

Carbon fiber impact on physico-mechanical performance of slag-silica fume based geopolymer composites

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Abstract

This study probed different ratios of carbon fibers (0.1, 0.2, 0.3 and 0.4%) by weight so as to reinforce the geopolymer composites made from Water cooled slag and silica fume activated by 6% sodium hydroxide using carboxylic superplasticizer in order to achieve a good dispersion of fibers into the paste. Compressive as well as flexural strength are reported as mechanical performance of the produced composites. Several techniques have been utilized to identify the resulting geopolymer mineralogical phases and formed structure such as X-ray diffraction and Fourier transform infrared spectra. The results yielded that both compressive as well as flexural strength of the produced geopolymer composites perfectly enhanced by using carbon fiber till 0.3 % by 152.9% and 56.6% respectively. Moreover, the drying shrinkage of the reinforced composites decreased significantly compared with unreinforced one confirming the effective role of fiber in limiting drying shrinkage.

Keywords: carbon fiber, flexural strength, slag, X-ray diffraction, Fourier transform infrared spectra
Kulcsszavak: szénszál, hajlítószilárdság, salak, röntgendifrakció, Fourier-transzformációs infravörös spektrum

1. Introduction

Geopolymers, also named “alkali-activated binders”, have been introduced as a promising alternative to OPC with less environmental impacts [1, 2]. Global cement production reached 4.1 billion tonnes. However, the cement industry is characterized by high levels of environmental pollution and high energy consumption. To produce 1 ton of OPC; it generates 10,000 m³ of dust and requires 3,300 MJ of energy [3, 4].

The production and use of OPC also lead to large CO₂ emissions. In 2019, the cement industry accounted for about 10% of CO₂ emissions globally [5]. Geopolymers are typically synthesized by the following reactions: Aluminosilicate precursors e.g. clay minerals, solid waste and volcanic ash activated with alkaline solutions such as (NaOH, Na₂SiO₃ solution), then cured at a specific temperature. The precursor undergoes a geopolymerization process, including dissolution, rearrangement, condensation and resolidification in concentrated solutions forming 3D polymeric chain and ring structure consisting of Si-O-Al-O bonds network of geopolymer.

In the present research the geopolymers are produced by mixing waste like water cooled slag (WCS) which is by-product of smelting iron ores or natural aluminosilicate sources, such as fly ash (FA), silica fume (SF), and metakaolin (MK), with alkali activators such as sodium hydroxide and sodium silicate. Although WCS in geopolymer offers high compressive strength and durability, it provides low flexural and tensile strength;

moreover high drying shrinkage thus fibers reinforcement are suggesting to solve these problems. Fiber reinforcement is widely recognized as an environmentally friendly technique that can efficiently improve the mechanical strength of materials while increasing their toughness. The opening and growth of cracks is controlled by the bridging action of fibers [6, 7] like polypropylene, PVA, and steel fiber as well as carbon fiber.

The physical filling and pozzolanic properties of the SF makes it an ideal candidate to be used as a supplementary cementitious material. Carbon fiber (CF) is the one of the most favorable fiber type which known the most common fiber reinforcements added to geopolymer composites [8] regarding its properties such as high strength and low thermal expansion [9] as well as alkaline resistance.

Carbon fiber helps to maintain the compactness of the concrete [10]. In addition, it is resistant to microcracks and enhances the tensile strength, Young's modulus, fatigue resistance, and ductility of materials.

2. Materials and methods

Material rich in aluminum silicate content and possess an amorphous form is a potential raw material for manufacturing geopolymer binders

Water – cooled slag (WCS): Granulated blastfurnace slag is a by-product of the manufacture of iron from iron ore, composes mainly of aluminosilicates and calcium oxide as illustrated in

Oxide content (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₂	K ₂ O	Na ₂ O	TiO ₂	MnO	P ₂ O ₅	Cl-	L.O.I	BaO	SrO	Total
Water cooled slag (WCS)	36.37	10.31	0.50	38.82	1.70	2.17	1.03	0.48	0.57	4.04	.04	0.05	0.12	3.28	0.18	99.96
Silica Fuma (SSF)	93.00	0.46	1.08	0.42	0.79	0.22	0.34	0.60	0.07	-	0.05		2.82	-	-	99.85

Table 1 chemical composition of raw materials, wt. %
1. táblázat A nyersanyagok kémiai összetétele, tömegszázalékban kifejezve

chemical analysis shown in Table 1. XRD pattern shows an almost completely amorphous structure Fig. 1.

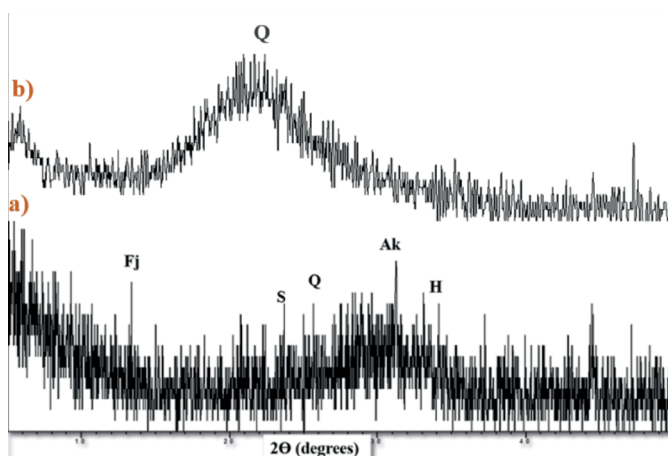


Fig. 1 XRD pattern of the starting raw materials
a) water cooled slag b) silica fume, Ak: Akermanite, Fj: Faujasite, H: Hematite, Q: Quartz and S: Sodalite

1. ábra A kiindulási nyersanyagok röntgendiffraktogramja (XRD)
a) vízzel hűtött salak b) szilícium-dioxid-füst, Ak: Akermanit, Fj: Faujasit, H: hematit, Q: kvarc és S: szodalit.

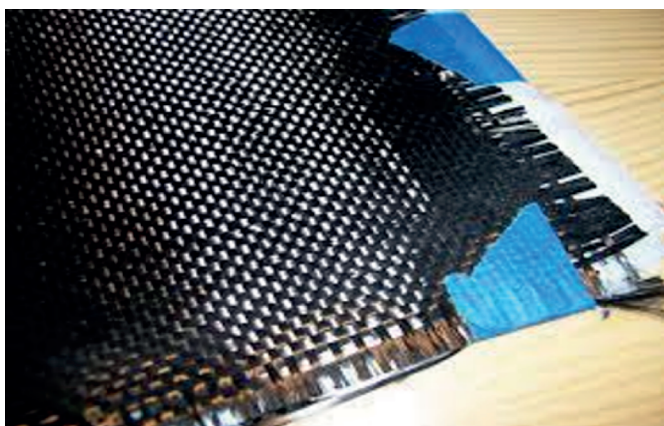


Fig. 2 Carbon fiber image
2. ábra Szén-szálak kép

Silica fume (SF): is obtained from Mast Egypt Company, is a sort of a fine non-crystalline polymorph of silica has SiO₂ ≥85% according to ASTM C 1240.

SF is a secondary product of silicon and ferrosilicon alloy producing industries. The composition of silica fume is represented in Table 1.

Alkaline solution: Sodium hydroxide solution of desired concentration is prepared by mixing 97–98% with tap water, the density of sodium hydroxide solution is about 1.15 g/cm³.

Carbon fiber (CF): with diameter of 7 μm and length of 6 mm, obtained from Sika Company, Egypt shown in Fig. 2.

Superplasticizer: Glenium Ace 30-polycarboxylate-based superplasticizer in order to attain superior workability and required flowability of the fresh paste is added to the mixture with stirring to yield the homogenous mixture as well as excellent mechanical properties.

2.1 Composite preparation and curing regime

The Processing of the geopolymer composites is taken place according to the manner illustrated in Fig. 3. As the raw materials were ground to a Blaine that allowed passing through a 90 m sieve. Dissolving sodium hydroxide pellets in mixing water with stirring for complete dissolution then leaving the solution to cool for a sufficient period until it reached room temperature results in an exothermic reaction and, therefore, generates high temperature of 25 ± 2°C. The alkaline activator NaOH solution was gently added to the raw material powder into a pan mixer as well as mixed for 5 minutes then the fiber added with well mixing to ensure a good homogeneity. The superplasticizer added in a sufficient amount to increase the workability of the mixture and achieve a good dispersion.

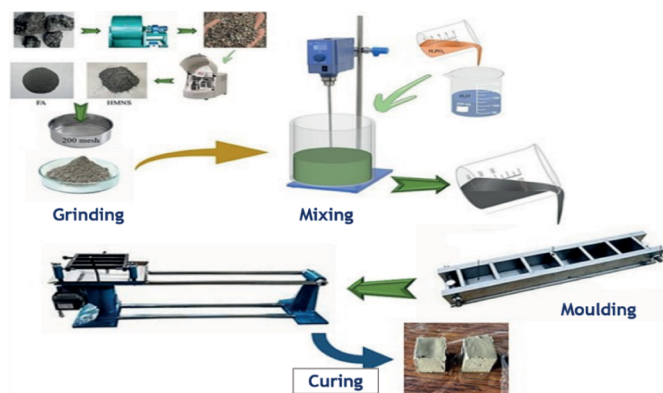


Fig. 3 Processing and casting of geopolymer composites
3. ábra Geopolimer kompozitok feldolgozása és öntése

A mold of dimensions 2.5 x 2.5 x 2.5 cm was first lubricated to prevent sample breakage through demolding. The paste was introduced and compressed manually into the mould. After the casting process was complete, the mold was covered and left at room temperature (23°C) for 24 hours to dry completely, after

which it was removed from the mold and stored in a humidity chamber at 38°C and 90% relative humidity.

2.2 Test parameters

The masses of the key components of geopolymer were determined prior to mixing using a predetermined Si:Al:Na ratio. The effect of adding carbon fiber into 0.1, 0.2, 0.3, 0.4 % to the WCS-SF geopolymer mix is investigated by XRD and FTIR techniques in order to study geopolymer reinforcement influence and investigate the physicochemical properties (water absorption and shrinkage) of the produced geopolymer composites as well as estimate mechanical performance. The schematic of the mix design of all mixtures is illustrated into Table 2.

Mix no.	Mix Composition slag % SF %	NaOH, %	Water/ binder ratio	Super- plasti- cizer %	Carbon fiber %
Q (control)	90 10	6	0.26	0.93	9
S1	90 10	6	0.21	1.07	0.1
S2	90 10	6	0.20	1.07	0.2
S3	90 10	6	0.20	1.21	0.3
S4	90 10	6	0.26	1.07	0.4

Table 2 mix composition of the geopolymer composites (Mass, %) 2. táblázat A geopolimer kompozitok keverékösszetétele (tömeg, %)

3. Results and discussion

3.1 Fourier transforms infrared spectroscopy

FTIR is an essential technique used to identify structural information of alkali-activated blast furnace slags and their hydration products [11]. FTIR technique was carried out to understand the reaction occurring between the matrix and fiber, Investigating for WCS-SF reinforced with 0, 0.1, 0.3 and 0.4 of carbon fiber cured for 90 days then displayed in Fig. 4. The bands in the region of 3400~3500 cm⁻¹ were assigned to the stretching vibrations of the octahedral OH bonds attached to the Al octahedron sheet and to the hydration binder formed during reaction, whereas the band at about 1645 cm⁻¹ was attributed to the bending vibrations of H-O-H [12].

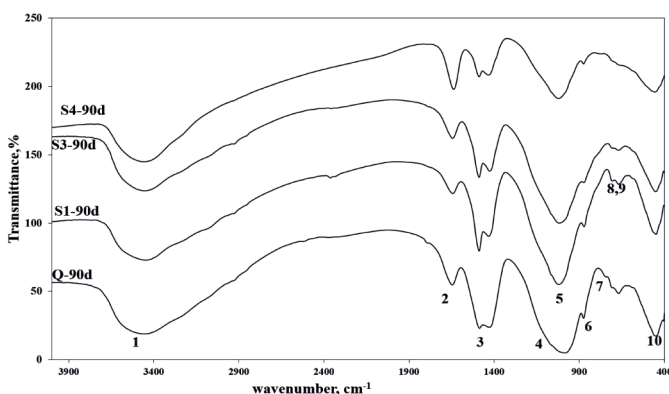


Fig. 4 FTIR spectra of slag-SF geopolymer composites reinforced by 0, 0.1, 0.3 and 0.4% of CF cured for 90 days

4. ábra A 90 napig keményített 0, 0.1, 0.3 és 0.4% CF-sel erősített salak-SF geopolimer kompozitok FTIR spektrumai

The figure revealed that the peak of asymmetric Si-O-Si for non-solubilized silica decreased slightly with shift for lower wave number. Which is due to the alkaline activation for the constituents of the geopolymer mixture confirmed by increasing in asymmetric T-O-Si bands (T, tetrahedral =Si or Al) [13]; as carbon fiber content ratio increase to 0.3% this signifies that the geopolymer reaction well occurred and enhanced the geopolymer composites structure with vitreous content of the matrix.

Excess inclusion of carbon fiber resulted in hindering for geopolymer chains propagation as well as the splitted carbonate are significantly exhibited large peaks located about 1448 and 870 cm⁻¹. On the other side, by studying geopolymer propagation during hydration age till 90 days for the 0.3% CF mix presented in Fig. 5. The figure clarifies that the principal asymmetric bands for amorphous geopolymer elucidate the propagation in the reaction with time. The broadness of OH bands around 3400 as well as 1645 cm⁻¹ worth noticed their upgrade indicating C-S-H and C-A-S-H formation with time.

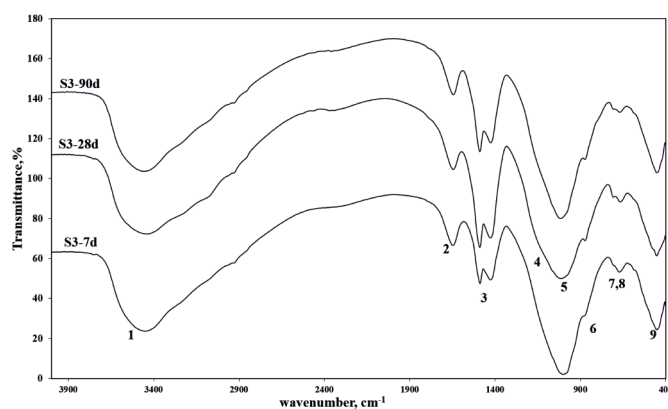


Fig. 5 FTIR spectra of slag-SF geopolymer composites reinforced by 0.3% of CF cured for 90 days

5. ábra A 90 napig keményített 0,3% CF-fel erősített salak-SF geopolimer kompozitok FTIR spektrumai

Band no.	Identification
1	Stretching vibration of (OH)
2	Bending vibrations of (HOH)
3	Stretching vibration of CO ₂
4	Asymmetric stretching vibration (Si-O-Si)
5	Asymmetric stretching vibration (T-O-Si)
6	Symmetric stretching vibration CO ₂
7,8	Symmetric stretching vibrations (Si-O-Si)
9,10	Bending vibration of (Si-O-Si)

Table 3 FTIR bands 3. táblázat FTIR-sávok

3.2 X-ray diffraction technique

In order to investigate the mineralogical composition and the amorphous structure of the reinforced composites compared with the unreinforced one, the XRD patterns of geopolymer composites of Slag-SF at 90 days of curing age (Q, S1, S3 and S4) reinforced with CF ratio 0,0.1,0.3 and 0.4 respectively, also XRD pattern of 0.3% CF along age of hydration represented in Fig. 6 and Fig. 7 respectively.

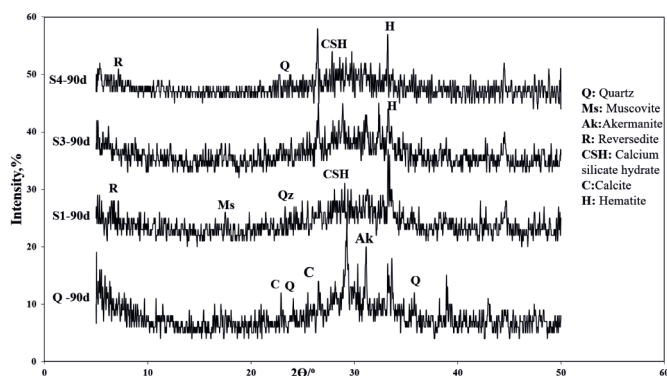


Fig. 6 XRD pattern of slag-SF geopolymer composites reinforced by 0, 0.1, 0.3 and 0.4 % of CF cured for 90 days

6. ábra A 90 napig keményített 0, 0,1, 0,3 és 0,4% CF-fel erősített salak-SF geopolimer kompozitok röntgen diffraktogramjai (XRD)

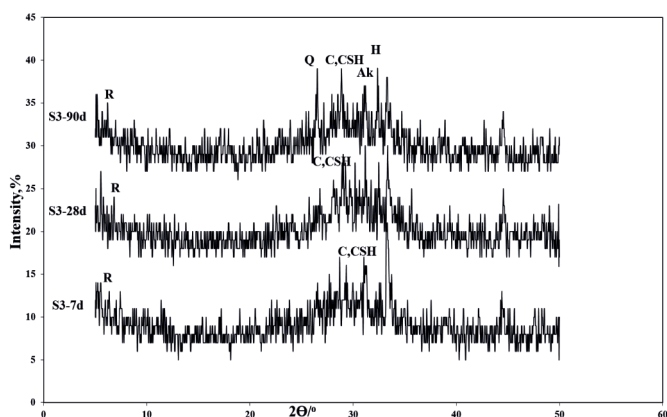


Fig. 7 XRD pattern of slag-SF geopolymer composites reinforced by 0.3% of CF cured for 90 days

7. ábra A 90 napig keményített, 0,3% CF-fel erősített salak-SF geopolimer kompozitok röntgen diffraktogramjai (XRD)

It is obviously shown that the hump peak between 17 and 35° which is characteristic for inorganic polymers [14] appeared significantly in the XRD pattern. It can be noticed also that most of the mineral components in the silica fume belong to vitreous substances, so obvious crystallization peaks cannot be found with little inclusion of Calcite and Hematite reflecting the formation of reinforced WCS-SF composites

The patterns signifies that the almost amorphous structure of the produced composites contained tiny inclusions of crystalline quartz and hematite which are inactive fillers beside traces from Akermanite (CaMgSilicate). Moreover, the WCS-SF reinforced by 0.3% of CF persevered the growth of CSH and vitreous structure with time. As the CF content increase the little presence of quartz may hinder more geopolymer formation affected negatively on the amorphous structure.

3.3 Compressive strength

Compressive strength test carried out on alkali activated WCS-SF geopolymer composites comparing between neat geopolymer composites and reinforced one with carbon fiber and the pattern represented in Fig. 8. The results obtained that the presence of carbon fiber affected positively and enhance mechanical properties of the geopolymer which illustrated that

the values of compressive strength increased considerably with carbon fiber content almost up to 0.3% by weight, moreover the pozzolanic properties of silica fume beside the filler effects was found to improve the tensile characteristics of geopolymer mixes by enhancing the bond strength between matrix and fiber which increase the carbon fiber bond with matrix in turn the interfacial adhesion increase. As the CF content increase the compressive strength noticed to be significantly decreased, the addition of excessive amount of fibers causes higher shear resistance to flow therefore, decreased flowability which in turn negatively affect compressive strength.

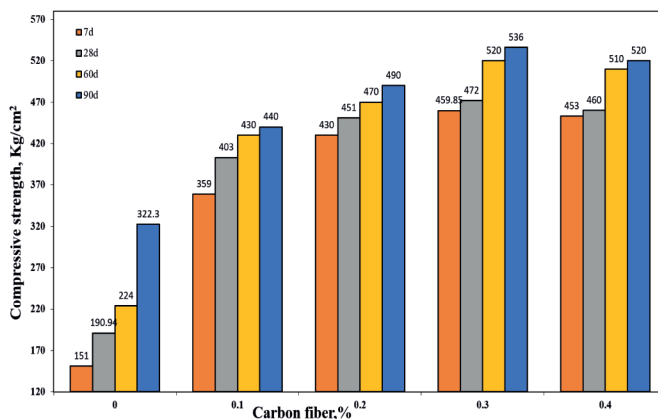


Fig. 8 Compressive of slag-SF geopolymer composites reinforced by 0, 0.1, 0.3 and 0.4 % of CF cured for 90 days

8. ábra A 0, 0,1, 0,3 és 0,4 % CF-fel erősített salak-SF geopolimer kompozitok 90 napon át történő keményítése

The results are well compatible with water absorption and shrinkage outcome showing that the imperfect dispersion of higher amounts of fibers resulted in forming fiber pockets accumulated caused hindering for geopolymer chain propagation which affected negatively on the strength when the CF exceeds 0.3%.

3.4 Effect of carbon fiber on water absorption of WCS-SF geopolymer composites

Water absorption occurs through the diffusion of water across the interfacial defects that exist between the fiber and matrix, as well as through the voids present in the matrix. The incorporation of geopolymer with fibers has been found to reduce the presence of water in the pores of the composites, resulting in a compatible structure. This is due to the water absorption capability of the fibers themselves, which decreases the amount of water absorbed by the matrix [15]. The water absorption of WCS-SF is illustrated graphically in Fig. 9, demonstrating the synergistic effect of reduced water absorption as a result of improved interfacial adhesion between the fiber and the matrix. It is also worth noting that water cooled slag exhibits rapid hardening behavior and higher calcium content, which can lead to accelerated geopolymerisation through the formation of semi-crystalline Ca-Al-Si gel, CSH, and reversedite phase.

The results clearly indicate that the presence of carbon fibers up to a concentration of 0.3% limits the water absorption within the matrix. However, as the content of carbon fibers increases, it weakens the bond between the fibers and the matrix.

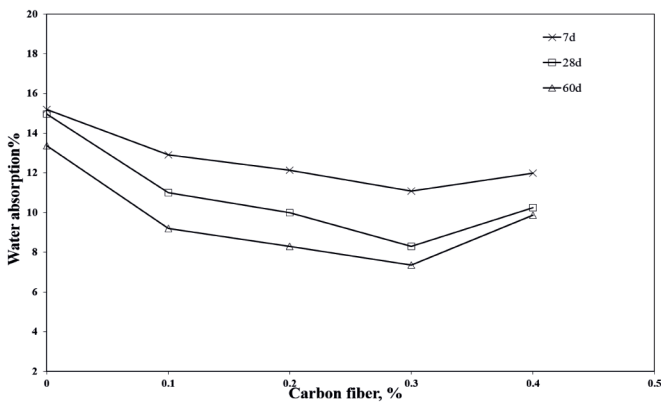


Fig. 9 Water absorption of slag-SF geopolymer composites reinforced by 0.3% of CF cured for 90 days

9. ábra A 0,3 % CF-fel erősített salak-SF geopolimer kompozitok vízfelvétele 90 napon át keményítve

3.6 Mechanical performance of the slag-SF geopolymer composites

Fiber reinforcements are required in order to address the brittle nature of geopolymers, which has sparked an increased interest in sustainability. The term flexural strength pertains to a material's ability to withstand deformation while under a load, as illustrated in Fig. 11. As a quantitative measure, flexural strength should represent the maximum stress experienced by a material at the point of rupture. The strong adhesion between the fiber and matrix at the interfaces has resulted in the fibers bearing the tensile forces that are exerted. Furthermore, the presence of fibers serves to prevent the formation and propagation of cracks within the reinforced composite.



Fig. 11 Flexural strength apparatus

11. ábra A hajlítószilárdságmérő készülék

3.5 Effect of carbon fiber on drying shrinkage of WCS-SF geopolymer composites

Literature has suggested that the use of fibers for reinforcement in geopolymer is an important technical solution to address the issue of shrinkage [2]. Additionally, it is anticipated that the presence of microfibers in geopolymer composites will effectively control the shrinkage of these composites. The findings related to shrinkage align with the results of water absorption tests, which confirm that the reinforcement of alkali activated WCS-SF geopolymer with carbon fiber has successfully mitigated the formation of microcracks, as illustrated in Fig. 10.

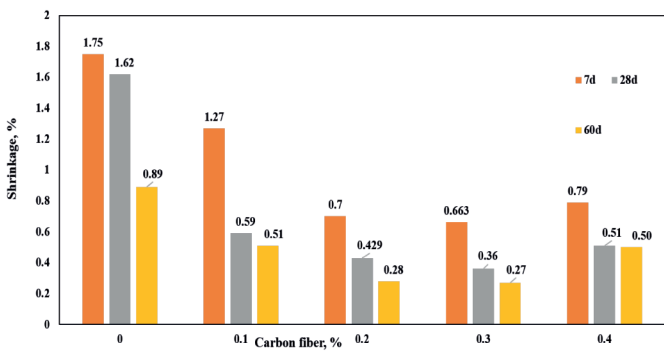


Fig. 10 Shrinkage of Slag-SF geopolymer composites reinforced with various ratio of CF fiber cured till 60 days

10. ábra A különböző arányú CF szálakkal erősített salak-SF geopolimer kompozitok zsugorodása 60 napig keményítve

The inclusion of carbon fibers (CF) in geopolymer composites serves to restrict the phenomenon of drying shrinkage, indicating that the occurrence of cracks is effectively managed due to the bridging influence exerted by these fibers. However, when the CF content surpasses the threshold of 0.3%, there is a slight increase in shrinkage, which can be attributed to the heightened dispersion of the fibers, resulting in composites with diminished mechanical properties and elevated shrinkage values. The graphical representation clearly illustrates that the shrinkage values diminish over time, owing to the continuous infiltration of pores by geopolymer chains and the CSH binder phase.

The examination of the flexural resistance of the inclusion of 0.3% carbon fiber in WCS-SF geopolymer composites was conducted in accordance with the standards outlined in ASTM-C1609.

The graphical representation depicting the correlation between the flexural and compressive resistance in megapascals (MPa) after 28 days is presented in Fig. 12.

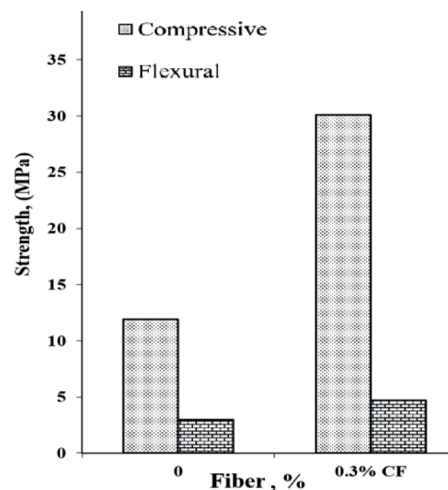


Fig. 12 Compressive and flexural strength of alkali activated 90%Slag-10%SF geopolymer mixes reinforced by 0.3% of CF at 28 days

12. ábra Lúggal aktivált 90% salak-10%SF geopolimer keverékek nyomó- és hajlítószilárdsága 0,3% CF-szállal megerősítve 28 nap után

The results implies that the incorporation of the carbon fiber enhanced the compressive strength by 152.9% while improved the flexural resistance of the geopolymer composites by 56.6% however, the content of the carbon fiber exceeds 0.3% it was noticeably that the flexural and compressive strength significantly show little decrease as this decrease in the flexural strength poor interaction and dispersion of fibers in matrices after a particular concentration underpins flexural strength may be attributed to the weak interfacial bond between the fiber and the matrix, probably due to the agglomeration of the fibers [16].

3.7 Scanning Electron Microscopy Investigation

One function of the fibers is to connect the pores in the geopolymer in order to improve the adhesion of the composites [17]. Studying morphology by SEM of WCS-SF geopolymer reinforced with 0.3% carbon fiber compared with neat one displayed in Fig. 13. Illustrating that the unreinforced geopolymer structure noticeably has pores according to many studies report that these micropore presence in geopolymers attributed to the quartz disturbance in geopolymer matrices. Quartz, as yielded from XRD, hinders the geopolymer reaction through causing interfacial separation, and interfacial microcracks formation. The image (B) cleared that carbon fiber moreover the presence of silica fume due to its filler are highly imbedded in CSH gel matrix confirming high bond strength among them leading to formation high dense structure and fill all voids effect consequently enhance mechanical characteristics this can prove that the adding superplasticizer did solve the CF agglomeration problem.

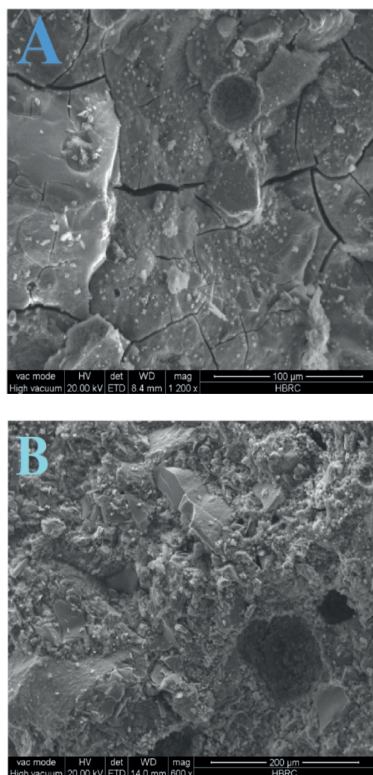


Fig. 13 SEM micrograph of Slag-SF geopolymer composites reinforced with of Carbon fiber
 A: unreinforced geopolymer B: reinforced geopolymer with 0.3% CF
 13. ábra A szénszállal erősített salak-SF geopolimer kompozitok pásztázó elektronmikroszkopos (SEM) felvétele
 A: nem erősített geopolimer B: 0,3% CF-vel erősített geopolimer

4. Conclusions

From the previous work the following concluded remarks can be drawn in the following points:

- Water-cooled slag offers a rapid hardening behavior, in addition to a higher calcium content, which may lead to an accelerated geopolymerization process.
- The presence of fibers has a positive impact on the healing of cracks and helps to reduce the drying shrinkage of the geopolymer material.
- Fibers significantly enhance the physicomechanical properties of the geopolymer, contributing to its overall performance.
- Silica fume serves to increase the pozzolanic activity and effectively improves the filling of voids within the material.
- The inclusion of silica fume has been found to be advantageous in terms of enhancing the compressive strength of the geopolymer.
- The addition of a superplasticizer to the mixture during the mixing process plays a crucial role in facilitating easy casting and ensuring a uniform distribution of fibers.
- The incorporation of 0.3% carbon fiber has a remarkably positive effect on the mechanical properties of the water-cooled slag-silica fume geopolymer, significantly increasing its compressive strength by 152.9% and also improving its resistance to bending by 56.6%.

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