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Enhancement effect of polypropylene fiber on the characteristic properties of alkali-activated metakaolin-slag mortar

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Abstract

Production of eco-friendly concrete is a global aim, to tackle this matter; Geopolymers have been identified as viable alternative replacements for ordinary Portland cement due to its excellent engineering properties and low CO_2 emissions. The advancement of geopolymer concrete (GPC) represents a significant milestone to make it more applicable and popular. An effort was made to investigate the impact of the blended Metakaolin (MK) and Ground Granulated Blast Furnace Slag (GGBFS) with Polypropylene Fiber (PPF) in various contents (0%, 0.5%, 0.1%, 1.5%, 2%, 2.5%) as reinforcement additive. For this, an experimental study was carried out to evaluate the effect of PPF on properties of the product. SEM, XRD, and FTIR analyses have been performed to determine the surface morphology and phases. Results show that incorporating PPF effectively reduces the shrinkage and improves its compressive strength compared with the control geopolymer mortar; optimum value was obtained with a PPF ratio of 0.5%.

Keywords: geopolymer, polypropylene fiber, metakaolin, slag, compressive strength.
Kulcsszavak: geopolimer, polipropilén szál, metakaolin, salak, nyomószilárdság

1. Introduction

The International Energy Authority (IEA) estimates that between 6 and 7 percent of all CO_2 emissions come from the production of Portland cement. In addition, the steel and aluminum industries consume the most energy, followed by the cement industry. In order to find solutions to these issues, numerous experiments have been carried out in order to identify new methods that can fulfill the requirements of the construction industry for an alternative to concrete [1]. 'Geopolymer' technology, coined by Davidovits, refers to the process of forming cement through the utilization of alkali minerals and aluminosilicates, Geopolymers are able to cure and harden at ambient temperatures [2]. When compared geopolymer to OPC, one of the most significant differences in the production process is that the production of OPC involves a high-energy manufacturing process "calcination". In contrast, Geopolymer employs industrial wastes (such as slags, fly ashes,...) which considered low-energy materials and a small quantity of high-energy materials (alkali hydroxides) to initiate a reaction. contrary to the hydration of Portland cement, just a limited quantity of H_2O has been used in the manufacturing of geopolymer gel; as a result, less mixing water is needed [3]. Finally, even though geopolymers exhibit less shrinkage than Portland cement materials, it is still an important consideration [4-6]. The incorporation of polypropylene fiber aims to create a geopolymer that is both strong and lightweight with low drying shrinkage. Polypropylene (PP) fibers are a set of synthetic fibers with certain features that arranged on a plastic matrix make them an excellent supplement to building materials *Fig. 1*. It is anticipated that the final geopolymer product will be appropriate for use as thermal insulation or fire resistance [7].

Noushini [8] employed the incorporation of polypropylene and polyolefin fibers to enhance the deformation and contraction characteristics of fly ash-based geopolymer with low calcium content. The findings indicated a reduction in drying shrinkage and an enhancement in compression creep within the geopolymer concrete, observed at both early and long-term stages, when a fiber volume fraction of 0.5% was added. Zhang et al. [9] investigated. The impact of polypropylene fiber on mechanical properties and volumetric stability, demonstrating that PP fiber can effectively bridge detrimental pores and defects. This modification alters the propagation pathways of cracks, leading to significant enhancements in both strength and toughness. Talib et al. [10] studied GPC preparation with (16M) of sodium hydroxide(NaOH), and with polypropylene fiber (PPF) and found there is a 50%, 30%, 16% enhancement in the flexural tensile strength, compressive strength, and split tensile strength with PPF addition. Haci Baykara et al. [11] Examined the interaction of PPF and zeolite-based geopolymer mortar and the influence of calcium hydroxide on the mechanical characteristics and thermal stability of the mortars and demonstrated that the optimum compressive strength was with PPF 0.5 wt.%. Zainab A. Mohammed et al. [12] revealed that The integration of PPF enhanced both the splitting tensile strength and the compressive strength.

Due to its high toughness and anticorrosion, (PPF) has been mostly used in reinforced cement and concrete industries. Although it was a conflict that stated that the incorporation of polypropylene fibers (PPF) significantly diminishes the compressive strength of concrete, Richardson [13] studied this conflict. He was noticed that the breaking of the cement bond due to the presence of PPF leads to a notable reduction in the compressive strength of the concrete. Although the similar strength reduction was expected in geopolymer by PPF inclusion. Zhang et al. [9] stated that the initial compressive strength of the fly ash/calcined kaolin geopolymer exhibited an increase of approximately 68% and 20% at 1 and 3 days, respectively, by the incorporation of PPF in 0.5 wt%. Additionally, the early flexural strength of the composite with 0.75% PPF was doubled for both days. Navid Ranjbar et al. [7] evaluated that the addition of PPF up to 3 wt% to the geopolymer paste enhances the composites energy absorption and minimizes the shrinkage. However, depending on the fiber content, it may lower the material flexural and compressive strength.

Fig. 1 Polypropylene fiber used in this study
1. ábra A vizsgálatban használt polipropilén szál

Arash Karimipour and Jorge de Brito [14] evaluated the impact of silica fume (SF) and polypropylene fibers (PPF) on the mechanical behavior and fracture mechanics of ultra-high performance geopolymer concrete (UHPGC). The results of the experiments indicated that PPF is crucial in enhancing both mechanical and fracture characteristics of UHPGC that contains SF and PPF.

Aisheh, Y. et al. [15] studied the presence of hybrid fibers in a GPC which successfully aids in the transition to a softer state and successfully reduces abrupt failure. According to Sangi, R. et al. [16] adding 1% steel fibers and 0.1% PPF in a Geopolymer Concrete specimen, significantly increases displacement ductility factor and capacity of energy absorption. Also Sangi, R. et al. [17] emphasized that the type and dosage of fiber affect the mechanical properties of GPC; the compressive strength was used to determine the ideal fiber dosage for different grades of GPC. By varying the fiber percentage, they found that the optimal dosage for polypropylene fibers in geopolymer concrete was 0.6%.

The objective of this paper is to examine the impact of the incorporation of polypropylene fibers to geopolymer matrix through measuring XRD, FTIR, mechanical properties, drying shrinkage and also their thermal stability compared to control mix. The goal is to identify the most effective matrix which can be applied in construction applications.

2. Materials and methods

Geopolymers or Alkali-activated materials (AAMs) are a class of inorganic binders that are formed by reacting aluminosilicate materials with an alkaline solution

2.1 Raw materials

The materials used in this study are Metakaolin (MK) obtained from Aluminum Sulfate Co. of Egypt (ASCE) and Ground Granulated Blast Furnace Slag (GGBS) obtained from Iron and Steel factory, Helwan, Egypt as starting materials. Polypropylene fiber 12 mm length obtained from polyfibers company (Turkey) used as additive in ratios 0.5, 1, 1.5, 2, 2.5%. *Table 1* shows the chemical composition of MK and GGBFS used in this work. *Fig. 2* shows the XRD patterns of MK and GGBS. Liquid Sodium Silicate LSS (Na_2SiO_3) used in the present work received from Fisher company and has a density of 1.46 g/cm^3, it was mixed proportionally at equal volume of (NaOH) solution as alkali activator. In order to prevent coagulation of the added fiber, superplasticizer was obtained from Sika Company and added in various ratios from the total weight of binder for better dispersion of the added compositions.

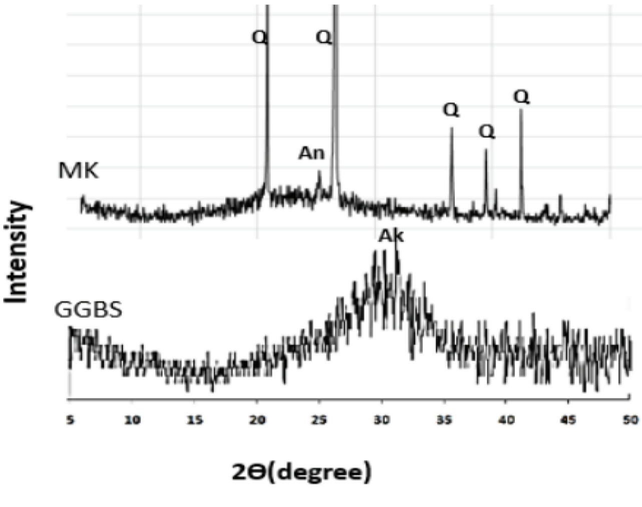

Fig. 2 XRD pattern of starting raw materials (Q= Quartz, An= Anataze, AK= Akermanite)
2. ábra A kiindulási nyersanyagok röntgendiffrakciós (XRD) mintázata (Q = kvarc, An = anatáz, AK = akermanit)

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	TiO_2	L.O.I	Total
MK	61.1	31.4	1.51	0.32	0.11	0.12	0.14	0.13	2.44	2.33	99.6
GGBFS	36.59	10.1	1.84	33.07	6.43	1.39	0.74	3.52	0.52	–	99.96

Table 1 Chemical composition of MK and Slag
Table 1 Chemical composition of MK and Slag

2.2 Methods

Geopolymer mortar activated by using an equal volume of sodium hydroxide (NaOH, 7M) and liquid sodium silicate LSS (Na_2SiO_3). PPF was added at ratios of 0.5, 1, 1.5, 2, and 2.5% by powder mass. Sonication used for well dispersion. Mixes were poured into 2.5 cm length cubic molds, smoothed with a thin edged trowel, and cured at room temperature for 24 hours, the demolded specimens cured at 40 °C with 100% relative humidity. Specimens were taken out from their curing condition, dried at 80 °C for 24 hours, then tested for compressive strength. To prevent further hydration, the crushed specimens being dried at 105 °C until the testing time, it kept in an airtight container. Compressive strength was carried out at 7, 21, 28, 60 and 90 days according to ASTM C109 M [18]. All the prepared mixes of different materials were left to be cured at the lab ambient condition of 25 °C for 24 hrs. Then, subjected to curing temperature of 40^{o}C at a 95±5% relative humidity in a controlled humidity chamber. *Table 2* shows the geopolymer mix design for the mortar.

mixes	GGBFS%	MK%	PPF%	SH (7M)	LSS	SP	w/b
SMP0	50	50	0	0.125	0.125	0.8	0.250
SMP1	50	50	0.5	0.125	0.125	1.0	0.250
SMP2	50	50	1	0.125	0.125	1.2	0.250
SMP3	50	50	1.5	0.130	0.130	1.4	0.260
SMP4	50	50	2	0.130	0.130	1.6	0.260
SMP5	50	50	2.5	0.130	0.130	1.8	0.260

Table 2 The composition and designation of the different mixes.
2. táblázat A különböző keverékek összetétele és jelölése

2.3 Investigation Methods

Starting materials investigated by using XRF-Axios (PW4400) WD-XRF Sequential Spectrometer Netherland, while mineralogical characterization was done by Philips PW 3050/60 diffractometer using a Cu-Kα source. The infrared spectral analysis used for elucidation of the amorphous constituents of geopolymer composites using Jasco-6100 –America, with the aid of KBr binder in the range from 400 to 4000 cm^{-1}. Mechanical testing was performed using 5 tons German Brüf digital compression testing machine with a loading rate of 100 kg/min [19, 20]. Morphology of the hydration products was examined by scanning electron microscope (SEM) Inspect S (FEI Company, Netherlands) equipped with an energy-dispersive X-ray analyzer (EDX). Drying shrinkage percent was calculated at ages of 28 and 90 days of curing. The thermo-mechanical properties tested for the composites by exposing them to high temperatures in the furnace. After curing at 28 d each sample subjected to thermal treatment at 300 °C, 500 °C and 800 °C at a sustained period of 2 h. weight loss, firing shrinkage and residual percent of the strength were calculated.

3. Results and discussion

Experimental studies were established to examine the impact of PPF on geopolymer mortars and Determine the best added ratio.

3.1 Fourier Transform Infrared Spectroscopy FTIR

Fig. 3 represents the FTIR spectra of alkali-aktivated mortar having various ratios of PPF. A significant peak is observed within the wavenumber range of 3467-3434 cm^{-1}, which is associated with the stretching vibration of the O–H bond found in hydroxyl groups present in water molecules or CSH and CASH phases. The presence of hygroscopic water is evidenced by the peaks detected in the range of 1645-1638 cm^{-1}, attributed to the bending vibrations of water molecules within the amorphous matrix of the geopolymer mortars [21]. The observed peaks in the range of approximately 3000–2800 cm^{-1} can be attributed to the presence of CH2 groups, which mainly from the inclusion of polypropylene within the geopolymer matrix. The increased broadness of O-H vibration band at 3467-3434 cm^{-1} favors the increased formation of CSH and CASH with polypropylene up to 0.5%. This is coherent with the increased intensity of asymmetric T-O-Si of amorphous geopolymer and decreased the intensity of non soulibilized silica at about 1100 cm^{-1} which dissolved by the enhancement effect of activator as well as the addition of polypropylene. The observed peak within the wavenumber range of 1455 to 1426 cm^{-1} can be attributed to the stretching vibration of the O–C–O bond, resulting from the formation of calcium carbonate and sodium carbonates. [7, 22].

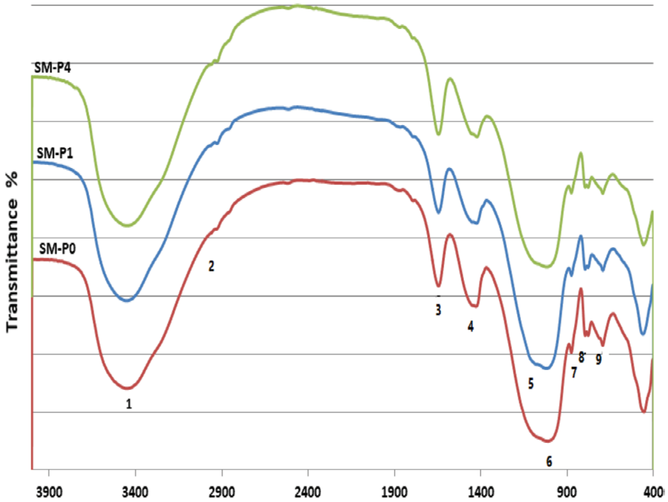

Fig. 3 FTIR spectra of 90 days alkali-activated composite having various ratios of PPF.
(1: OH asymmetric and symmetric stretching vibration, 2: bending vibration of (HOH), 3: stretching vibration of CO_2, 4: asymmetric stretching vibration (Si–O–Si), 5: asymmetric stretching vibration (T–O–Si), 6: symmetric stretching vibration of CO2,7: symmetric stretching vibration (A1–O–Si), 8: symmetric stretching vibration (Si–O–Si), 9: bending vibration (Si–O–Si and O–Si–O))
3. ábra A 90 napos lúgosan aktivált kompozit FTIR spektrumai különböző PPF arányok mellett.
(1: OH aszimmetrikus és szimmetrikus nyúlási rezgés, 2: (HOH) hajlítási rezgés, 3: CO_2 nyúlási rezgés,4: (Si–O–Si) aszimmetrikus nyúlási rezgés, 5: (T–O–Si) aszimmetrikus nyúlási rezgés, 6: CO_2 szimmetrikus nyúlási rezgés, 7: (Al–O–Si) szimmetrikus nyúlási rezgés, 8: (Si–O–Si) szimmetrikus nyúlási rezgés, 9: (Si–O–Si és O–Si–O) hajlítási rezgés)

3.2 XRD Analysis

Fig. 4 shows the X-ray diffraction patterns of 90 days GGBFS –MK composites without and with PPF for specimens dosages 0.5% and 2%, It was consisted mainly from quartz as crystalline phases, a rise between hump in the region 20 and 35° in 2θ, which is a normal behavior in amorphous materials related to geopolymer matrix, observed for specimens with dosage of 0.5% PPF. When dosage increased the degree of crystallinity increase. Forming intense peak of sodalite which is one of the zeolites, in addition to the increased intensity of calcite as a result of carbonation of unreacted sodium ions [11]. The figure reflects also the absence of calcite peak at 23.05° where most of the added alkalis consumed in the geopolymer formation and not subjected to carbonation. This will lead to an increased porosity within the matrix. XRD analysis demonstrated that the addition of PPF to the geopolymer matrix did not lead to the emergence of any new crystalline phases within the composite material.

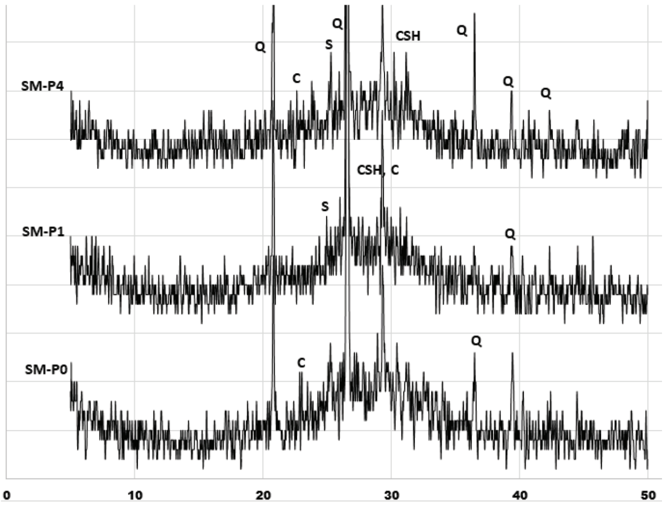

Fig. 4 X-ray diffraction pattern of 90-days alkali activated GGBFS-MK composite with various ratios of PPF (Q: Quartz, C: Calcite, S: Sodalite, CSH: Calcium Silicate Hydrate)

4. ábra A 90 napos lúgosan aktivált GGBFS–MK kompozit röntgendiffrakciós mintázata különböző PPF arányok mellett (Q: kvarc, C: kalcit, S: szodalit, CSH: kalcium-szilikát-hidrát)

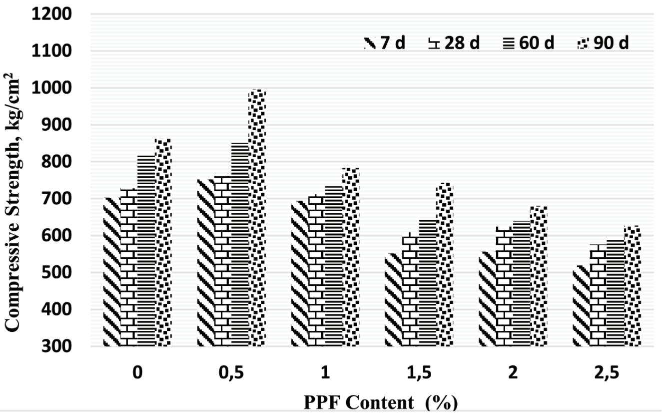

Fig. 5 Compressive strength of GGBFS-MK matrix with various ratios of PPF

5. ábra A GGBFS–MK mátrix nyomószilárdsága különböző PPF arányok mellett

3.3 Compressive Strength

The utilization of PPF as a reinforcing agent within alkali activated matrix presents significant advantages, suggesting that these plastics / polymers may be suitable for recycling and warrant evaluation for their potential applications in the construction and civil engineering sectors [7]. The composite material reinforced with 0.5 wt.% PPF exhibits a significant enhancement in compressive strength relative to the other samples. Conversely, the mechanical performance of the composites containing the highest concentration of polypropylene fibers was negatively impacted (>0.5 wt.%). This negative effect is most likely caused by the PPF heterogeneous distribution and agglomeration within the geopolymer mortar matrix. The less workability of geopolymer mortars would lead to the decrease in collision of the reactants which cause the formation of geopolymer. *Fig. 5* indicates that an increase in the percentage of PPF correlates with a decrease in mechanical resistance. This observation aligns with the reduced workability exhibited by the mixtures, which can be attributed to the minimal interaction between the nonpolar characteristics of PPF and the polar nature of the geopolymeric matrix.

3.4 Scanning Electron Microscopy

Fig. 6 shows Scanning Electron Microscopy pictures of the geopolymer both with and without PPF and revealing a notable presence of amorphous material. *Fig. 6/B* showed more compact matrix by the coupling of polypropylene fiber (0.5%) with geopolymer matrix compared with control mix (0.0%) *Fig. 6/A*. The dense and amorphous matrix is mainly associated with the geopolymeric structure (calcium silicate hydrate) that develops during the preparation of the geopolymer. By increasing polypropylene fiber content (2%) it seems that PPF did not undergo any chemical degradation and it didn't have a chemical bond with the aluminosilicate matrix *Fig. 6/C*. These results supported by similar studies [23, 24] which suggested that the morphology of PPF explained the low mechanical properties of geopolymers reinforced with. The observed low compressive strength in reinforced composites with a high content of PPF can be attributed to the influence of hydrophobicity and the contraction that takes place during the fiber-matrix interaction.

3.5 Drying Shrinkage

Numerous research investigations have demonstrated that the interaction of fiber content plays a crucial role in regulating the shrinkage of reinforced geopolymer composites [24]. *Fig. 7* shows the influence of PPF on controlling the shrinkage of GGBFS-MK geopolymer. PPF can bridge cracks and stop them from growing, and its incorporation, even in small amounts (0.5%), can greatly reduce the drying shrinkage of the composite specimens [25]. While, an adverse effect with the increase in fiber content to 2.5%. This can be attributed to the poor compactness of the geopolymer composition and these types of plastics polymers. Finally, the addition of PPF to geopolymer improves the uniformity of matrix and reduces the porosity.

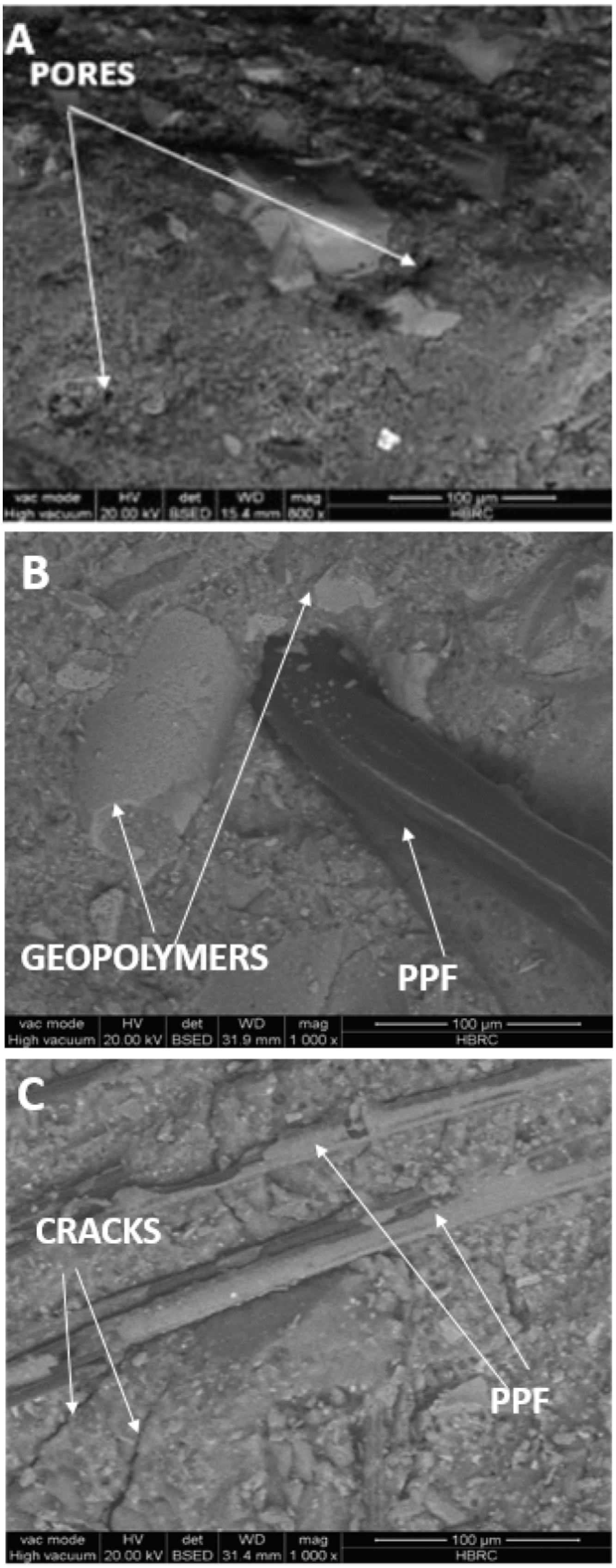

Fig. 6 *SEM of 90 days alkali- activated GGBFS-MK geopolymer containing PPF in varies ratios*
A: 0%, B: 0.5% and C: 2%
6. ábra *A 90 napos lúgosan aktivált GGBFS–MK geopolimer SEM-felvétele különböző PPF arányokkal*
A: 0%, B: 0,5%, C: 2%

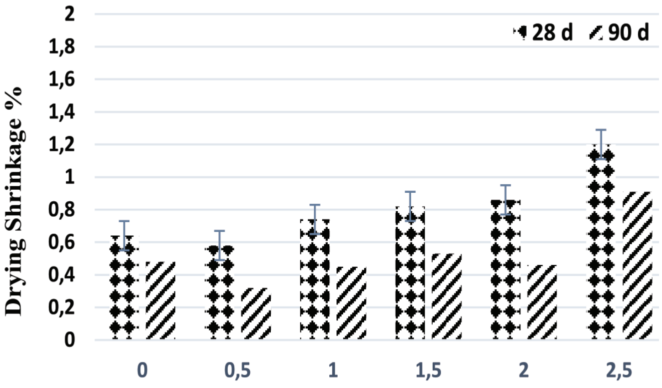

Fig. 7 *Drying Shrinkage of all mixes at 28 and 90 days of curing*
7. ábra *Az összes keverék száradási zsugorodása 28 és 90 napos érlelés után*

3.6 Thermal resistance

In this section, we are going to study the effect of firing on neat GGBFS-MK and GGBFS-MK coupled with 0.5% PPF (the optimum ratio). At 300, 500 and 800 °C for 2 hours with heating rate 5 °C/min. There are various parameters that will be monitored in this section: mass loss, firing shrinkage and residual compressive strength via strength change factor (SCF).

3.6.1 Mass loss

Generally, the increase in temperature of firing leads to an increase in mass loss percentage as known for all geopolymer matrices. Evaporation of free water and some chemically bonded water from geopolymers results in mass loss between room temperature and 300 °C, t might be linked to the hydration products degradation Al-OH, Si-OH, and Ca-OH groups dehydroxylate at 300–550 °C. More than 750 °C the mass loss is related to the carbonate species decomposition [26, 27]. The mass loss for control specimen was 5.05, 7.5 and 8.8% at 300, 500 and 800 °C respectively. *Fig. 8* shows that the mass loss of PPF paste was lower than that of control mix. At about 170 °C, PP fibers melt and create more voids, which help reduced the built-up internal vapor pressure. At 300 °C, the first weight loss occurs [28]. On the other hand, since the melting of higher amount of PPF led to higher amount of mass loss for the exposed temperatures of 500 and 800 °C, the weight loss for all pastes was nearly the same. Giving 4.45, 7.2 and 8.3% by mass loss reduction 12, 4, and 6% at 300, 500 and 800ºC respectively.

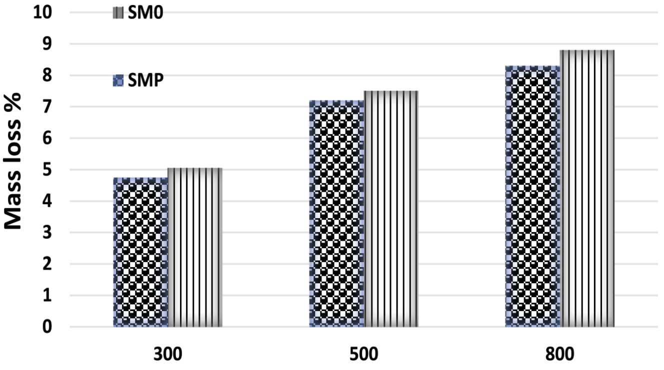

Fig. 8 *Weight loss% for mixes versus treatment temperatures*
8. ábra *Tömegveszteség (%) a keverékek esetében a kezelési hőmérsékletek függvényében*

3.6.2 Firing shrinkage

Behaviour of Cracking and smashing up the geopolymer could be noticed on account of contracted pores, free water evaporated from the pores leading to cracks propagation resulting in shrinkage correspondingly. The appearance of a considerable shrinkage leading to cracks. In the temperature range of 100 °C–300 °C, early shrinkage occurs because of dewatering. Up to 500 °C, the growth occurred continually, at a temperature of 800 °C, a notable size reduction was observed. The thermal shrinkage of geopolymers at 28 days of age exhibited an increase in response to rising temperatures, as illustrated in the accompanying *Fig. 9*. Average of firing shrinkage % can be calculated using the formula below:

$$F.S\ \% = (DL-FL)/DL*100$$

Where: DL for dried length and FL for fired length.

The Figure shows that the treated specimen had a lower shrinkage value than the control one. The thermal shrinkage was brought on by the removal of free water during the poly-condensation stage. Additionally, capillary tensions were created as a result of this process, drawing the particles together and producing more water as the temperature rose [29]. The significant increase in thermal shrinkage that happened when the geopolymer was heated to 800 °C is probably due to the formation of a new crystalline phase. At temperature 300 °C and 500 °C there was a slightly shrinkage increased, but at 800°C, there was a significant shrinkage increase. Finally, geopolymer treated with polypropylene fiber gives firing shrinkage 1.5, 2.4 and 9.14% by shrinkage reduction 53, 37 and 24% at 300 °C, 500 °C and 800 °C respectively.

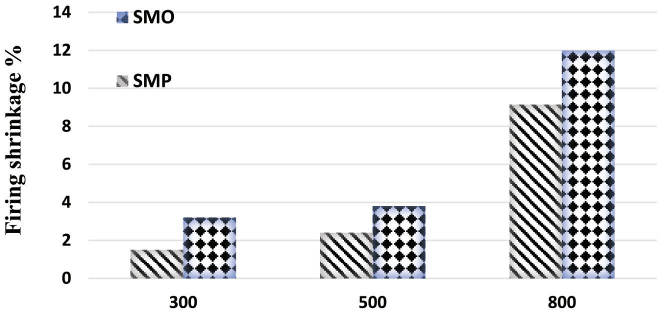

Fig. 9 *Firing shrinkage of all mixes versus temperatures 300, 500 and 800°C*
9. ábra *Az összes keverék égetési zsugorodása 300, 500 és 800 °C hőmérsékleteken*

3.6.3 Strength Change Factor (SCF)

Geopolymers formed from rich aluminosilicate materials offer an enhancement of the geopolymers strength at room temperature, but sometimes not when subjected to high temperatures. The strength of geopolymer paste depends on two contradictory processes, the damage process because of the thermal effect and the process of further geopolymarization. Transformation of amorphous geopolymer constituents into crystalline feldspars depending on the composition of the matrix. to calculate the average of Strength Change Factor, the equation below can be used.

$$SCF\ \% = (S_0-S_f)/S_0*100$$

Where: S_0 = strength after drying at 80°C and S_f = strength after firing. The strength change factor versus temperature is represented in *Fig. 10*. Control geopolymer gives 5.5, 7.4 and 31% at 300 °C, 500 °C and 800 °C respectively. The compressive strength of geopolymer treated with PPF were -8.33% (strength gain), 4.08 and 6.05% after heating to 300° C, 500 °C and 800 °C respectively. Firing at 300 °C resulted in increased geopolymerization reaction of the system thus the compressive strength increased slightly. In addition, Ju et al. [30] explained by Van der Waals force that the evaporation of free water at 300 °C caused the gel layer in matrix to come closer together which also led to the increase in strength of matrix. At high temperature the compressive strength reduced which may be due to the decomposition of calcium resulting in the deterioration of alkali-activated paste.

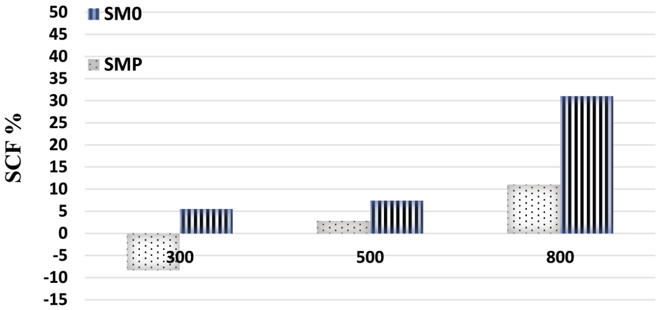

Fig. 10 *Strength Change Factor (SCF) % for mixes versus temperature temperatures 300, 500 and 800°C.*
10. ábra *A szilárdságváltozási tényező (SCF) % a keverékek esetében 300, 500 és 800 °C hőmérsékleteken*

4. Conclusions

This study courage using geopolymers for enhancing environmental safety and sustainability in construction through reduction of CO_2 emission and re-use of industrial by-products (waste management). Addition of PPF enhances the mechanical strength via bridging effect making it very appropriate to use in constructions that require high mechanical properties as bridges & dams.

Conclusion can be drawn according to the experimental results:

1. Addition of PPF enhanced the strength of geopolymer matrix the suitable content percent was 0.5% Where it recorded the highest compressive strength.
2. The addition of PPF reduced drying shrinkage of geopolymer matrix as compared with control mix.
3. Incorporation of PPF within the geopolymer paste enhanced thermal properties after firing of specimens at 300, 500 and 800 °C by measuring of mass loss, drying shrinkage and strength change factor

Conflicts of Interest

The authors declare no conflicts of interest.

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