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The Effect of Alkaline Activator Components on the Properties of Fly Ash Added Pumice Based Geopolymer

Cemal KARAASLAN^{1, 2*}, Engin YENER³

ABSTRACT: The effect of sodium hydroxide (SH) concentration and sodium silicate (SS) content in alkali activator on mechanical properties and wet-dry resistance of fly ash added pumice based geopolymer paste were investigated. In the study, the concentration of SH was used as 8, 10, 12 and 14 molar, while the weight ratio of SS to SH was used as 0, 1, 2 and 3. While the density of geopolymer paste samples increased significantly with the increase of SH molarity in mixtures with low SS content, it was less affected by SH molarity in mixtures with high SS content. For all SH molarity values, the compressive strength of the geopolymer paste samples increased with the increase of the SS/SH ratio, while a slight decrease was observed with the SS/SH ratio increasing from 2 to 3. However, with the increase of the SH molarity, the compressive strength of the samples was less affected by the SS content. As large cracks or splits occur in paste samples activated only with SH under the effect of wet-dry, their compressive strength could not be measured. The wet-dry performance of the paste samples activated with SS + SH was not affected much by the SH molarity and the SS/SH ratio and were close to each other. When looking at the microstructure studies, FTIR analyzes show that SS content provides a better geopolymerization, and SEM images show that micro-cracks relatively decrease with SS content.

Keywords: Geopolymer materials, durability, pumice, natural pozzolan, green concrete

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INTRODUCTION

The search for alternative binders has arisen due to the consumption of a lot of energy in the production of Portland cement (PC), which is one of the main components of concrete used as a building material, and excessive CO₂ emission to the nature as a result of these processes (Singh et al., 2015). Geopolymers (GP), one of the important alternatives to Portland cement, are obtained by activating powder binders with high Si and Al content with alkali hydroxide and alkaline silicate solutions (Atiş et al., 2015). Aluminosilicate sources such as fly ash, slag and metakaolin are generally used as powder binders (Duxson et al., 2007). Geopolymers produced with the specified powder binders and hot cured have excellent properties such as early high strength, low shrinkage, high freeze-thaw resistance and sulphate resistance (Davidovits, 1994).

Recently, the use of natural pozzolan (NP) as a powder binder in geopolymer production has increased significantly (Bondar et al., 2011c; Yadollahi et al., 2015; Haddad and Alshbuol, 2016; Vafaei and Allahverdi, 2016; Djobo et al., 2017; Kantarcı et al., 2019). While these studies show that natural pozzolan can be used as aluminosilicate sources in geopolymers, they also show that new studies should be done. It is emphasized that although the mechanical properties of NP based geopolymers have been investigated, their durability has not been studied much (Duxson et al., 2007; Djobo et al., 2016b, 2017; Firdous et al., 2018). In addition, decarbonization, which aims to reduce coal burning processes, limits the production of fly ash obtained by burning coal, increasing the importance of using NP that do not require any additional treatment (Firdous et al., 2018). The use of pumice, which is one of the NP, as a source material in geopolymer production is being investigated (Allahverdi et al., 2008; Kani et al., 2012; Yadollahi et al., 2014; Yadollahi et al., 2015; Safari et al., 2020).

In addition to all these, since NP has low reactivity, the mechanical properties and durability of natural pozzolan based GP binders are not as good as artificial pozzolan based GP binders (Djobo et al., 2017). Different methods are tried to increase the reactivity of NP based geopolymer binders. Aluminosilicate source calcining in NaOH environment, calcination at 700-900 °C and mechanical activation methods performed by extending the grinding time are some of these (Bondar et al., 2011b; Tchakoute et al., 2013; Djobo et al., 2016a). One of the effective methods that improves the strength and durability of NP based geopolymer binders by increasing their reactivity is the method of substituting another powder binder with high reactivity to NP (Bondar et al., 2011a; Kouamo et al., 2012; Risdanareni et al., 2016; Vafaei and Allahverdi, 2016). Within the scope of this study, fly ash added pumice based geopolymer (FAPGP) binder were produced by replacing fly ash with 25% pumice.

One of the most important parameters affecting GP binder properties is alkali activator type and content (Palomo et al., 1999). The alkaline activator is usually obtained from a combination of NaOH or KOH and Na₂SiO₃ or K₂SiO₃ solutions (Palomo et al., 1999; Hardjito and Rangan, 2005). In order to investigate the effect of alkali activator on FAPGP binder properties, the activator was prepared by mixing NaOH solution with different molarities with Na₂SiO₃ solution in different proportions. FAPGP paste samples were produced with these alkaline activators and their physical properties, compressive strength and wet-dry performance were determined. In addition, Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) analyzes were carried out to learn about the microstructure of the binder.

MATERIALS AND METHODS

Materials

Pumice used as a powder binder was obtained from Ağrı Mountain, Turkey. This pumice is used by local companies in the production of pumice briquettes. Coarse-grained pumice obtained from the

quarry was ground in a ball mill in order to increase its reactivity. Fly ash substituted for pumice was obtained from Çatalağzı Thermal Power Plant in Zonguldak, Turkey. When class C fly ash with high calcium (Ca) content, are used as the aluminosilicate source in GP binders, Ca can interfere with the geopolymerization process and disrupt the microstructure, so class F fly ash with low Ca content was used (Temuujin et al., 2009). The chemical contents of pumice and fly ash obtained by X-Ray Fluorescence (XRF) method are given in Table 1.

Table 1. Chemical composition of aluminosilicate sources

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃	LOSS
Pumice	3.56	67.49	13.07	3.98	0.69	2.63	2.36	0.18	3.69
Fly Ash	2.46	55.51	23.09	6.64	1.58	0.14	3.56	0.24	1.63

In Figure 1, X-Ray Diffraction (XRD) method analyzes of pumice and fly ash used in this study are given.

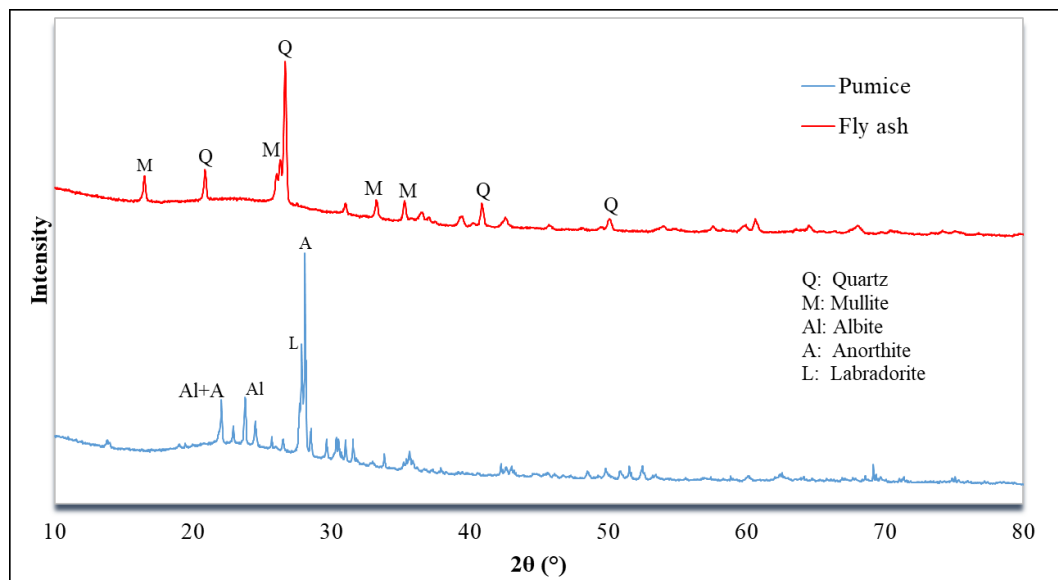


Figure 1. XRD analysis of pumice and fly ash

SEM images of powder binders used in this study show that fly ash has a spherical shape and the ground pumice has an irregular shape and angular structure (Figure 2). This suggests that in GP binders produced with pumice, the amount of alkaline activator required to wet the surface area of the powder binder will be higher.

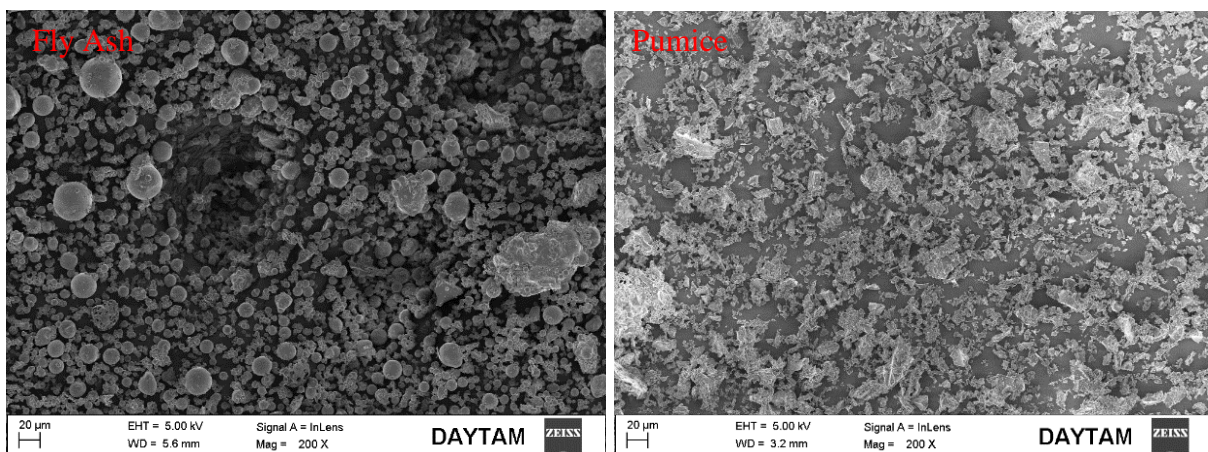


Figure 2. SEM images of powder binders

In the production of the geopolymer binder, NaOH (Sodium Hydroxide: SH) and Na₂SiO₃ (Sodium Silicate: SS) were used as alkali activators. Some physical and chemical properties of alkali activators are given in Table 2. Distilled water was used in the preparation of SH solution to eliminate possible side effects of impurities. (Hardjito et al., 2004).

Table 2. Some physical and chemical properties of alkaline activators

	SH (solid)	SS (liquid)
Molecular formula	NaOH	Na ₂ O.2SiO ₂
Molecular mass (g/mol)	40.01	182.15
Color	White	Gel (Colorless)
Density (g/cm ³)	2.13	1.39
Na ₂ O (%)	-	12.00
SiO ₂ (%)	-	24.50
H ₂ O (%)	-	63.50

Methods

Preparation of the ingredients for the mixture

The fly ash and the ground pumice were kept in the oven at 105 °C for 24 hours before mixing the GP paste. Solid SH particles in flake form were gradually added to distilled water to prevent overheating of the prepared solution. The values presented by Hardjito and Rangan (2005) were used in the preparation of SH solutions with different concentration values (Hardjito and Rangan, 2005). Accordingly, 262 grams of solid NaOH particles were used in the preparation of 1 kg of 8 M SH solution. And in the preparation of 1 kg of 10 M, 12 M and 14 M SH solutions, 314, 361 and 404 grams of solid NaOH particles were used, respectively. The SH solution, which was prepared by completing the mixing process, was mixed with the SS solution after waiting for 24 hours. SS-SH mixture was used after waiting for 24 hours in order for the two solutions to be completely dissolved in each other (Davidovits, 2002; Hardjito and Rangan, 2005).

Mixture proportions

In the study with 16 mixtures, four different molarity values (8 M, 10 M, 12 M and 14 M) were used for the SH solution. The ratio by weight of the SS solution to the SH solution (SS/SH) was determined as four different values (0, 1, 2 and 3). Fly ash has been replaced by 25% of pumice by weight. The ratio by weight of alkali activator to the total powder binder consisting of P and FA was determined as 0.45. The table showing the amount of use of all the ingredients in the mixture and the naming accordingly is given below (Table 3). The first digit used in naming the mixtures shown in Table 3 indicates the molarity of the SH solution, and the second digit indicates the weight ratio of the SS solution to the SH solution.

Mixing, placing and curing

The mixing process was started by mixing FA and P solids. Powder binders were mixed in the mixer for 5 minutes to ensure their homogeneous distribution. At the end of this period, the previously prepared SS + SH solution mixture was slowly added to the powder binder mixture and the mixing process was continued for another 5 minutes by mixing manually.

The prepared fresh GP paste mixtures were cast into 20x20x20 mm plastic cube molds. The samples placed in the mold were dropped 240 times on the jolting table to remove the entrapped air.

During curing, the molds are wrapped with stretch film to prevent evaporation of the alkaline activator in the mixture (Hardjito et al., 2004).

The prepared samples were left in the oven at 65 ° C without waiting. The samples were stored in the oven for 48 hours. At the end of this period, the samples were taken out of the oven, removed from the mold and kept in laboratory conditions until the testing day.

Table 3. Mixture proportions of samples

Mix name	SH (g)	SS (g)	FA (g)	Pumice (g)
8-0	54	0		
8-1	27	27		
8-2	18	36	30	90
8-3	13.5	40.5		
10-0	54	0		
10-1	27	27		
10-2	18	36	30	90
10-3	13.5	40.5		
12-0	54	0		
12-1	27	27		
12-2	18	36	30	90
12-3	13.5	40.5		
14-0	54	0		
14-1	27	27		
14-2	18	36	30	90
14-3	13.5	40.5		

Testing methods

In order to determine the water absorption and density of the geopolymer paste samples, on the 28th day, the weight of three cubic samples from each mixture was measured as A. The samples were then soaked in water for 24 hours and weighed in water as B. Finally, the saturated surface dry weight was measured in air as C. Water content values, as percentages, were calculated by Equation 1. Density values are calculated by Equation 2.

$$\text{Water absorption} = ((C-A) A^{-1}) \times 100 \quad (1)$$

$$\text{Density} = (A (C-B)^{-1}). \quad (2)$$

The 28-day compressive strength of the geopolymer paste samples was determined according to TS EN 12390-3 (EN). 28-day samples were used to determine the wet-dry performance. After the samples were left in tap water for 24 hours, they were removed from the water and allowed to dry in 48 hours under laboratory conditions. This 72-hour period was considered as 1 cycle. At the end of the 5th cycle, the compressive strength of the samples was determined. While the effect of wet-dry was investigated in some studies, samples were allowed to dry by keeping them in the oven at different temperatures after 24 hours of water soaking. (Slavik et al., 2008; Lemougna et al., 2014; Djobo et al., 2016b; Firdous et al., 2018). However, since it was thought that the temperature would create a new curing effect on the samples and thus, the GP binder would not show the real wet-dry behavior, it was preferred to allow it to dry for 48 hours at room temperature. The percentage loss in compressive strength after wet-dry cycles indicates its wet-dry performance. The average of 3 samples for each test was taken into account.

FTIR tests were carried out, in order to examine the effect of alkali activator to the structural changes and reaction products of FAPGP paste samples. Agilent Cary 630 model spectrometer device (650-4000 cm^{-1}) was used for FTIR analysis. The morphology and phase composition of FAPGP paste samples were scanned by SEM-EDS (Zeiss Sigma 300).

RESULTS AND DISCUSSION

Physical Properties of FAPGP Samples

Experimental results show that the density of FAPGP paste samples varies between 1.78-2.01 g/cm^3 . The change in density depending on the SS/SH ratio and the SH molarity is given in Figure 3.

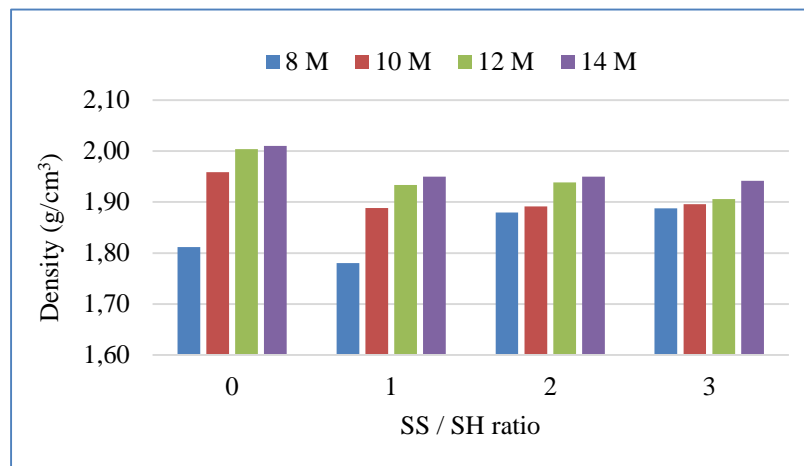


Figure 3. Density of the samples prepared with different concentrations of SH depending on the SS/SH ratio.

As can be easily seen from the Figure 3, the sample density increased with the increase of SH molarity for each SS/SH ratio. However, as the SS/SH ratio increases, the effect of SH molarity on density decreases. The probable reason for this is that the SH content in the alkali activator decreases with the increase in the SS/SH ratio, and thus the effect of the SH concentration is limited. Considering the water absorption of FAPGP paste samples, it is seen that the water absorption generally decreases with increasing molarity for a fixed SS/SH value (Figure 4). However, as can be seen in Figure 4, the water absorption of mixtures with low SS content (SS/SH = 1 and 2) is highly affected by molarity, while the water absorption of mixtures with high SS content is less affected by molarity.

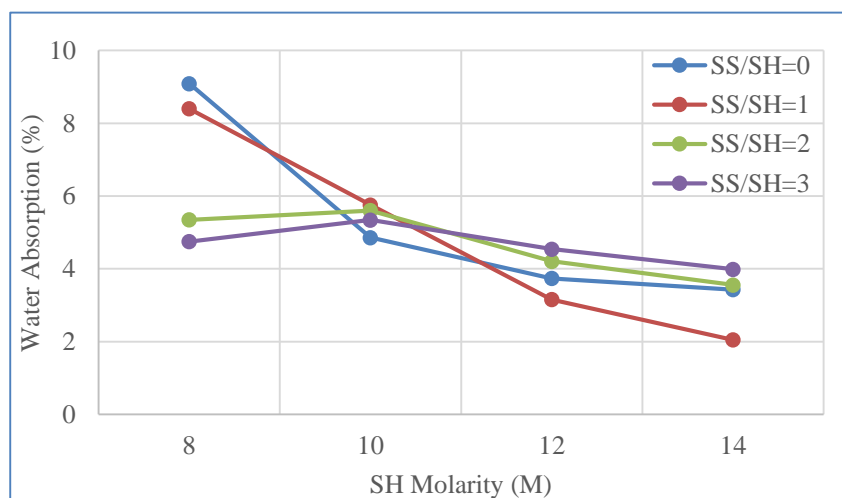


Figure 4. Water absorption of the samples prepared with different concentrations depending on the SS/SH

Compressive Strength of FAPGP Samples

Compressive strength tests were carried out in order to have information about the mechanical properties of the binder obtained in the study. Experiments have shown that the compressive strength of FAPGP paste samples is higher than the compressive strength of natural pozzolan (without additives) based geopolymers performed in other studies (Ndjock et al., 2017; Kantarcı et al., 2019). The change of compressive strength depending on the SH concentration of the samples with different SS/SH ratio is given in Figure 5. As can be seen from Figure 5, in all SH molarity values, while the compressive strength increases with the increase of SS/SH ratio up to 2, a slight decrease was experienced after this value. For the SH molarity values in this study, it can be said that the optimum SS/SH ratio is 2. The highest compressive strength was obtained with 74.8 MPa from the 8-2 sample. Only SH-activated samples had lower compressive strength values than SS-SH-activated samples. In order to obtain a good GP binder, it is recommended by other studies to use SS as an activator in addition to SH (Palomo et al., 1999; Xu and Van Deventer, 2000; Hardjito et al., 2004; Görhan and Kürklü, 2014; Nematollahi and Sanjayan, 2014). This situation is explained by the use of sodium silicate together with sodium hydroxide, which increases the amount of reaction product Si and consequently its strength increases (Criado et al., 2005).

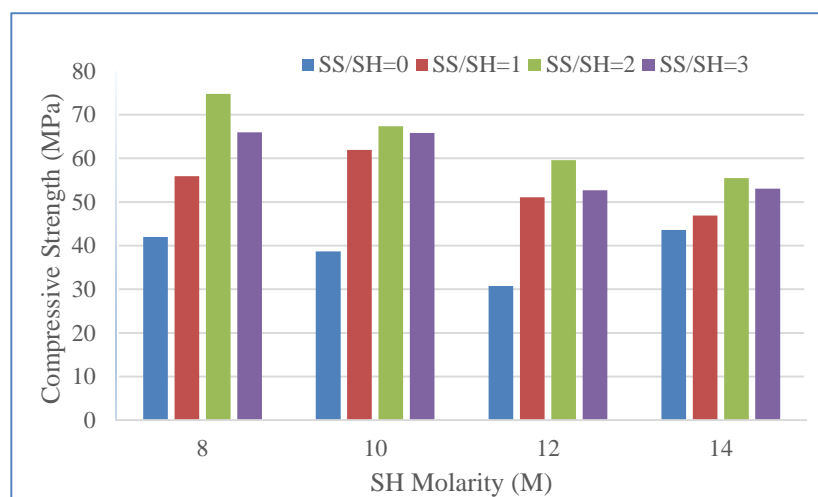


Figure 5. Change of compressive strength depending on SH molarity

As can be seen from Figure 5, when samples without sodium silicate were excluded, the compressive strengths of the samples prepared with 8 M and 10 M sodium hydroxide were higher than those prepared with 12 M and 14 M sodium hydroxide. Another remarkable point is that the compressive strength of the samples prepared with 14 M SH solution is relatively less affected by the SS content. This was also observed in a study conducted by Hardjito and Rangan (Hardjito and Rangan, 2005).

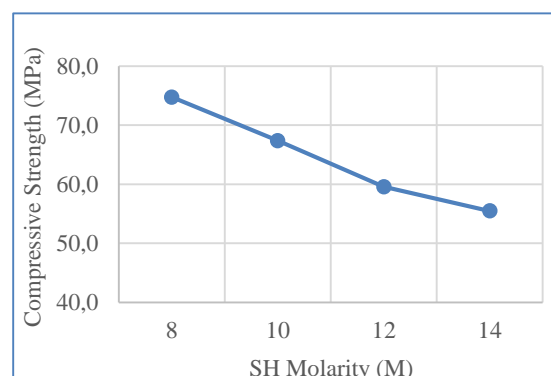


Figure 6. The change in the compressive strength of the samples with SS/SH ratio of 2

The compressive strength of mixtures using both solutions generally tended to decrease with increasing SH molarity (Figure 5). This trend is more clearly observed in mixtures with an SS/SH ratio of 2 (Figure 6). As can be clearly seen from Figure 6, the compressive strength has continuously decreased with the increase of the SH molarity. However, the compressive strength of mixtures with an SS/SH ratio of 1 increased a little when the SH molarity increased from 8 M to 10 M and started to decrease in the subsequent increases in the molarity value.

Wet-Dry Performance of FAPGP Samples

Large cracks were formed at the end of the first wet-dry cycle in samples activated with SH without using sodium silicate solution (SS). At the end of the second cycle, the cracks in the 12-0 and 14-0 samples expanded and caused the sample to split. Therefore, the compressive strength of these samples could not be measured. Although the 8-0 and 10-0 samples were not completely split, wide cracks occurred in the samples. This makes it necessary to use SS and SH solutions together as activators in order to obtain a more durable structure in pumice based GP binders.

The change in the wet-dry performance of the samples in which SS and SH solutions are used together, depending on the SS/SH ratio is given in Figure 7. As can be seen from the Figure 7, it can be said that the SS/SH ratio and SH molarity are not very effective on the wet-dry performance in the mixtures where SS solution is also used. Especially for mixtures with SS/SH ratio 1 and 2, the strength loss after wetting-drying is very close to each other around 42%.

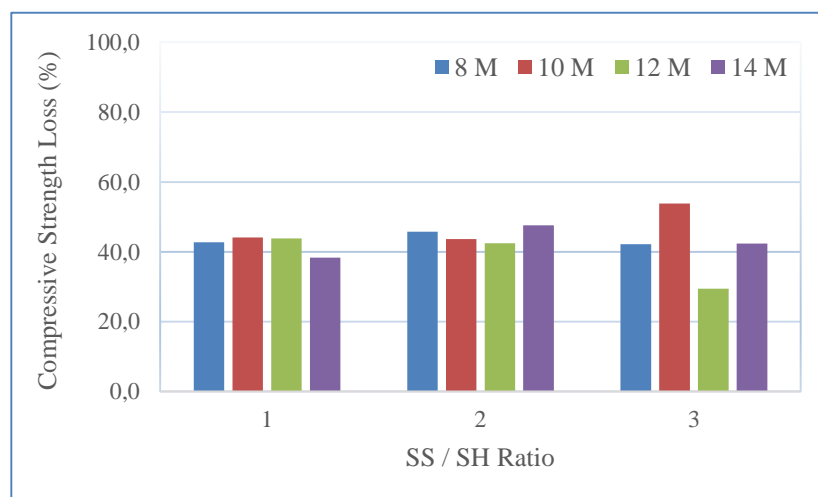


Figure 7. The change in the wet-dry performance of FAPGP pastes depending on the SS/SH ratio

The specified wet-dry performance (around 42%) can be considered a good value for natural pozzolan-based geopolymers such as pumice and tuff. In some studies, it is seen that the samples kept in water for 24 hours in natural pozzolan based geopolymers experience a loss of strength up to 89% (Lemougna et al., 2014; Djobo et al., 2017).

The mixture with the highest wet-dry performance for FAPGP paste samples was 12-3 with 29.5% strength loss. Although the compressive strength of the samples activated only with SH solution is at a reasonable level, their wet-dry performance is insufficient. For the values used in this study, it is seen that FAPGP binders produced with activators without sodium silicate will not be durable if exposed to water effects.

Results of FTIR Analysis

The results of FTIR tests conducted to examine the effect of alkali activator content on the geopolymerization process and reaction products of the prepared paste samples are evaluated below. For

molecular bond characterization, the absorption data of the test samples in the spectrum range of 650-4000 cm^{-1} were obtained using the Agilent Cary 630 model spectrometer device. Using these data, "Transmittance (%) - Wave number (cm^{-1})" graphics of the samples were drawn. Figure 8 shows FTIR spectrum graphs of samples produced in four different SS/SH (SS/SH: 0, 1, 2, 3) ratios using 10 M SH solution and 2 module SS solution.

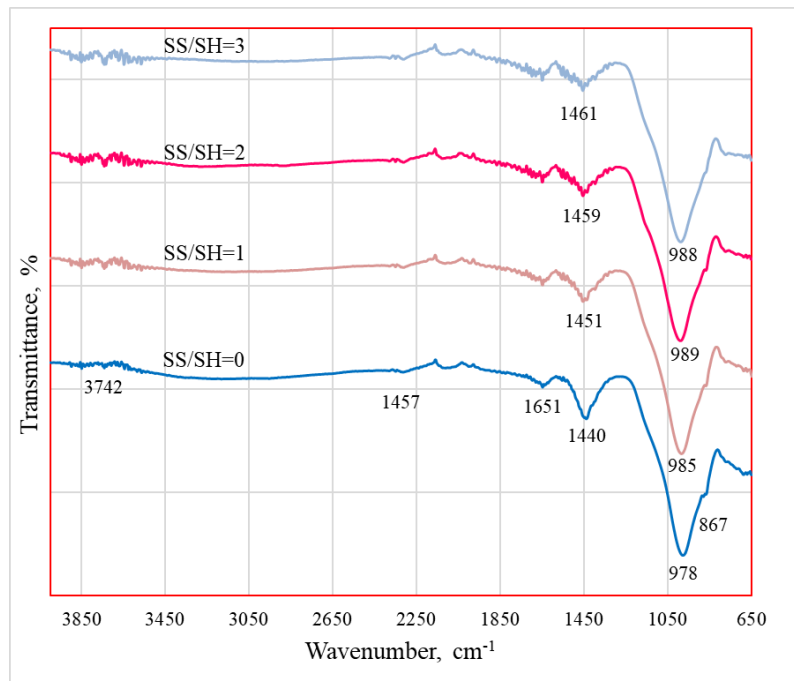


Figure 8. FTIR spectra showing the effect of SS/SH ratio on the molecular structure of geopolymers

The FTIR spectra of the SS/SH=0 sample, in which only 10 M SH solution was used as an activator, shows a wide but weak pit in the 2650-3450 cm^{-1} band. The presence of this wide pit becomes a straight line as the SS/SH ratio increases and disappears almost completely. This band indicates the presence of -OH (hydroxyl) groups in compounds that have not fully reacted in the geopolymer matrix (North and Swaddle, 2000; Zhang et al., 2012; Jansson et al., 2015). This indicates that the sodium silicate added to the starting alkali activator contributes to the advancement of geopolymerization. Spectrum peak in the range of 1440-1461 cm^{-1} are attributed to the asymmetric elongation vibration of the O-C-O functional groups (Bernal, 2016). Therefore, it is understood that the unbalanced alkali cations (Na^+) in the hardened geopolymer structure react with CO_2 in the air and form sodium carbonates (Na_2CO_3) (Bernal, 2016; Yener and Karaaslan, 2020). When only SH solution is used, it is seen in Figure 8 that the spectrum peak at the position of 1440 cm^{-1} shifts towards 1461 cm^{-1} and its amplitude decreases as the SS/SH ratio increases. It indicates that as the SS/SH ratio increases, the carbonation decreases. This decrease in carbonation level can be explained by the balancing of Na^+ cations by negatively charged AlO_4^- groups in the more coordinated geopolymer structure (Zhang et al., 2012; Bernal, 2016; Davidovits, 2017).

It is an important fingerprint of wide and deep spectrum geopolymer materials in the range of 900-1300 cm^{-1} and shows the asymmetric stress functional group of Si-O-T (T: Si, Al Tetrahedral) (Erdogan, 2015; Król et al., 2016; Petrus et al., 2019). The position of the peak value in this location has shifted from 978 cm^{-1} to 988 cm^{-1} with the increase in SS/SH ratio. This shows that geopolymerization progresses and the level of coordination in the SiO^n structure is increased by using SS in alkaline solution (Davidovits, 2008; Criado et al., 2012; Erdogan, 2015). The shoulder located in the 867 cm^{-1} band shows the Al-O structure, and the loss of clarity of the shoulder in this region and the decrease in the amplitude

of the peak indicate that AlO_4^- units take place more in the geopolymer network. As a result, SS used in alkaline solution contributes to the increase of the SiO^n coordination level, the inclusion of AlO_4^- structures in the geopolymer network, the balancing of Na^+ cations, the decreasing carbonation level and the development of a more durable geopolymer structure.

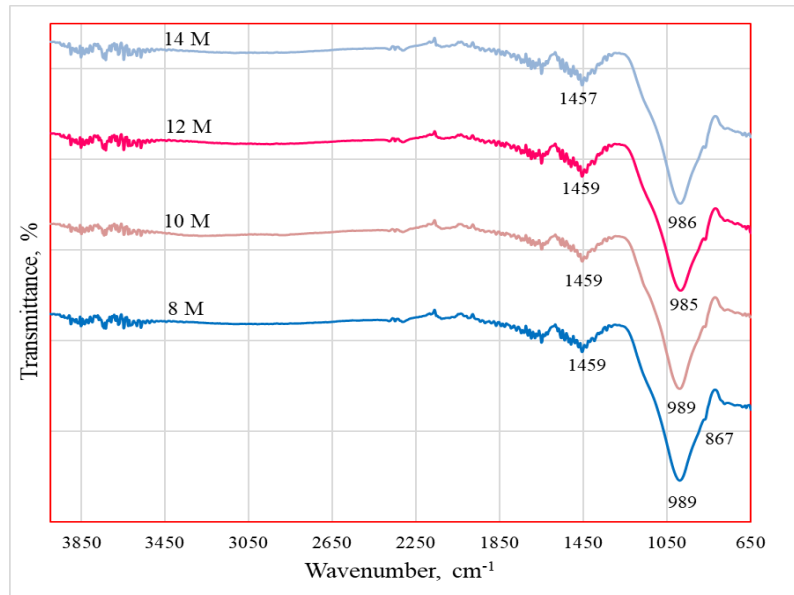


Figure 9. FTIR spectra showing the effect of SH molarity on the molecular structure of geopolymers

In Figure 9, FTIR spectra of the mixtures prepared at different molarity values at SS/SH=2 ratio are given. Figure 9 shows that the molecular structures are highly similar. When the peak around 1459 cm^{-1} is examined, it is seen that the amplitude of the peak decreases in case the sodium hydroxide solution molarity decreases, thus the carbonation level decreases a little. This may be related to the decrease in the amount of Na_2O in the starting solution in parallel with the decrease in NaOH molarity. When the spectrum peak in the wavelength range of $900\text{-}1300\text{ cm}^{-1}$ is examined, as increasing the SH molarity the wavenumber located of the spectrum peak is lowered. This situation can be attributed to the decrease in geopolymer quality due to the shortening of the dissolution time, which is the first stage of the geopolymer formation process. Because the increase in alkaline solution concentration causes shortening of the induction time required for dissolution from alumina-silica raw materials (Rees et al., 2007). When the shoulder band at 867 cm^{-1} wavelength is examined, it is seen that the increased molarity does not make a noticeable change in the studied range.

Microstructure of FAPGP Pastes

The SEM images show that the geopolymer paste has a heterogeneous structure consisting of unreacted or partially reacted powder binder particles, micro-cracks, voids and GP binder rather than a homogeneous material. Partly or completely unreacted powder binder particles were found in the GP matrix of all samples whose microstructures were examined (Figure 10 and 11). When the samples are viewed on a large scale, it is seen that the 10-2 sample has a denser structure than the others (Figure 10). Because of this dense structure, the compressive strength value of these samples was higher (Figure 5). Considering the chemical contents of the materials in the mixture, there are slight differences from the results obtained from the EDS analysis (Table 4). This is due to the differences in the regions where the image was taken (Tho-In et al., 2018). The heterogeneous nature of the samples mentioned earlier confirms this.

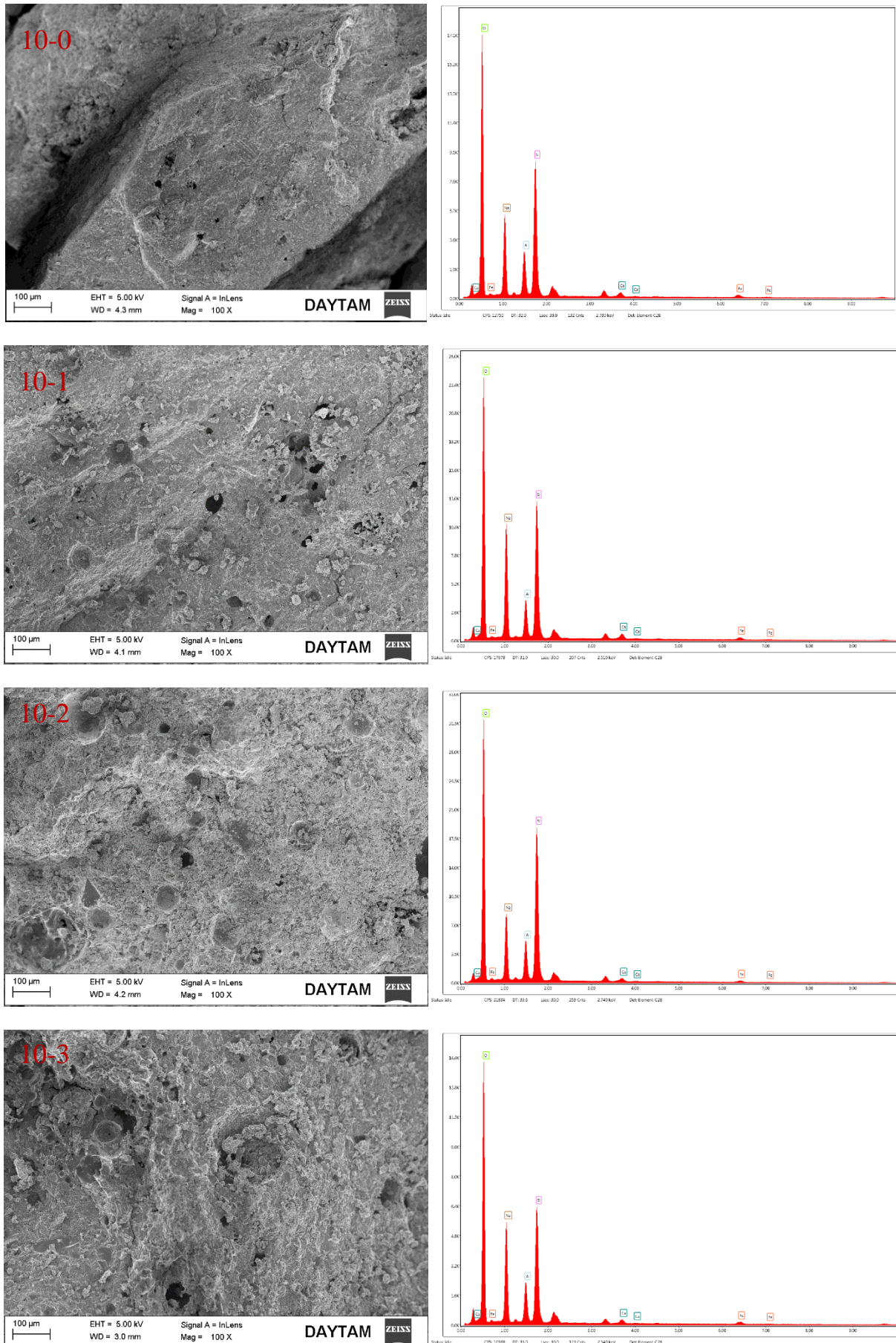


Figure 10. Selected-area and EDS analysis of FAPGP pastes with different SS/SH ratios.

Table 4. Percentages of elements obtained from EDS analysis

Element	10-0 (%)		10-1 (%)		10-2 (%)		10-3 (%)	
	weight	atomic	weight	atomic	weight	atomic	weight	atomic
O	45.74	58.87	42.96	55.53	47.26	60.21	45.01	57.63
Na	17.57	15.74	23.27	20.94	15.01	13.31	22.03	19.63
Al	7.47	5.70	5.73	4.40	6.68	5.04	6.54	4.97
Si	23.71	17.38	23.07	16.98	27.39	19.87	21.64	15.78
Ca	1.98	1.02	2.16	1.11	1.55	0,79	1.62	0.83
Fe	3.52	1.30	2.81	1.04	2.11	0.77	3.17	1.16

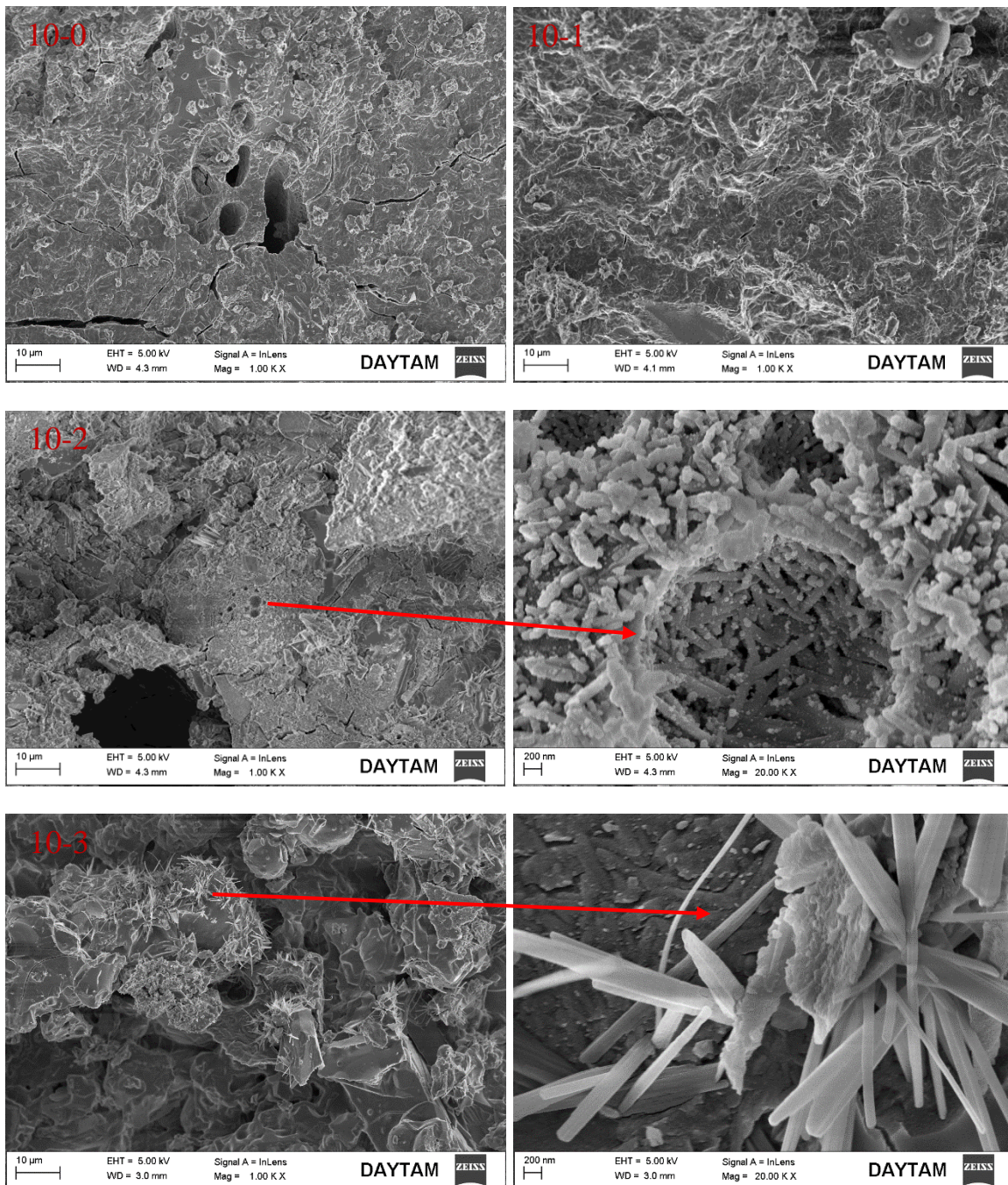


Figure 11. Micrographs of FAPGP paste samples prepared with 10 M SH solution

SEM images show that the GP binder products in which only SH solution is used as an activator is more homogeneous and that the products in the needle shaped crystalline structure increase with the increase of SS content (Figure 11). The 10-3 sample, in which needle-like structures were seen mostly, had a relatively more porous structure. In the sample 10-2, the inter-surface phase of fly ash and GP binder matrix is encountered in two regions of hemispherical surfaces (Jiang et al., 2020). Another case that needs to be stated is that although the reaction products of the sample activated only with SH are more homogeneous, it has larger micro-cracks than the samples activated with SS + SH.

CONCLUSIONS

The powder binder consisting of pumice and fly ash was activated with alkaline activators obtained by mixing SH with different molarities (8 M, 10 M, 12 M and 14 M) with SS solution in different proportions (SS/SH = 0, 1, 2 and 3). The effects of SH molarity and SS content on physical properties, compressive strength and wet-dry performance of the FAPGP binder were investigated. The following conclusions were drawn from the experimental investigation:

- The density of FAPGP pastes increased with increasing SH molarity. The amount of this increase decreased in mixtures with high SS content.
- Compressive strength increased with the SS/SH ratio increasing up to 2 for all SH molarity values in this study, and experienced a slight decrease with this ratio increasing to 3.
- The highest compressive strength value was obtained from the 8-2 mixture with 74.8 MPa. Solution mixtures in which the ratio of SS to 8 M or 10 M SH is 2 was evaluated as the optimum alkali activator.
- In order for FAPGP binders to be resistant to water effects, SS should be used in addition to SH in alkaline activator.
- With the presence of SS solution in the alkaline activator, geopolymerization improves and micro-cracks in the GP matrix decrease.

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Conflict of Interest

The article authors declare that there is no conflict of interest between them.

Author's Contributions

The authors declare that they have contributed equally to the article.

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