the mechanical properties, i.e., three point bending and compressive strengths were deteriorated in both coated and heat-treated commercial fibers doped GFRC samples with respect to the standard concrete. To overcome this undesired result, an extra chemical additive, ADVA Flow 501, during the GFRC's production step was used. Finally, by the combination of chemical (CA) and commercial glass fibers (CFG) in the proportion of 0.5 wt. % CA+CFG, the obtained samples' mechanical properties after 2, 7, and 28 days cure were considerably increased when compared to those of standard samples. Considering the micro-structural evolutions of GFRC composites with CA+CFG as 0.5 wt. %, it was seen that the commercial glass fibers were well-dispersed in the GFRC matrixes, further explaining that why such enhanced mechanical properties were obtained.

Consequently, the study on the relationship between micro-structure and mechanical properties in GFRC advanced-materials was carried out. It is anticipated that successfully controlling the glass fibers' micro-structural behavior will pave the way for the production of GFRC advanced-composites in constructional applications.

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References

1. www.concretecountertopinstitute.com
2. http://en.wikipedia.org/wiki/Fiberglass
3. www.graceconstruction.com