Evaluation of compressive and split tensile strength of slag based aluminosilicate geopolymer reinforced by waste polymeric materials using Taguchi method

Amirreza Khezrloo, Morteza Tayebi, Abbas Shafiee, and Alireza Aghaie

1 Young Researchers and Elites Club, Science and Research Branch, Islamic Azad University, Tehran, Iran
2 School of Mechanical Engineering School, Sharif University of Technology, Tehran, Iran
3 Department of Civil Engineering, Shams Institute of Higher Education, Gonbad Kavoos, Iran

E-mail: Morteza.tayebi95@yahoo.com, Amirreza.khezrloo@srbiau.ac.ir, Abbasshaafie@alum.sharif.edu and Alirezaaghaie1607@gmail.com

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Abstract

In this work, slag based aluminosilicate geopolymer was reinforced with polymeric fibers including, polyester (PES) (waste tire cap plies), polymeric particles including polyethylene terephthalate (PET) (waste water bottle), styrene-butadiene rubber (SBR) (waste tire), and polyvinyl chloride (PVC) (waste water hose). The tensile and compressive strength of the material was evaluated. Taguchi method was employed to assess the influence of the effective parameters on the mechanical characteristics of the geopolymer composite. QUALITEK-4 software was used to create the L32 orthogonal array with 192 geopolymer specimens and 32 + 32 experiments. Analysis of variance (ANOVA) was utilized to analyze the results of the experiments. The prepared geopolymers were characterized by SEM analysis. The results revealed that the compressive and split tensile strength of the aluminosilicate geopolymer was substantially improved after reinforced by the PES fibers compared with other particles such as SBR, PVC, and PET.

1. Introduction

Nowadays, one of the basic needs of any society is a waste management system to reduce the negative effects of the waste materials on the environment [1, 2]. Recently, the use of polymeric materials has extensively increased due to their superior properties. For instance, they are used in the automotive industry as a car tire, in the water and fire extinguishing industries as a fire hose, and in the packaging and food industries as bottles of water.

In fact, it is a global concern to prevent the accumulation and release of waste polymeric materials such as PVC, PET, SBR, and PES. One simple and inexpensive method is to melt or burn them; however, it will result in producing toxic greenhouse gases such as carbon dioxide. So, one solution is the utilize polymeric materials in construction and concrete reinforcement [3–5].

Therefore, using polymeric waste for construction not only supports the waste management system but also improves the mechanical properties of the structures [6–10]. The geopolymers that have received considerable attention over the last few years can be made from waste materials. An aluminosilicate source that can be made from waste materials will result in the formation of a low-cost material with great strength and excellent thermal resistance [11–13].

Although there have been many reports on using polymeric fibers/particles in concretes [3–6, 9, 10], the application of these fibers/particles in geopolymers only has been reported in few studies [14–17]. Geopolymers are a group of inorganic polymers that are predominantly amorphous or semi-crystalline at the nanoscale and are usually produced at temperatures below 120 °C through polymerization of aluminosilicate monomers [12, 18]. Based on the ratio of silicon to aluminum, geopolymers are divided into three main categories
including, poly(sialate) – Si–O–Al–O–(Si:Al=1), Poly(sialate-siloxo) – Si–O–Al–O–Si–O– (Si:Al=2) and Poly (sialate-disiloxo) Si–O–Al–O–Si–O–Si–O– (Si:Al=3) [19]. Geopolymers have greater compressive strength and corrosion resistance compared with mineral cement materials [12, 13, 20]. In fact, due to the low cost of the raw materials used for producing the geopolymers, they are considered as cost-effective alternative options for all cement types and certain polymers. Also, as the temperature of the synthesis process for the geopolymers is lower than that of the conventional cement materials and polymers, they produce fewer pollutants such as CO₂ [21, 22]. The aforementioned advantages have increased the number of studies conducted on evaluating the properties of the geopolymers in recent years [12].

Many research studies have attempted to improve the mechanical properties of concrete or geopolymer by addition of waste materials for example Aly et al [23] investigate the effect of different percentages of crumb rubber as a partial substitution of both; fine, and coarse aggregates by volume percentage (0, 10, 20 and 30%) on the hardened properties (compressive, tensile and flexural strength) and impact resistance of slag based geopolymer concrete (replacing the cement by; ground granulated blast furnace slag (GGBFS) activated with sodium silicate and sodium hydroxide). Their work provides the mix with high compressive strength, ductility, and impact resistance to be used in structural elements subjected to impact and dynamic load such as (bridge approach slabs, railway buffers, and airport runways).

Bhutta et al [24] investigate the effects of six types of microfibers (high-strength steel, waste steel wool, polyvinyl alcohol (PVOH), polypropylene (PP), polyester (PES), and carbon) on the physical properties (workability, and total porosity) and flexural behavior of fly ash-based geopolymer mortars. High strength steel fibers imparted the lowest change in workability whereas with PES fibers the lowest flow was observed. PES-reinforced samples showed the highest porosity followed by steel, wool, and PP-reinforced samples.

To conduct a research study an experimental design is required. As the number of the effective parameters on the compressive and tensile strength of geopolymer reinforced by polymeric fiber was a lot, the Taguchi method was utilized for experimental design. By the Taguchi experimental design, the number of necessary experiments can be determined to avoid massive experimental labor, analyze the results, and point out the optimal conditions. The analysis of variance (ANOVA) method was used to evaluate the results [12, 25, 26].

This study aimed at investigating the compressive and split tensile strength of the aluminosilicate geopolymer reinforced by polymeric fibers and particles, and finding the value of the optimum parameters. After determining the effective parameters, 32 + 32 experiments were determined using the Taguchi experimental method. It should be noted that if the Taguchi method was not used, $4^5 = 65536$ experiments would be performed to find out the optimum parameters. The results of the experiments were assessed using the QUALITEK-4 software. The scanning electron microscope (SEM) was employed to investigate the microstructure of the geopolymers.

### 2. Materials and method

#### 2.1. Materials

In this work, the rockwool furnace slag was used as the source for preparing the aluminosilicate geopolymers. The aluminosilicate slags were ground by a ball mill and passed through the 50-mesh sieve. The chemical analysis of the powder was performed using the x-ray fluorescence (XRF) method, and the results are demonstrated in table 1.

| wt.%     | SiO₂ | Al₂O₃ | TiO₂ | Fe₂O₃ + FeO | CaO  | MgO  | Na₂O | H₂O  |
|----------|------|-------|------|-------------|------|------|------|------|
| Aluminosilicate | 46   | 17    | 1.5  | 7.5         | 18   | 10   | —    | —    |
| WG       | 33.11| —     | —    | —           | —    | —    | —    | —    |

Figure 1 illustrates the particles and fibers used in this study. The initial diameter of the reinforcement particles was in the range of 7–12 mm, and the length-to-diameter ratio of the fibers was around 30. To avoid the inconsistency, the waste polymeric particles were shredded and sorted by a 1-mesh sieve, then they were mixed with the raw materials.
2.2. Experimental design
By considering the parameters affecting the mechanical properties of the aluminosilicate geopolymer composites, eight factors at four levels were defined (table 2). According to the Taguchi method, the L32 orthogonal array was obtained, which means 32 experiments need to be conducted. As seen in table 3, in the first experiment, the concentration of NaOH was 12 molar. Also, the weight ratio of sodium silicate to NaOH was 0.5. The sample in this experiment contained 6 wt.% of PET particles, with the curing regime time of 3 days, oven curing temperature of 60 °C, and with the solution (alkali activator (NaOH + WG)) to solid ratio (alkali activator to the aluminosilicate source (slag)) was 0.24.

2.3. Geopolymers preparation process
Based on the designed experiments, the weight ratio of sodium silicate to alkali metal hydroxide (NaOH) solution was 0.5, 1.0, 1.5, and 2.0. Also, the weight ratio of the alkali activator to the slag portion was 0.24, 0.26, 0.28, and 0.3. The weight of the geopolymer was obtained by calculating the mold volume and practical density of about 2300 kg m⁻³ for the geopolymer. The mold used in the pressure test was cubic with dimensions of...

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**Table 2.** The parameters and levels used for evaluating the properties of the geopolymers.

| Parameter                                      | Level 1 | Level 2 | Level 3 | Level 4 |
|------------------------------------------------|---------|---------|---------|---------|
| Concentration of NaOH (molar)                  | 12      | 14      | 16      | 18      |
| Weight ratio of water glass to NaOH            | 0.5     | 1       | 1.5     | 2       |
| Weight ratio of alkali solution to slag        | 0.24    | 0.26    | 0.28    | 0.3     |
| Oven curing time in furnace (hour)             | 2       | 3       | 4       | 5       |
| Oven Curing temperature (°C)                   | 60      | 70      | 80      | 90      |
| Curing regime time (day)                       | 3       | 7       | 11      | 28      |
| Type of waste polymer                          | PET     | PVC     | PES     | SBR     |
| Weight percentage of fibers /particles         | 6       | 8       | 10      | 12      |

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**Figure 1.** (a) Pieces of waste water bottles used as PET particles, (b) pieces of waste water hose used as the PVC particles, (c) pieces of waste tires used as SBR particles, and (d) pieces of waste tire cap plies used as PES fibers.
The mold used in the test had a cylindrical shape with a diameter of 25 mm and a height of 50 mm. The mold used in the test had a cylindrical shape with a diameter of 25 mm and a height of 50 mm.

First, the alkali activator solution (NaOH + WG) precursor, and the fibers/particles were well mixed using a mechanical stirrer for 10 min. The produced mixture was a paste with medium viscosity (not so rigid and not so diluted), so it could readily fill the mold. The produced paste was poured into the mold in two steps. First, half of the mold was filled followed by vibrating the mold for 45 s, then the second half was filled and vibrated for 45 s. The reason for vibrating the mold was to obtain a homogeneous mixture and exit the air from the material.

The specimens were also heated in an oven for curing 2, 3, 4, and 5 h at 60, 70, 80, 90 °C. After the mentioned steps, the samples (molds) were placed in a water bath to complete the curing regime (3, 7, 14, and 28) days. The oven curing time was also considered for the water curing regime.

The cubic and cylindrical specimens for the compression and tensile strength test were prepared in accordance with the ASTM C873 and ASTM C496 standards [27, 28]. The force required to break the specimens was obtained. Then, according to the force obtained from the indirect tensile test in the previous step, the split tensile strength of the specimens was calculated using equation (1).

\[
f_{ct} = \frac{2P}{\pi LD}
\]

Where \( p \) is the force required to break the material, \( L \) is the length, and \( D \) is the diameter of the specimens.

Finally, the compression tests were carried out according to figure 2 illustration. Then the results were analyzed using the QUALITEK-4 software and the compressive strength of the specimens was calculated using equation (2).
The microstructure of the specimens was studied using a scanning electron microscope (SEM, TESCAN model VEGA).

Chemical functional groups of four samples (one of each level) were identified via Fourier-transform infrared spectroscopy (FT-IR, Bruker model VERTEX 70 V). The IR spectra were obtained in the region of 400–4000 cm\(^{-1}\) using the KBr pellet technique.

3. Results and discussion

3.1. Compression test

Figure 3 demonstrates the results of the compression test for the geopolymer composite specimens. As seen in figure 3, the G3 specimen (geopolymer composite containing PES fibers) and the G29 specimen (geopolymer composite containing PET Particles) had the highest and lowest compressive strength, respectively. It is also worth mentioning that the strength above 70 MPa for the geopolymer specimen cured for 14 days is exceptionally high. Table 4 represents the parameters used for producing the geopolymer specimen with the highest compressive strength.

In a study on geopolymer properties, which used borax instead of silica-rich source, results reported the compressive strength of the borosilicate geopolymer was around 57 MPa [29]. In another investigation, which used fly ash as an aluminosilicate source to produce boro-aluminosilicate geopolymer, a maximum compressive strength of around 64 MPa was reported [30]. Moreover, a recent study on fly-ash/sand geopolymers revealed that a peak of 44 MPa was obtained for the compressive strength [31]. As can be seen, the attained values for the compression strength for previous geopolymers were considerably lower than the results of the current study.

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\text{Compressive strength} = \frac{\text{Max load carried by specimen}}{\text{Top surface area of specimen}}
\] (2)

Figure 2. Illustration of the indirect (split) tensile test test (a), and the compression test test (b).

Figure 3. The graph showing the compressive strength of the geopolymer composites.
| Factor no. | Description                                      | Value |
|-----------|--------------------------------------------------|-------|
| 1         | Concentration of NaOH (molar)                    | 12    |
| 2         | Weight ratio of water to glass to NaOH           | 1.5   |
| 3         | Weight ratio of alkali solution to slag          | 0.23  |
| 4         | Oven curing time in furnace (hour)               | 4     |
| 5         | Oven curing temperature (°C)                     | 80    |
| 6         | Curing regime time (day)                         | 14    |
| 7         | Type of waste polymer                            | PES   |
| 8         | Weight percentage of fibers/particles            | 10    |

Table 4. Optimum parameters for obtaining geopolymer composite sample with the maximum compression strength.
The reason can be due to the effects of aluminosilicate source, fiber reinforcement, and curing conditions. In general, geopolymers do not form calcium-silicate-hydrates in their matrix structure, they take advantage of the polycondensation of aluminosilicate precursors to obtain structural strength. The optimal Ca/Si ratio promotes the formation of calcium-silicate-hydrate phases in the matrix [32, 33]. In particular, the higher compressive strength of the samples of this study with respect to previous studies is the presence of polymeric fibers and their strong interfacial bonding to the geopolymer matrix. The significance of this influence is individually examined by Timakul et al [33] when evaluating the effect of the presence of basalt fibers in a fly ash-based geopolymer. Moreover, it has been proven that the existence of the fibers in the mixture help reduces the porosity of the geopolymer composites [24].

3.2. Split (indirect) tensile test

Figure 4 illustrates the results of the split (indirect) tensile test for the geopolymer composite specimens. As shown in figure 4, the lowest and highest split tensile strength was obtained by the T29 specimen (containing PES fibers) and the T3 specimen (containing PET particles), respectively, which was similar to the results of the compression test. The parameters used to achieve the geopolymer composites with the highest split tensile strength are shown in table 5, which is exactly similar to the results of the compression test analysis.

Earlier studies conducted on ash-based geopolymers [12, 34, 35] have proven that elevating the curing temperature results in enhancing the tensile strength of geopolymers. This is related to the completion of geopolymerization reaction at higher temperatures. Additionally, processing at ambient temperature may be impractical due to a delayed beginning of setting. Curing at temperatures around 80 °C for 4 h has shown satisfactory tensile strength properties. Under these conditions, the majority of the geopolymerization is completed and the thermal treatment yields a rapid development in the tensile strength.

As described in section 3.1, the presence of reinforcing fibers highly developed the mechanical properties of geopolymer composites. In contrast, non-reinforced geopolymers prepared in previous investigations [12, 31] showed much lower tensile strength values (a maximum value of 6.41 MPa). This can be attributed to the addition of fibers to the composite mixture that provides high crack control in the geopolymers [36]. In addition, the strong bonding of the reinforcement fibers/geopolymer matrix is another stated contributing factor in the high tensile strength of geopolymer composites [33, 37, 38]. The reason for similar behavior of the samples during compression and tensile tests can be attributed to the uniform distribution of the particles/fibers in the samples. Moreover, high strength of PES fibers as well as the spun structure of the cap ply fibers played an important role in significant reinforcing of the fibers [39].

3.3. Analysis of results using ANOVA

Analysis of variance (ANOVA) was performed to assess the mean for the results of the tensile and compression tests. The results are presented in tables 6 and 7. Among the 8 factors affecting the tensile and compressive strength of the aluminosilicate geopolymer composites, the type of fiber/particles had the greatest impact of 96.33% and 93%, respectively. While the effect of the weight percentage of the fiber/particles on the compressive strength of the specimens was not significant. Since the fibers were added in the range of 6–12 wt.%, it can be concluded that even though the presence of the fibers was significant, the amount of the fibers in this range was not important. In a previous investigation, Khalaj et al [12] showed that the water curing regime and NaOH concentration with the contribution percentages of 55.12% and 16.83% had the most significance in the

![Figure 4](image_url). The bar chart demonstrating the tensile strength of the geopolymer composites.
Table 5. Optimum parameters for obtaining the geopolymer composite sample with the highest tensile strength.

| Factor no. | 1  | 2  | 3  | 4  | 5  | 6  | 7  | 8  |
|------------|----|----|----|----|----|----|----|----|
| Description | Concentration of NaOH (molar) | Weight ratio of water glass to NaOH | Weight ratio of alkali solution to slag | Oven curing time in furnace (hour) | Oven Curing temperature (°C) | Curing regime time (day) | Type of waste polymer | Weight percentage of fibers/particles |
| Value      | 12 | 1.5| 0.28| 4  | 80 | 14 | PES | 10   |
strength properties of the OPC-based geopolymers. In a similar study, Abdullah et al. [40] used waste coconut fiber as the reinforcement for composite cement samples and reported that the presence of 3–15 wt.% of the coconut fibers can result in obtaining a compressive strength of 43.84 MPa for the composite cement samples. This report displayed the significance of fiber present in cement-based materials. In another study, Fan et al. [38] investigated the effect of different parameters on the compressive strength properties of fly ash-based geopolymers. They stated that fly ash content had a considerable effect on the compressive strength of the samples. They also found that aluminosilicate/alkali cations and Si/Al ratios had significant effects on the strength and thermal properties of the composite geopolymers. Moreover, other studies demonstrated that NaOH concentration was the most contributing factor to the fracture energy and strength of the geopolymer composites [41–43]. On contrary, in the current investigation, it was found that NaOH concentration had a contributing factor of 0.83%, which is much lower than the earlier studies. This was because, the lowest selected NaOH level in this study was 10 molar, which was reported as the optimal concentration for obtaining the highest mechanical properties [11, 12, 44]. As can be seen, higher NaOH levels did not have a huge effect on the strength of the geopolymer composites, since the critical concentration (12 M) was already provided for all samples [42].

The curing time and weight percentage of fibers/particles did not substantially influence the compressive and split tensile strength of the specimens. The zero contribution of the curing time was contrary to the previous studies [12, 26]. This was due to the differences in the performance of the fibers/particles and the effect of the other parameters (tables 6 and 7). Figures 5 and 6 demonstrate the effect of each factor on the compressive strength of the geopolymer composites. As seen in figures 5 and 6, the PES fibers significantly affected the compressive and split tensile strength of the geopolymer composites. This behavior was also reported in former studies for PES-reinforced Portland concrete and micro fiber-reinforced geopolymer mortars. It is mentioned that the main reason for the improvement of strength of PES-reinforced composites is the high tensile properties and great flexural strength, which is introduced into the matrix properties [6, 24].

Also, the presence of other particles (SBR and PET) revealed a similar trend but with less effect on the strength compared with that of the PES fiber [8, 10, 37]. A very recent investigation demonstrated that waste PVC had a considerable influence on the concrete properties [5]. However, the effect of other parameters cannot be accurately stated. For example, it is evident that as the concentration of the NaOH enhanced, the strength of the material first reduced, and then increased.
3.4. SEM analysis

The Microstructure of the G3, G10, G12, and G14 specimens (the ones with the lowest and highest strength) was evaluated using the scanning electron microscope (SEM), as shown in figure 7. The porosities were marked by...
red squares in the figure. As can be seen, minor porosities are evident, which can be due to formation of micro bubbles during curing.

The microstructure of the geopolymers is affected by the type of material used for the alkali activator, the amount of the aluminosilicate source, and the curing temperature and time. In general, the aluminosilicate geopolymer composites are composed of a gel-like matrix and unreacted crystalline phases. The crystalline phases are originated from the impurities in the raw materials or resulted from recrystallization of the matrix phase. The Crystallinity of the unreacted phases depends on the curing temperature and the time \[16, 45\].

As demonstrated in figure 7, the two above-mentioned phases can be easily distinguished in the SEM micrograph. The matrix phase consisted of silicon oxide, aluminum oxide, and sodium oxide compounds, and the second phase consisted of silica or quartz particles.

Comparing figure 7(a) (the specimen with the highest strength) and figures 7(b)–(d) (the specimens with the lower strengths) revealed that the specimen with the greatest strength had fewer pores compared with that of the other specimens, which was expected. In other words, the more the practical density close to the theoretical density, the higher the strength. Geopolymer preparation procedures that offer the minimum porosity, i.e. proper stirring time, adequate mold vibration time, and vibration repetition results in attaining samples with the lowest porosity and the highest mechanical properties \[32, 43\]. Regarding the G14, G10, and G12 specimens, the decreased strength might be attributed to the polymeric particles poorly bonded to the matrix \[36\].

3.5. FT-IR spectroscopy

FT-IR spectroscopy was performed to evaluate the geopolymerization progress of the composites specimens with respect to the used fibers. The obtained spectra are demonstrated in figure 8. According to the results, the peaks can be classified into 5 wavenumber ranges including 400–800 cm\(^{-1}\), 800–1000 cm\(^{-1}\), 1000–1500 cm\(^{-1}\), 1500–2000 cm\(^{-1}\), and 2000–2500 cm\(^{-1}\). By comparing the results to a previous study, which used the same precursor \[12\], the band at the wavenumber of 3747 cm\(^{-1}\) was attributed to O–H stretching vibration. According to figure 8, this peak can be seen in all samples. Furthermore, the main Si–O–Si and/or Si–O–Al
stretching bands of the geopolymer samples are observed at approximately 1200–950 cm\(^{-1}\), which confirms the completion of geopolymerization reaction \([46, 47]\). Moreover, the band at 960 cm\(^{-1}\) is related to the Si–O–Si bond. Small wide peaks at the wavenumber of about 700 cm\(^{-1}\) are assigned to the Al tetracoordinates and Si–O–Al bonds. The peak at 911 cm\(^{-1}\) is related to stretching bonds of Si–O–T, which T represents a metal \([48, 49]\). In the geopolymeric composites, the peak was shifted after the geopolymerization process, which indicated the occurrence of microstructural changes during the hydration process and led to the formation of a new product (geopolymer gel). For example, in the case of samples G14, G10, and G12, the peak shifted to a higher wavenumber, which implied the weakening of this bond. The peaks in the wavenumber range of 2100 cm\(^{-1}\) are related to the vibrations of the H-Si(\textit{amorphous}) bond \([49]\). Typically, high mechanical strength is obtained during a short time after the mixing of raw materials, which is due to the strong Si–O–Al bonds in the structure. Regardless of the position of the bands, the broader the peak width in the spectrum, the more likely the material is amorphous and highly inhomogeneous \([46, 47, 49, 50]\). Evaluation of the FT-IR spectra of samples G14, G10, and G12 indicated the relative similarity of structural bonds in the samples, and the main difference is only the peak shifting of the bonds. Based on this conclusion, the remarkable point is the effect of fibers presence on the compressive and tensile strength of the geopolymer composite specimens. That is, no additional chemical bonds were found in the spectra and the strengthening of the matrix is attributed to the adhesion, crack stitching, crack growth prevention, and porosity reduction mechanisms \([36]\).

4. Conclusions

In this research, the compressive and split tensile strength of the aluminosilicate geopolymer reinforced with polymeric fibers/particles were studied and the optimum value of the parameters was found. After determining the effective parameters, 32 experiments were determined using the Taguchi experimental method. The results of the experiments were assessed using the QUALITEK-4 software. The SEM was employed to investigate the microstructure and of the geopolymers. Then presenting the following as the main results of this research study:

1. The Taguchi method was successfully used for the experimental design and investigating the influence of factors affecting the compressive and split tensile strength of the slag based aluminosilicate geopolymer composites containing polymeric fibers and particles.

2. Based on the results of the compressive and split tensile test, the obtained optimum parameters were found to be the curing time of 14 days, NaOH concentration of 12 molar, the weight ratio of sodium silicate to NaOH of 1.5, curing temperature of 80°C, polyester-type polymeric fibers, the weight percentage of the fiber/particle of 10%, and the weight ratio of solution to solid of 0.28.

3. Regarding the aluminosilicate geopolymer composite specimens, according to the results of the ANOVA analysis, among all the factors, the type of polymeric reinforcement revealed the greatest influence on the compressive and split tensile strength with the contribution percentage of 96.33% and 93%, respectively.
4. The results of the SEM microstructure analysis demonstrated that the geopolymer aluminosilicate composites were composed of a matrix phase as the polymeric gel and a secondary phase as unreacted particles of quartz and silica. Also, results depicted that other mechanisms such as crack growth prevention and adhesion to the matrix may substantially affect the tensile and compressive strength of the geopolymer composites.

5. FT-IR spectroscopy indicated the relative similarity of structural bonds in all samples and the main difference is only the peak shifting of the bonds. In fact, no additional chemical bonds was found in the spectra and the strengthening of the matrix is attributed to the adhesion, crack stitching, crack growth prevention, and porosity reduction mechanisms.

ORCID iDs

Amirreza Khezrloo @ https://orcid.org/0000-0002-0375-9739
Morteza Tayebi @ https://orcid.org/0000-0002-6237-5938

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