

**INVESTIGATION OF KAOLIN BASED ONE PART GEOPOLYMERS**Sezer ÇETİN<sup>1\*</sup>, Mehmet Uğur TOPRAK<sup>2</sup>, Seher GÜZ<sup>3</sup><sup>1</sup>Dumlupınar University, Hisarcık Vocational School, Land Registry and Cadastrate Pr., KütahyaORCID No : <http://orcid.org/0000-0002-8976-3594><sup>2</sup>Dumlupınar University, Faculty of Engineering, Department of Civil Engineering, KütahyaORCID No : <http://orcid.org/0000-0001-5483-2871><sup>3</sup>Dumlupınar University, Faculty of Engineering, Department of Metallurgy and Materials Engineering, KütahyaORCID No : <http://orcid.org/0000-0003-4926-8035>

Keywords	Abstract
Kaolin, Geopolymer, Aluminosilicate, Silica fume, Sodium hydroxide.	<i>Kaolin based one-part geopolymers (produced by adding water to preground aluminosilicate+alkali mixture) were produced in this study. The effect of silica fume addition and pre-curing time on microstructure, mechanical and mineralogical properties of geopolymers evaluated with X-ray Fluorescence (XRF), X-ray diffractometer (XRD), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectrometer (FT-IR). 7-day compressive strength of 66.73 MPa was obtained from kaolin-based one-part geopolymers. With the addition of silica fume, the strength enhanced up to 88.57 MPa.</i>

**KAOLİN ESASLI TEK BİLEŞENLİ JEOPOLİMERLERİN İNCELENMESİ**

Anahtar Kelimeler	Öz
Kaolin, Jeopolimer, Alüminosilikat, Silis dumanı, Sodyum hidroksit.	<i>Bu çalışmada tek bileşenli (beraber öğütülerek bekletilen aluminosilikat ve alkali karışımına su eklenerek üretilen) kaolin bazlı jeopolimerler üretilmiştir. Silis dumanı eklenmesinin ve ön bekleme süresinin jeopolimerlerin mikroyapısına, mekanik ve mineralojik özelliklerine etkileri X-ışını kırınımölçeri (XRD), X-ışını Floresans (XRF), Taramalı Elektron Mikroskopu (SEM), Fourier Dönüşümlü Kızılötesi Spektrometre (FT-IR) kullanılarak incelenmiştir. Kaolin bazlı tek bileşenli jeopolimerlerden 66,73 MPa 7 günlük basınç dayanımı elde edilmiştir. Silis dumanı eklenmesi ile dayanım artarak 88,57 MPa'a ulaşmıştır.</i>

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**1. Introduction**

Victor Glukhovsky, in the 1960s and 1970s made major contribution in identifying both calcium silicate hydrates, and calcium and sodium aluminosilicate hydrates as solidification products in geopolymerization (Davidovits, 2020).

Davidovits began to work on new heat-resistant “plastic materials” after the catastrophic fires in France in 1970/73. Davidovits stated that these requirements can only be provided by inorganic polymer materials and reported that a mineral such as kaolin may form a binder by alkali activation at a temperature as low as 100-150 °C, instead of heat treatment at 1000-1300°C. He introduced the term of geopolymer in 1978 (Davidovits, 1988). Davidovits has defined Si–O–Al bond as sialate bond and Si–O–Si bond as siloxo bond for aluminosilicate structures and this aluminosilicate structures known as “inorganic polymers”, “mineral polymers” (Davidovits, 1982). If Si/Al molar ratios are 1, 2 and 3, these structures respectively defined as poly(sialate), poly(sialate-siloxo) and poly(sialate-disiloxo) (Davidovits, 1976).

The geopolymer is an amorphous semi-crystalline structure with a three-dimensional Si–O–Al polymeric network. Molecular sequences of geopolymer systems with different Si/Al ratios and share of all oxygen atoms of SiO<sub>4</sub> and AlO<sub>4</sub> tetrahedrals shown in Figure 1 (Palomo, Grutzeck and Blanco-Varela, 1999; Provis, Lukey and van Deventer, 2005).

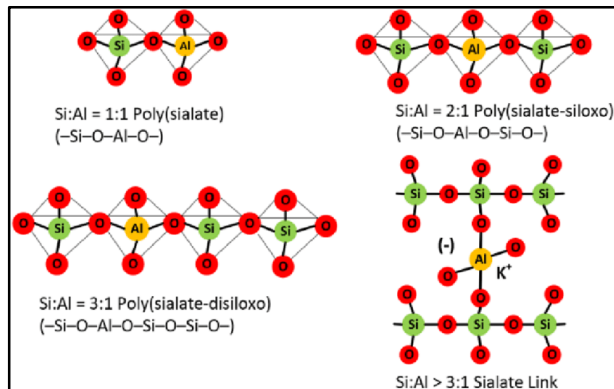


Figure 1. Molecular Sequences of Geopolymer Systems (Al Bakri Abdullah, Liew, Heah and Mohd Tahir, 2018)

In quadruplex coordinated Al, negative charges are balanced with cations such as Na<sup>+</sup>, K<sup>+</sup>, Li<sup>+</sup>, Ca<sup>+2</sup>, Ba<sup>+2</sup> and H<sub>3</sub>O<sup>+</sup> (Allahverdi, Mehrpour and Kani, 2008)

Geopolymerization is an exothermic reaction and forms three-dimensional macro molecular structures through oligomers. The structure proposed by Davidovits is given in Figure 2 (Davidovits, 1991) (Davidovits, 1994).

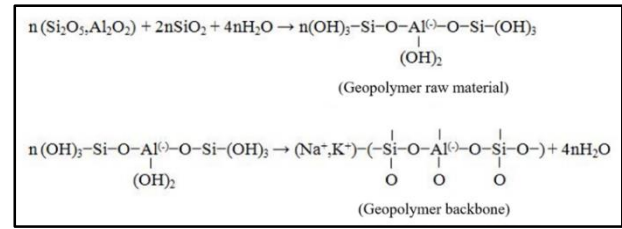


Figure 2. Schematic Representation of Geopolymerization Process

The presence of hydroxyl ions in the alkaline medium initiates the dissolution process of the aluminosilicates and liberates the silicate and aluminate species to promote further-polymerization reaction (Komnitsas and Zaharaki, 2007) (Davidovits, 1988). The degree of dissolution depends on fineness, ion-exchange ability, concentration of alkali solutions and the structure of the precursors. It has been suggested that the geopolymerization reactions occur simultaneously and in multiple steps (van Jaarsveld, van Deventer and Lukey, 2002), (Alonso and Palomo, 2001), (Dimas, Giannopoulou and Panias, 2009).

Geopolymers are divided into two according to the production method. First is conventional (two-part) geopolymers that formed by a reaction between a concentrated aqueous solution of alkali hydroxide, sulfate, silicate, or carbonate. (Provis and van Deventer, 2009) (Duxson, et al., 2007) (Provis, 2014) (Provis, et al., 2014) (Provis, 2009).

Second one is one-part geopolymers that demonstrate significant point on geopolymer technology having been described by the first time in 2007 (Koloušek, Urbanova, Andertova, Hulinsky and Vorel, 2007) (Shi, Fernández-Jiménez and Palomo, 2011). In one-part geopolymers, solid activators such as calcium hydroxide, sodium hydroxide, calcium carbonate, or sodium metasilicate are blended with geopolymer precursors like rice husk ash, silica fume, or fly ash, then just water is added to initiate the geopolymerization. The dissolution process of the solid phase starts rapidly after adding water to the dry mixture (Abdollahnejad, Pacheco-Torgal and de Aguiar, 2015) (Ma, Long, Shi, & Xie, 2018).

Researchers have studied on one-part geopolymer system because of its economic value, whereby geopolymer compound can be prepared by just adding water to aluminosilicate-alkali mixes (Sturm, Greiser, Gluth, Jäger and Brouwers, 2015) (Ke, Bernal, Ye, Provis and Yang, 2014) (Peng, Wang, Shen and Xiao, 2015). With this approach, it is not necessary to use highly alkaline compound or sodium hydroxide solutions which prevent problems with carbonation and ageing of these solutions as well as safety issues. (Hajimohammadi, Provis and van Deventer, 2008). But

recent investigations (Peng, Wang, Shen and Xiao, 2015) support that one-part geopolymers show low mechanical strength.

Usually, the method for producing metakaolin consists in heating kaolin between 700 and 850 °C, giving an amorphous material. Then it contains exclusively 50–55% of SiO<sub>2</sub> and 40–45% of Al<sub>2</sub>O<sub>3</sub> by weight and is very reactive with respect to an alkaline and calcium-rich solution (Konan, et al., 2009) (Poon, Lam, Kou, Wong, and Wong, 2001) (Shvarzman, Kovler, Grader, and Shter, 2003).

The aims of the present study are i) to evaluate usability of kaolin as raw material in geopolymers ii) to determine the effect of silica fume (SF) on properties of geopolymers. For this purpose, mechanical, mineralogical, and microstructural analyses were conducted on geopolymer samples.

## 2. Materials and Methods

In this study, research and publication ethics principles were followed.

Table 1.

Chemical Composition of Kaolin and Silica Fume

Composition	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	SO <sub>3</sub>	MnO	LOI
w % of K	47.05	37.07	0.83	0.31	0.08	1.90	0.07	0.12	0.06	0.04	-	13
w % of SF	92.41	0.74	0.86	1.15	0.43	1.09	0.41	0.23	-	0.21	0.07	2.36

LOI: Loss on Ignition

## 2.2. Methods

Geopolymer samples were produced by one-part production method described below. Composition and production details of geopolymer series were given in Table 2.

One-part geopolymer(OPG): aluminosilicates (K or K+SF) and the alkali (NaOH pellets) were ground together with ring mill (Figure 3) for 30 minutes. After pre-cure in plastic bags for 1 or 2 day in ambient conditions, ready mix was only mixed with distilled water and pressed in 25 mm cylindrical mold (Figure 4.a) under 12 tons with hydraulic press (Figure 4.b).

Studies have indicated that pre-cured samples show some minor cracks compared to open cured samples without pre-curing. During pre-curing, the binder gains some strength. At a low temperature such as room temperature, the water evaporation and hence the capillary forces are not too high. Therefore, crack

## 2.1. Materials

The Blaine fineness of kaolin (K) used in this study was 5000 cm<sup>2</sup>/g. XRD patterns reveal that SF was completely amorphous and its specific surface area (measured with the BET method) typically ranges from 150000 to 300000 cm<sup>2</sup>/g. (Yeğınobalı, 2009) (Tonak, Sipahi and Atay, 1997) (Aldred, et al., 2000).

Ye et al. (2016) stated that the addition of silica fumes up to 20–25% clearly has a positive effect on the compressive strength of the one-part geopolymers. This is because silica fume, which has small particle sizes and consists of a high amount of amorphous silicon dioxide, is a highly reactive pozzolanic material (Jithendra and Elavenil, 2020).

In this study, 8.48 wt% of SF with respect to the mass of total solid were used to produce different geopolymer precursors. Sodium hydroxide pellets (98% purity) was obtained from a local market in Eskişehir/TURKEY.

Chemical composition of K and SF obtained by using an X-ray Fluorescence Spectrometer (Panalytical Axios Spectrometer) at Dumlupınar University İLTEM department were given in Table 1.

formation is prevented. After pre-curing, the binder acquires enough strength to withstand capillary forces and gains its ultimate strength without too many cracks (Peys, Rahier and Pontikes, 2016). For this reason, the effect of the increase in pre-curing time on compressive strength was investigated.



Figure 3. Ring Mill

Steam Cure (SC) was applied to one-part geopolymer samples in the digital water bath shown in Figure 5 (a)

and the samples (25x50mm cylinder) obtained were given in Figure 5 (b).

Table 2.

Composition and Production Data of Geopolymer Series

Sample Code	K, g	SF, g	NaOH, g	W, ml	Pre-curing, d	Curing Type	Curing Temp. (°C)	Curing Time. (h)
K-1	88.90	-	11.10	5	1	SC	60	5
K+SF-1	80.42	8.48	11.10	5	1	SC	60	5
K-2	88.90	-	11.10	5	2	SC	60	5
K+SF-2	80.42	8.48	11.10	5	2	SC	60	5

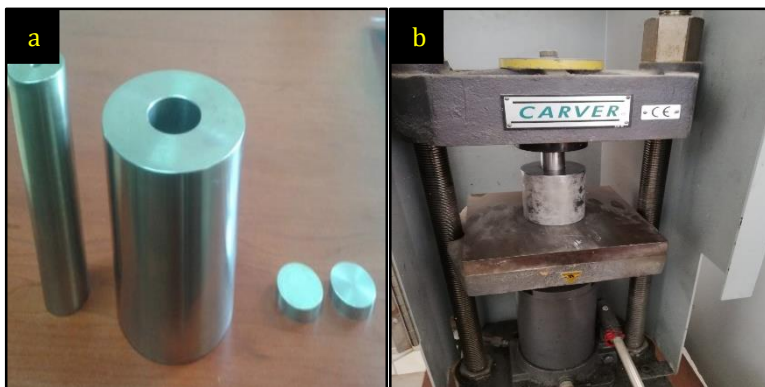


Figure 4. (a) 25 mm Cylindrical Mold (b)Hydraulic Press

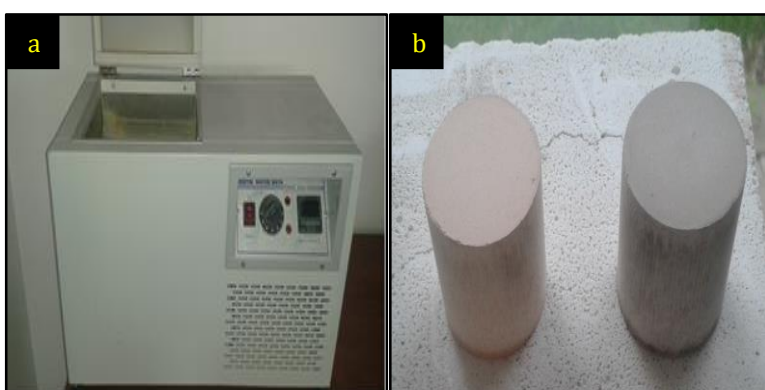


Figure 5. (a) Digital Water Bath Used for Steam Cure(b)One-Part Geopolymer Samples

### 2.3. Characterization

After steam curing, geopolymers were stored at an ambient temperature up to testing time. The loading speed of the uniaxial compression test device used in

the test should be selected as a constant loading speed between 0.2 MPa/sec - 1.0 MPa/sec with respect to TS EN 12390-3. Therefore, compressive strengths of the geopolymer series were measured at 7 d. with uniaxial compression test device under loading speed of 0.6

MPa/sec. All the reported results are the means of three samples. Phase analysis of raw kaolin and one-part geopolymer samples (K-2 and K+SF-2) were obtained by using Panalytical Empyrean XRD device at Dumlupinar University İLTEM department with a scanning speed of 1 ° in a minute between 5–70 °. FT-IR spectroscopy was performed between 400–4000 cm<sup>-1</sup> wave number using Bruker Alpha brand FT-IR device for K-2 and K+SF-2. Zeiss Supra 50VP scanning electron microscope (SEM) was used for SEM microstructure analysis of K+SF-2 (the highest compressive strength).

### 3. Results

#### 3.1. Compressive Strength Analysis

SF addition has positively affected the compressive strength of geopolymers as expected (Figure 6). Extending the pre-cure time up to 2 days almost doubled the compressive strength of the 1-day pre-cured geopolymer samples.

A study conducted in the literature, the 3-day compressive strength of calcined kaolin with amorphous structure and high reactivity found 63 MPa at most. In this study, calcined kaolin and NaOH or Na<sub>2</sub>CO<sub>3</sub> were used (Peng et al., 2015). However, in our study, 7-day compressive strength was obtained with a maximum of 66.73 MPa with a mixture of uncalcined kaolin and NaOH, and this value was increased up to 88.57 MPa with silica fume additive.

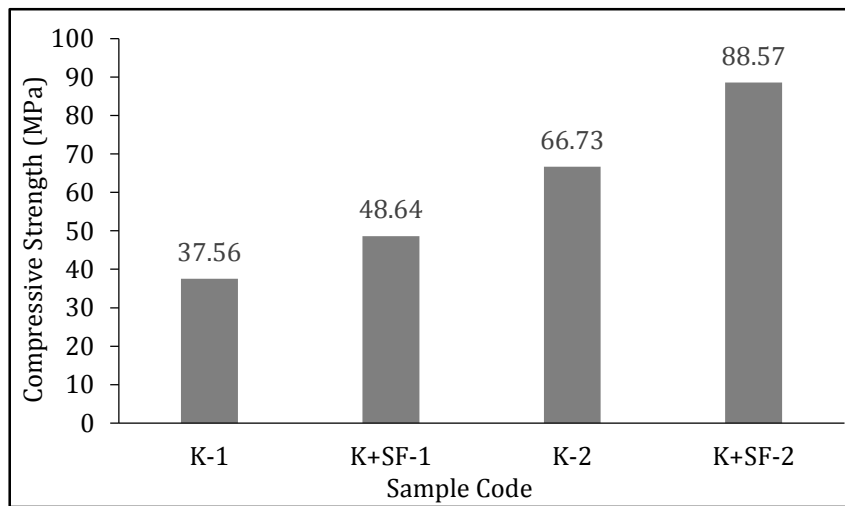


Figure 6. 7-Day Compressive Strength of Geopolymer Samples

#### 3.2. XRD Phase Analysis

Kaolinite, anorthite, stishovite and illite were obtained in kaolin. Kaolinite peaks are observed at 2θ (degrees); 12.3° - 24.9° - 45.4° - 55.1° - 62.2°. The intensities of kaolinite and illite decreased in the XRD pattern of K-2 and K+SF-2 samples in consequence of geopolymerization. Geopolymers show generally amorphous X-ray diffraction (XRD) pattern without sharp peaks at 2θ between 25 and 30° (Lecomte, Liégeois, Rulmont, Cloots and Maseri, 2003).

As the Si/Al ratio in K+SF-2 increased, intensity of peaks at the 2θ range from 20° to 25° decreased as well as the amorphous pattern considerably disappeared. Increasing Si/Al ratio increases the angles of sharp peaks (Lizcano, Kim, Basu and Radovic, 2012).

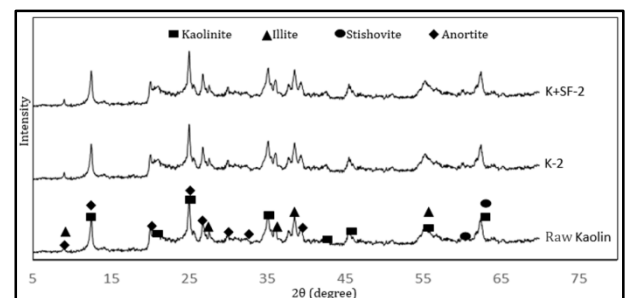


Figure 7. XRD Patterns Obtained from Test Samples

#### 3.3. FT-IR Analysis

FT-IR analysis was performed to support geopolymer formation. In the FT-IR spectra of geopolymers, Milkey observed the relationship between the frequency of the

absorption bands and the Si:Al ratio in the aluminosilicate frame. According to this, as the amount of Al increases, the wavenumber decreases (Milkey, 1960). In the FT-IR analysis shown in Figure 8, the bands in the 3600-3800  $\text{cm}^{-1}$  wave number region are caused by hydration of water connected to the structure.

Peaks around 690  $\text{cm}^{-1}$  wave number indicate Si-O symmetrical vibration band. Peaks around 565  $\text{cm}^{-1}$  wave number indicate Si-O-Si or Si-O-Al symmetrical strain. Si-O-Si or Si-O-Al network structure is observed at peaks around 460  $\text{cm}^{-1}$ . Inorganic polymers consist of Si-O tetrahedra. These tetrahedras are connected to each other by oxygen bridges at their corners. Considering the FT-IR analysis, peaks around 1030 and 1000  $\text{cm}^{-1}$  wave number is taken to 900  $\text{cm}^{-1}$  wave number with heat treatment and geopolymerization. This withdrawal is indicative of structural changes. FT-IR spectra of geopolymers showed similar wavenumber with different intensities regardless of silica fume addition. The main peak analyzed in the FT-IR spectrum of geopolymers was in the region of 900-1250  $\text{cm}^{-1}$  corresponding to the Si-O-Al or Si-O-Si linkages. K+SF-2's main peak was lower than K-2's main peak.

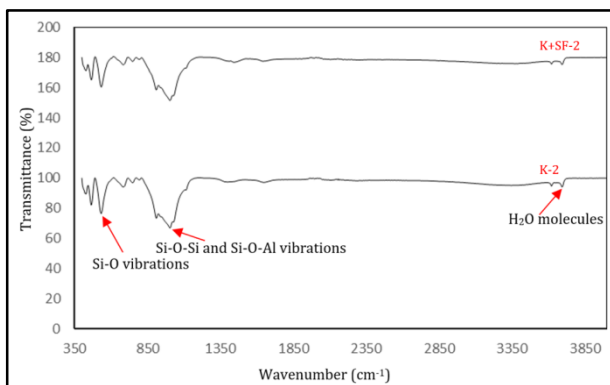


Figure 8. FT-IR Analysis Obtained from Test Samples

### 3.4. SEM Microstructures and EDX Spectrum Analysis

Figure 9-10 showed the microstructure of the geopolymers at different magnifications. EDX (Energy dispersive X-ray) analysis of the study were given in Figure 11-15. The rod structures seen in Figure 9-10 are insoluble sodium hydroxide crystals. The weight ratio (%) of sodium in the structure at the selected areas was 29.86% which is the average of the spectrum 1-5. The reason of the high sodium ratio was insoluble sodium hydroxide crystals given in Figure 9-10.

Sodium ratio in geopolymer structure in the spectrum 2 region shown in Figure 12 was only 6.53 %.

SEM analysis shows that the geopolymer region is much denser than the insoluble alkali region. Joints at the grain boundaries and dense structure are manifested by the increase in compressive strength values in mechanical test results. It was determined by EDX analysis that sodium aluminosilicate structures were formed as a result of geopolymerization and the best results were observed in the samples produced by SF addition.

The EDX analysis of one-part geopolymer sample showed major element of Si, Al and Na. The physical and mechanical properties of geopolymer were a function of  $\text{SiO}_2/\text{Al}_2\text{O}_3$  and  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  ratios (Sathonsaowaphak, Chindaprasirt and Pimraksa, 2009).

The geopolymer sample with the respectively  $\text{SiO}_2/\text{Al}_2\text{O}_3$  ratios of 1.53, 1.96 and the  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  ratios of 0.37, 1.37 (as showed in Fig. 12 and 14) verified with reported of SEM images which the presence of strong geopolymer.

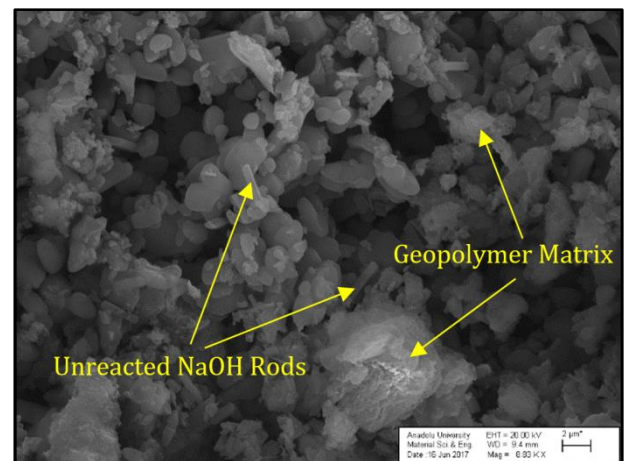


Figure 9. SEM Images Of K+SF-2 Sample

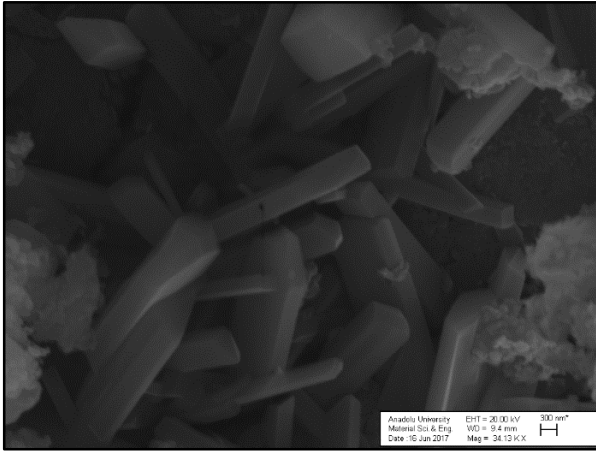


Figure 10. SEM Images Of K+SF-2 Sample at Different Magnifications

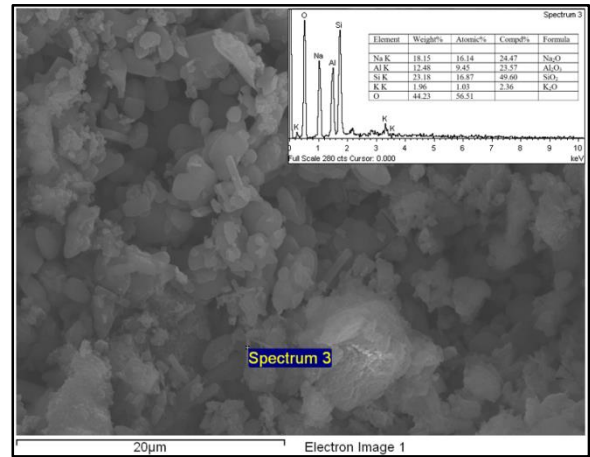


Figure 13. Microstructure Image and EDX Analysis from Different Point Of K+SF-2 Sample

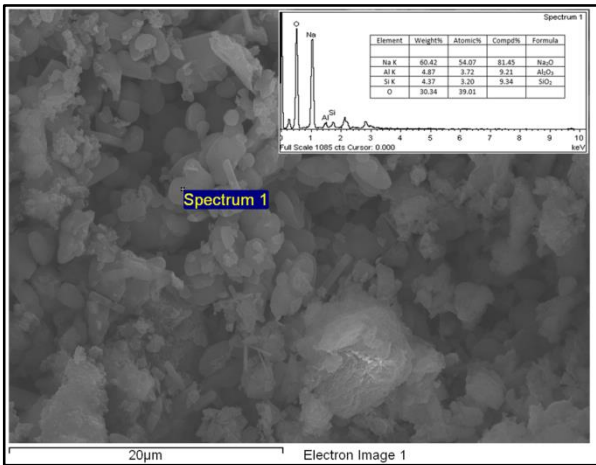


Figure 11. Microstructure Image and EDX Analysis K+SF-2 Sample

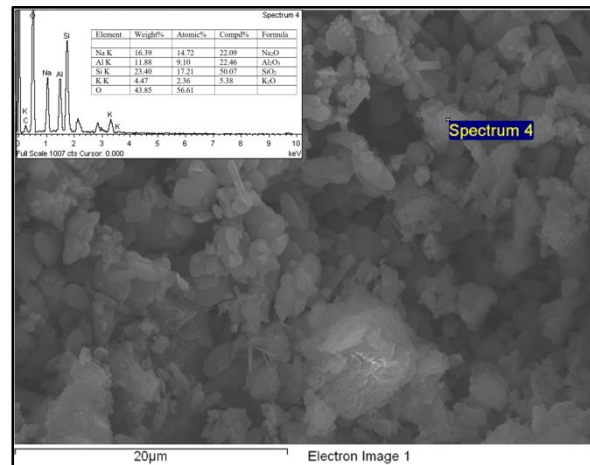


Figure 14. Microstructure Image and EDX Analysis from Different Point Of K+SF-2 Sample

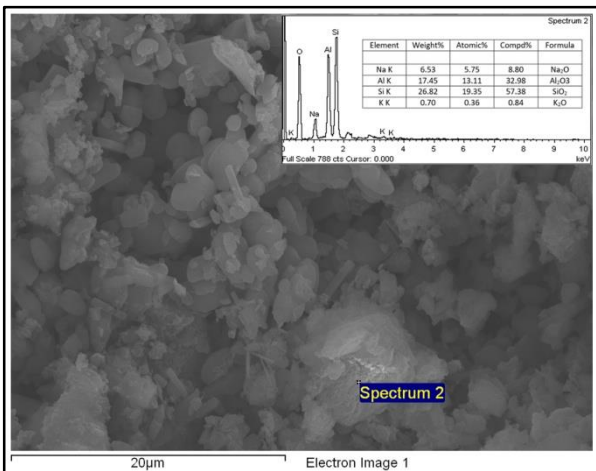


Figure 12. Microstructure Image and EDX Analysis from Different Point Of K+SF-2 Sample

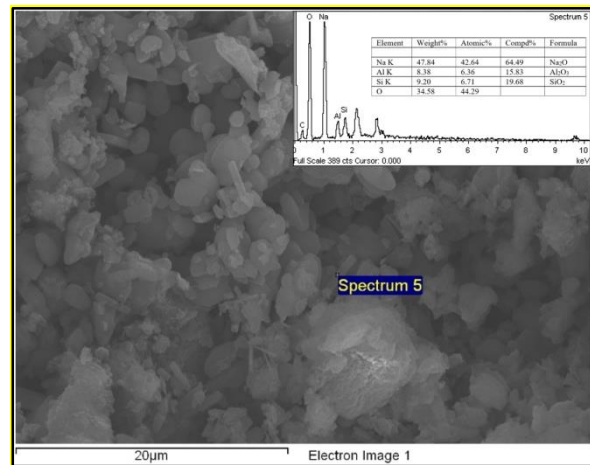


Figure 15. Microstructure Image and EDX Analysis from Different Point Of K+SF-2 S

#### 4. Conclusion

Results of this experimental study can be summarized as follows:

- The interatomic bonds examined in the FT-IR analysis reveals geopolymer formation.
- The SEM observations supports the presence of geopolymer bonding at the grain boundaries.
- High strength one-part geopolymers were obtained using kaolin as aluminosilicate source.
- Kaolin can be preferred for geopolymer production considering economic and ecological reasons instead of metakaolin generally used for geopolymer production.
- 37.56 MPa compressive strength was obtained in the sample K-1 containing kaolin + NaOH + water, which was pre-curing for 1 day, but 66.73 MPa compressive strength was obtained in the sample K-2 containing kaolin + NaOH + water for 2 days.
- 48.64 MPa compressive strength was obtained in the sample K+SF-1 containing kaolin + Silica Fume + NaOH + water, which was pre-curing for 1 day, but 88.57 MPa compressive strength was obtained in the sample K+SF-2 containing kaolin + Silica Fume + NaOH + water for 2 days.
- In a kaolin based one-part geopolymer a decrease in the reaction rate was observed as the ratio of  $\text{SiO}_2/\text{Al}_2\text{O}_3$  increased. In addition, the increase in the ratio of  $\text{SiO}_2/\text{Al}_2\text{O}_3$  due to the addition of silica fume caused the formation of silica-rich phases and increased the amount of unreacted silica.
- Silica fume addition enhanced geopolymerization and the compressive strength of geopolymers.

The study was produced from the first author's thesis. Therefore, the article does not require legal / special permission (Çetin, 2017).

#### Author Contributions

In this article; Sezer ÇETİN, contributed to the concept, designed the research, experimental design, experimental work, analysis of results, discussed the results and reviewed the manuscript. Mehmet Uğur TOPRAK, contributed to the concept, designed the research, analysis of results, discussed the results and reviewed the manuscript. Seher GÜZ, contributed to

experimental work, analysis of results, discussed the results and reviewed the manuscript.

#### Conflict of Interest

There is no conflict of interest.

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