



Research article

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Mechanical properties of ceramic powder based geopolymer mortars

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Keywords: mechanical properties, mortar, geopolymer, ceramic powder, binder, optimization

Abstract. In this study, the properties of geopolymer mortars produced by activating ceramic powder with Sodium Hydroxide (NaOH) and Sodium Silicate (Na_2SiO_3) were investigated. Activator mixture was prepared by mixing NaOH containing 11 %, 13 % and 15 % sodium in proportion to the binder weight and Na_2SiO_3 in different proportions. The silicate modulus of the activator mixtures were set at ratios ranging from 0 to 0.3. Geopolymer samples were prepared by mixing ceramic powder with activator, water and sand in a standard cement mixer. After fresh mortar mixtures were placed in the molds, they were cured at 105 °C for 24 hours. The samples were taken out of the molds after curing. They were kept at room temperature for up to 28 days. Then, the samples underwent unit weight test, apparent porosity, water absorption ratio, ultrasound pulse velocity, flexural and compressive strength tests. As a result of the tests, compressive strength between 10.42 MPa and 41.53 MPa, flexural strength between 2.34 MPa and 10.38 MPa were determined. Optimum mixing ratio and strength values were determined.

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1. Introduction

The properties of the geopolymer such as mechanical strength, acid resistance, high temperature resistance, low shrinkage, low thermal conductivity and relatively low cost have attracted the attention of researchers in recent years [1–6]. With the production of geopolymer, the emission during traditional portland cement production is reduced by 70 %. The geopolymer is therefore known to be environmentally friendly [7]. Geopolymer concrete is considered as an alternative binder to conventional concrete in the construction industry due to its advantages such as low emission, energy saving, reduction of storage area problem, conservation of natural resources and relatively low cost [8]. In geopolymer production, industrial wastes such as fly ash [9], blast furnace slag [10], ceramic waste [11], palm oil clinker [12], rice husk ash [13] agricultural wastes and clay [14], kaloin [15] and lime-based natural materials are used as binders. For activation of the binders, NaOH, glass water [9], potassium hydroxide [16], potassium silicate [17] etc. activators are needed. Geopolymer synthesis is described as a simplified pathway such as dissolution of aluminosilicate precursor in alkali activator, final polycondensation of monomers, polymer network and solid state conversion [18]. Variables such as binder type, binder amount, activator type, activator ratio, curing temperature, water/binder ratio are important in geopolymer strength. $\text{SiO}_2/\text{Na}_2\text{O}$ ratio, known as silicate modulus (M_s), is important in gaining strength of the geopolymer. Increase of silicate module up to a certain value causes an increase in strength [19]. In recent years, ceramic powder or waste has also been used extensively in geopolymer production [11, 17, 20–22]. Ceramic is a building material widely used in the construction industry. A large amount of ceramic waste is generated during ceramic production and destruction. It is stated that ceramic production has reached 13.552 million m^2 as of 2017. However, it is reported that 1 m^2 of ceramics creates about 1.9 kg of waste, in which case 25.75 million tons of ceramic waste comes out [23]. Rapid development in the

ceramic industry causes more waste and limits sustainable development as well as negative effects on the environment [24–26]. Ceramic waste also creates a storage problem by covering a large area [27]. For this reason, it is important to recycle ceramic powder by using it in geopolymer production. Ceramic powder used in geopolymer studies is primarily in the form of calcined clay as ceramic raw material [17]. The other method is obtained by grinding the waste ceramic into powder [11]. Activation of waste ceramics with alkalis by pulverizing is a common method, and studies with raw ceramic powder are limited. Huseien et al. [28] prepared mortar samples by activating mixtures of ceramic powder, fly ash and blast furnace slag with NaOH and Na₂SiO₃. In their studies examining the bond strength of the mixtures, it was determined that alkali activated mortars containing high amounts of ceramics and fly ash show good performance against acid attack [11]. Martin Kepperta et al. [20] stated that the geopolymers they produced using two different red-clay ceramic powders had satisfactory mechanical properties. Abdollahnejad et al. [29] produced steel fiber, PVA fiber and polypropylene fiber mortar samples by activating the ceramic powder and slag mixture with Na₂SiO₃. They found that the fiber addition improves the mechanical properties. Kovářík et al. [17] determined that the geopolymer samples produced by using metakaolin, potassium silicate ceramic aggregate have approximately 12 MPa flexural strength and 90 MPa compressive strength after 1000 °C temperature effect. Amin et al. [30] found that the geopolymeric bricks they produced using ceramic powder waste, slaked lime and NaOH increased the 28-day geopolymerization degree. Huseien et al. [31] exposed the samples produced by the activation of fly ash, furnace slag and high amount of waste ceramic powder to 900 °C high temperature, the samples containing 70 % waste ceramic powder 20 % blast furnace slag and 10 % fly ash have optimum resistance to high temperature. In another study, they produced alkali activated mortar using high amounts of ceramic powder, fly ash and blast furnace slag, and they found that with the increase in the amount of fly ash from 0 % to 40 %, the compressive strength increased and the resistance against acids and sulphates increased. They stated that drying shrinkage increased and freeze-thaw performance decreased with the increase of blast furnace slag in the mixture [28]. Shoaiei et al. [32] the mortar samples they produced by activating the waste ceramic powder with a mixture of NaOH and Na₂SiO₃ cured at different temperatures. They determined the optimum curing temperature as 90 °C and the alkali solution/binder ratio as 0.6. They stated that 27.9 MPa compressive strength and 6.65 MPa flexural strength were achieved in samples of 28 days. In this study, physical and mechanical properties of geopolymer mortars produced by alkali activation of raw ceramic powder used in ceramic production were investigated. The aim of this study is to pioneer the studies for the production of geopolymer ceramics with higher strength than traditional tiles and ceramics by alkali activation of ceramic powder.

2. Materials and Methods

2.1. Materials

2.1.1. Ceramic powder

In this study, raw ceramic powder used in ceramic production was used. The chemical content of ceramic powder is given in Table 1.

Table 1. Chemical content of ceramic powder.

Compound	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	P ₂ O ₅
Mass %	54.58	22.02	0.91	8.89	0.21	0.05	0.21	0.81	0.26

The ceramic powder used in the study is shown in Fig. 1.



Figure 1. Ceramic powder used in the study.

The SEM (Scanning Electron Microscope) images of the ceramic powder is given in Fig. 2.

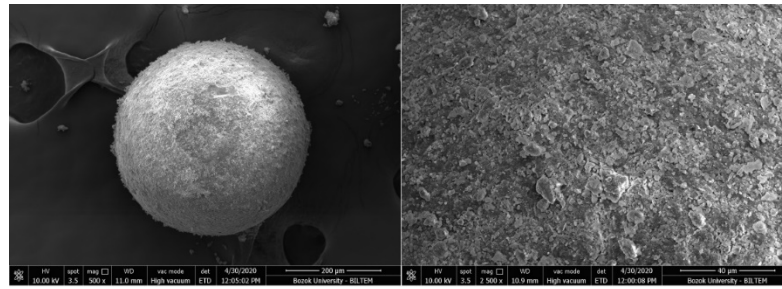


Figure 2. SEM images of ceramic powder.

The X Ray diffraction Analysis (XRD) of the ceramic powder is given in Fig. 3.

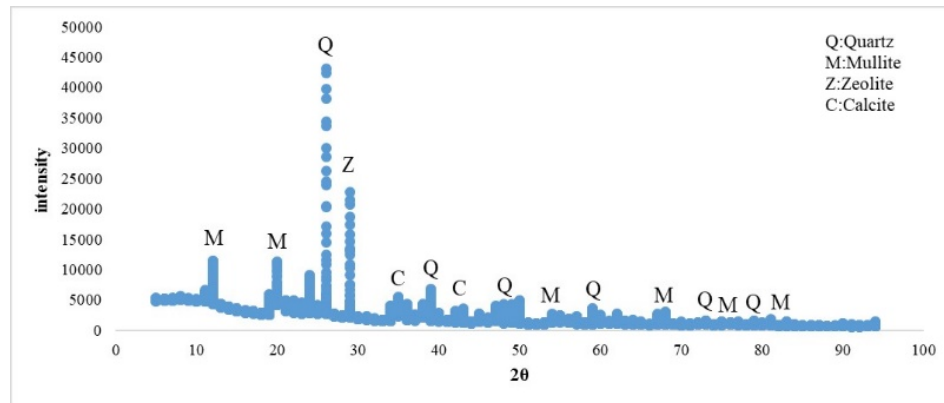


Figure 3. XRD analysis of ceramic powder.

2.1.2. Sodium Hydroxide (NaOH)

The properties of NaOH used in the study are given in Table 2.

Table 2. Properties of Sodium Hydroxide (NaOH).

Chemical Name	Sodium hydroxide
Chemical Formula	NaOH
Molecular weight	40 g/mol
Asidimetric	≥97
Na ₂ CO ₃	≤1
Cl	<0,01
SO ₄	≤0,01
Heavy metal	≤0,002
Al	≤0,002
Fe	≤0,002

2.1.3. Sodium Silicate (Na₂SiO₃)

The properties of Sodium silicate used in the study are given in Table 3.

Table 3. Properties of Sodium Silicate (Na₂SiO₃).

Chemical Name	Sodium Silicate
Chemical Formula	Na ₂ SiO ₃ nH ₂ O
Molecular weight	122.06 g/mol
Density	1.39 g/cm ³ (20 °C)
Molecular Module SiO ₂ /Na ₂ O	2.07
Na ₂ O	%11.71
SiO ₂	%23,46
Fe	39 ppm
Cl	%0.01
SO ₄	%0.01

2.1.4. Aggregate

Basalt origin aggregate with a maximum grain size of 4 mm and a specific gravity of 2.83 g/cm³ was used in the study.

2.1.5. Water

Tap water was used to prepare the mortar mixes.

2.2. Methods

In this study, mortar samples with a water/binder (W/B) ratio of 0.40, 0.45 and 0.50 and containing 11 %, 13 %, 15 % Sodium (Na) by weight and silicate modulus (Ms) of 0, 0.1, 0.2 and 0.3 were prepared. Mixing ratios of the samples are given in Table 4.

First of all, NaOH and Na₂SiO₃ were mixed with the required amount of water and kept until it came to room temperature. Aggregate and ceramic powder were mixed in a standard cement mixer for 60 seconds. Then, by adding a mixture of activator and water, it was mixed for 120 seconds. The prepared mixtures were placed in standard molds of 40×40×160 mm. Shoei et al.[32] applied a cure between 60 °C and 105 °C in the geopolymer production they made with ceramic powder and determined the optimum temperature as 90 °C. They stated that the compressive strength increased with the increase in curing temperature. In different studies, 105 °C curing temperature was used during the production of geopolymer [33–34]. All of the samples produced in this study were kept in an oven at 105 °C for 24 hours. Samples removed from the oven were cured at room temperature for up to 28 days. At the end of 28 days, unit weight (UW), apparent porosity (AP), water absorption (WA) ratio, ultrasound pulse velocity (UPV), flexural strength (FS) and compressive strength (CS) tests were performed on the samples. Flexural and compressive test device is given in Fig. 4.

Table 4. Mixing ratio of samples.

No	Sample code	Na/binder (%)	water/binder (W/B)	Ms=SiO ₂ /Na ₂ O	binder/aggregate
1	N11ST40MS0	11	0.40	0	1/3
2	N11ST40MS1	11	0.40	0.1	1/3
3	N11ST40MS2	11	0.40	0.2	1/3
4	N11ST40MS3	11	0.40	0.3	1/3
5	N11ST45MS0	11	0.45	0	1/3
6	N11ST45MS1	11	0.45	0.1	1/3
7	N11ST45MS2	11	0.45	0.2	1/3
8	N11ST45MS3	11	0.45	0.3	1/3
9	N11ST50MS0	11	0.50	0	1/3
10	N11ST50MS1	11	0.50	0.1	1/3
11	N11ST50MS2	11	0.50	0.2	1/3
12	N11ST50MS3	11	0.50	0.3	1/3
13	N13ST40MS0	13	0.40	0	1/3
14	N13ST40MS1	13	0.40	0.1	1/3
15	N13ST40MS2	13	0.40	0.2	1/3
16	N13ST40MS3	13	0.40	0.3	1/3
17	N13ST45MS0	13	0.45	0	1/3
18	N13ST45MS1	13	0.45	0.1	1/3
19	N13ST45MS2	13	0.45	0.2	1/3
20	N13ST45MS3	13	0.45	0.3	1/3
21	N13ST50MS0	13	0.50	0	1/3
22	N13ST50MS1	13	0.50	0.1	1/3
23	N13ST50MS2	13	0.50	0.2	1/3
24	N13ST50MS3	13	0.50	0.3	1/3
25	N15ST40MS0	15	0.40	0	1/3
26	N15ST40MS1	15	0.40	0.1	1/3
27	N15ST40MS2	15	0.40	0.2	1/3
28	N15ST40MS3	15	0.40	0.3	1/3
29	N15ST45MS0	15	0.45	0	1/3

No	Sample code	Na/binder (%)	water/binder (W/B)	Ms=SiO ₂ /Na ₂ O	binder/aggregate
30	N15ST45MS1	15	0.45	0.1	1/3
31	N15ST45MS2	15	0.45	0.2	1/3
32	N15ST45MS3	15	0.45	0.3	1/3
33	N15ST50MS0	15	0.50	0	1/3
34	N15ST50MS1	15	0.50	0.1	1/3
35	N15ST50MS2	15	0.50	0.2	1/3
36	N15ST50MS3	15	0.50	0.3	1/3



Figure 4. Compressive and flexural test device.

3. Results and Discussion

3.1. Unit weight (UW) test results

Unit weight values of the samples are given in Fig. 5. The unit weights of the samples vary between 1.66 g/cm³ and 2.22 g/cm³. The lowest unit weight was observed as 1.66 g/cm³ in the sample coded N11ST45MS3 containing 11 % Na, silicate modulus Ms = 0.3 and produced with 45 % water.

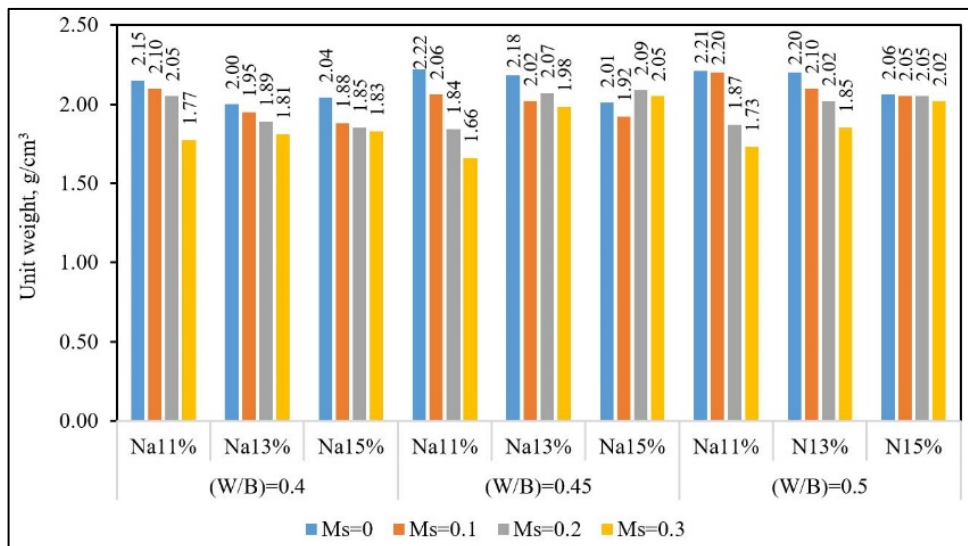


Figure 5. Unit weights of the samples.

The largest unit weight was observed in N11ST45MS0 sample with 11 % Na and silicate module Ms = 0. It was determined as 2.22 g/cm³. It is observed that the unit weight values of all samples decrease with the increase of silicate modulus. Average unit weights were determined as 1.94 g/cm³, 2.01 g/cm³, 2.03 g/cm³ for water/binder (W/B) ratio 0.40, W/B = 0.45, and W/B = 0.50 respectively. Unit weight values showed an insignificant increase with the increase in the amount of water in the mixture. In a study where geopolymers mortar was produced by alkali activation of waste ceramic powder, it was determined that the unit weight of samples produced by curing at 105 °C and having a W/B ratio of 0.40 was 1.849 g/cm³. In samples with a W/B ratio of 0.5, it was found to be 1.861 g/cm³ [32]. In another study where alkali activated mortar was produced by mixing ceramic tile wastes with fly ash and blast furnace

slag, the unit weight of the alkali activated mortar produced using completely ceramic powder was determined to be 2.20 g/cm^3 at the end of 28 days [35]. It is known that the unit weight decreases as the curing temperature increases in geopolymer mortars [19, 32]. In this study, unit weight change is not significant since all samples are cured at 105°C at constant temperature. In a study where fabricated geopolymer brick was produced with alkali activation of waste ceramic powder, it was stated that bulk density increased with 0.50 % increase in NaOH concentration. Unit weight increased in samples with high W/B ratio with the increase in $\text{Ca}(\text{OH})_2$ ratio in the mixture [30].

3.2. Apparent porosity (AP) and water absorption (WA) test results

Determination of AP and WA ratio was made according to ASTM C642-3 standard [36]. AP values of the samples vary between 2.19 % and 21.63 %. The lowest AP was observed as 2.19 % in N15ST40MS1 sample. The highest AP was observed as 21.63 % in N13ST40MS3 sample. It is observed that the porosity increases with the increase of the silicate modulus in mixtures. The porosity generally decreases with the increase in the sodium (Na) content in the samples. The WA ratio of the samples ranges from 1.06 % to 11.74 %. While the lowest WA ratio was observed as 1.06 % in N15ST40MS1 sample, the highest WA ratio was found as 11.74 % in N13ST40MS3 sample. It is observed that WA ratios increase with the increase of silicate modulus. With the increase of Na content in samples, WA ratio generally decrease. In a study investigating the effect of ceramic powder additive in slag pastes, it is stated that ceramic powder additive reduces the WA ratio in 28-day samples. The WA ratio is observed around 17 % in samples without ceramic powder. The WA ratio decreases to approximately 13 % by increasing the ceramic powder in the mixture to 50 % [37]. In a study where waste ceramic powder and blast furnace slag were evaluated as self compacted concrete as a result of alkali activation, the WA ratio was 6.8 % in samples produced only with blast furnace slag (BFS). It was determined that it was 10.1 % in samples replaced with 50 % ceramic powder, and 14.1 % in samples replaced with 80 % ceramic powder [38]. This situation is explained by the fact that the particles that do not react due to the size of the particles of the waste ceramic powder increase the WA feature [39]. In a study in which an alkali activated paste was produced using ceramic powder and slag, it was stated that the ceramic powder additive reduced to workability [37]. In another study where geopolymer was produced by adding waste ceramic powder into fly ash and BFS, it is stated that the waste ceramic powder additive reduces the WA ratio [35]. On the other hand, it is known that increasing the silicate modulus decreases the workability in the mixture. For this reason, it is observed that the porosity and the WA ratio increase with the increase of silicate modulus in the samples. With the increase in the activator ratio in the mixture, some workability increases, so the porosity and WA ratio decrease. Curing temperature also has an effect on the porosity ratio and WA ratio. It is known that the porosity increases due to the loss of water in the sample with the increase of the curing temperature [19, 30, 40]. AP and WA ratios of the samples are given in Fig. 6.

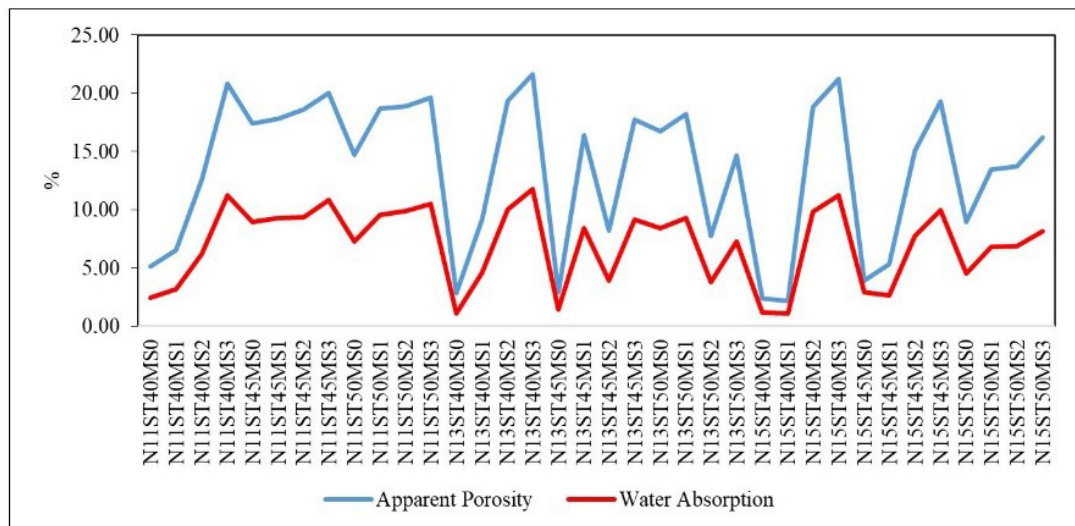


Figure 6. AP and WA ratios of the samples.

3.3. Ultrasound pulse velocity (UPV) test

The ultrasound pulse velocities of the samples were measured with a device called pundit (portable ultrasonic non-destructive digital indicating tester) according to EN 12504-4 [41]. UPV is a non-destructive testing method. It is a method used to identify voids and cracks and to determine the relative quality of concrete. UPV is used to determine properties such as void structure and cracks, and indirectly in estimation of compressive strength [42–44]. UPV values of the samples are given in Fig. 7. The UPV of the samples vary between 2108 m/s and 3661 m/s. The lowest UPV was determined as 2108 m/s in

N11ST45MS3 sample. The highest UPV was determined as 3361 m/s in N11ST50MS0 sample. A decrease in UPV was observed due to the increase in the void structure with the increase of silicate modulus in the samples. In a study in which waste brick dust and ceramic powder were activated with NaOH and Na_2SiO_3 to produce geopolymer paste, the UPV of the samples for 28 days were determined between 2635 m/s and 3724 m/s [45]. In another study where geopolymer was produced with ceramic powder, fly ash and BFS, a decrease was observed in UPV values with the increase in the amount of waste ceramic powder in the mixture [31]. In a study examining the strength and durability properties of self compacting concrete produced with Alkali activated waste ceramic powder and BFS, it is stated that the increase in ceramic dust in samples reduces the amount of loss in UPV after acid attacks [38].

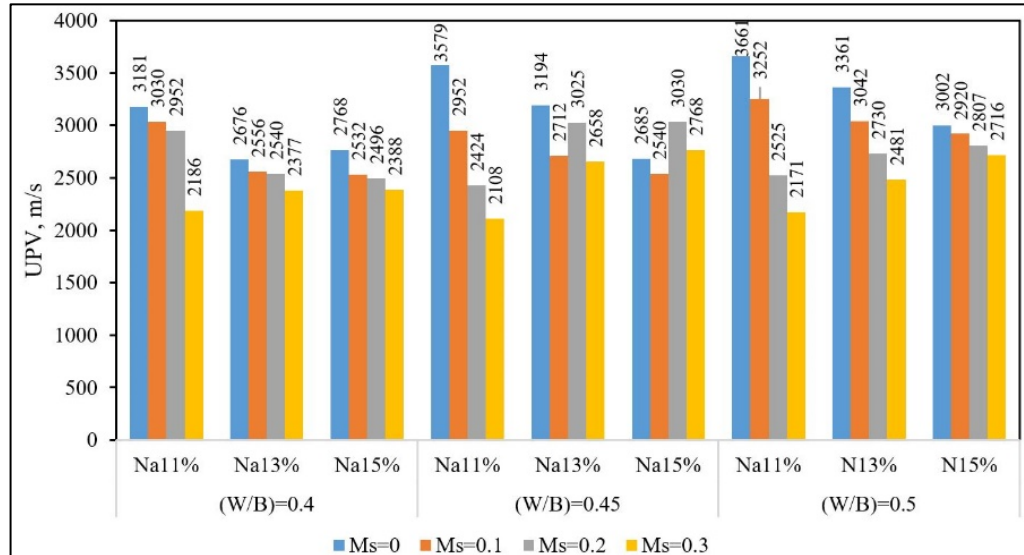


Figure 7. UPV values of samples.

3.4. Flexural Strength (FS) Test Results

The geopolymer samples produced within the scope of the study were loaded from a single point with a 100 mm support opening in accordance with EN 1015-11, 2000 [46]. Flexural strength test result are given in Fig. 8. The FS of the samples varies between 1.89 MPa and 10.38 MPa. The lowest FS was observed as 1.89 MPa in N13ST40MS3 sample produced with Ms = 0.3 with 0.40 W/B containing 13 % Na, while the highest FS was determined as 10.38 MPa in N15ST40MS0 sample produced with 0.40 W/B containing 15 % Na. In samples containing 15 % Na and 0.40 W/B the FS decreased by 77.4 % as the silicate modulus increasing to 0.3. In samples with 15 % Na and W/B ratio 0.50, the FS decreased from 5.52 MPa to 5.51 MPa with the silicate modulus increasing to 0.3. In samples produced with 0.40 and 0.45 W/B, a significant decrease in FS is observed with the increase of silicate modulus. Besides, in samples with a W/B ratio of 0.50, the decrease in FS with the increase of silicate modulus is not significant. With the increase in the W/B ratio in the mixture, it causes an increase in workability and an increase in porosity. The increase in Na_2SiO_3 in the mixture has a negative effect on workability. For this reason, the differences between the FS in mixtures with W/B 0.50 are not significant. In a study where ceramic powders of various gradations are used as aggregates and metakaolin based geopolymer is produced, it is stated that the FS of the samples varies between 9 MPa and 12 MPa [47]. In another study where geopolymer was produced by adding waste ceramic powder into fly ash and BFS, it is stated that the waste ceramic powder additive reduces the FS [35]. Shoaei et al. [32] stated that in geopolymer mortars produced with waste ceramic powder (WCP), FS increases with the increase of curing temperature. They stated that the 28-day FS of the samples cured at 105 °C increased from 4.88 MPa to 6.25 MPa with the increase in W/B ratio from 0.40 to 0.50. In this study, it is seen that the average FS increased from 5.34 MPa to 5.50 MPa with the increase of water ratio in samples from 0.40 to 0.50 ratio. It is observed that the FS is affected by 105 °C, which is kept constant in sample production. The FS increases a little with the increase in the amount of water. Some studies indicate that moisture is required to produce a good geopolymer [45–48]. Huseien et al [38] in their geopolymer study with a mixture of BFS and waste ceramic powder, found that the FS decreased with the increase in the amount of waste ceramic powder in the mixture. The samples produced with BFS stated that while the FS was 2.1 MPa, the FS decreased to 1.2 MPa with 80 % WCP replacement. It is stated that this situation is caused by the fact that WCP is larger than BFS and therefore the amount of ceramic powder involved in activation is low [39].

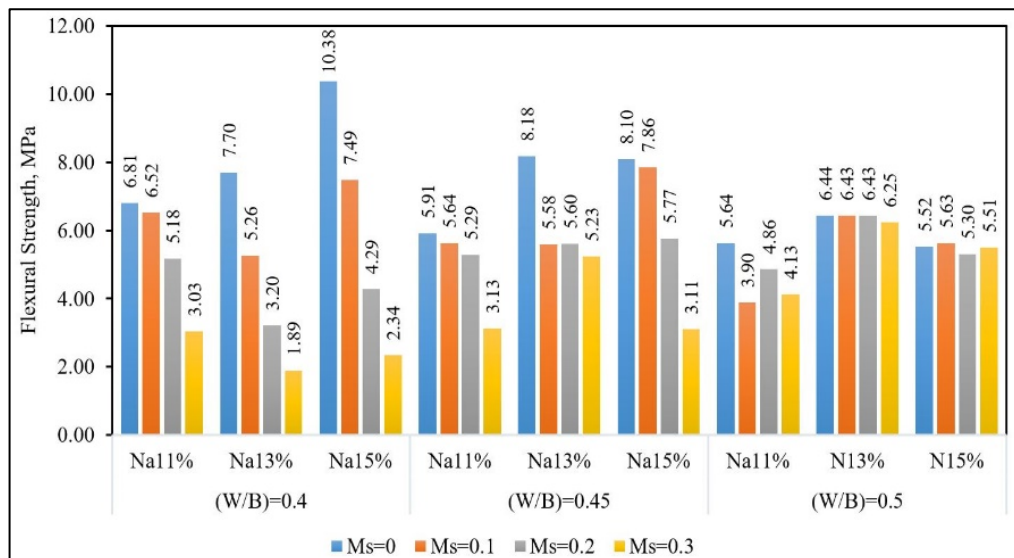


Figure 8. FS of the samples.

3.5. Compressive Strength (CS) Test Results

CS test is done according to EN 1015-11, 2000 standard [46]. CS of the samples are given in Fig. 9. The CS of the samples ranged from 10.42 MPa to 41.53 MPa. The lowest CS was observed as 10.42 MPa in N13ST40MS3 sample with $M_s = 0.3$ produced with 0.40 W/B containing 13 % Na. The highest CS was determined as 41.53 MPa in the sample N15ST40MS0 with $M_s = 0$ produced with 0.40 W/B containing 15 % Na. In samples containing 15 % Na and 0.40 W/B, the CS decreased by 70.8 % with the silicate modulus increasing from 0 to 0.3. In samples with 15 % Na and W/B ratio of 0.50, the CS decreased by 7 % with the silicate modulus increasing from 0 to 0.3. In samples with W/B ratios of 0.40 and 0.45, significant decreases in CS are observed with the increase of the silicate modulus. The difference in compressive strength is not significant for samples with a ratio of W/B 0.50. In a study where geopolymer was produced by adding waste ceramic powder (WPC) into fly ash and BFS, it was determined that the 28-day CS decreased from 70.4 MPa to 34.8 MPa with the increase of WPC in the samples from 50 % to 70 % [37]. This situation is explained by the decrease in the amount of calcium in the environment and the increase in the amount of silicon [35, 49]. Hwang et al. [45] found that the CS develops with time in geopolymer samples, which are cured at ambient temperature and contain different amounts of waste brick dust, WPC and BFS. Ca^{+} ions of Cao content and Si^{4+} and Al^{3+} ions rich in alkali cause the formation of C-S-H gel and C-A-S-H gel [33–34]. In this study, it was determined that the average CS of the samples cured at 105 °C at constant temperature was 24.47 MPa, W/B = 0.40. 25.11 MPa for W/B = 0.45, 24.54 MPa for W/B = 0.50. Average compressive strengths were determined as 22.86 MPa, 24.86 MPa and 26.38 MPa for 11 % Na, 13 % Na and 15 % Na values for all samples, respectively. With the increase in the Na ratio in the samples, an increase was observed in the CS. In geopolymer mortars, it is known that the CS increases with the increase of the sodium ratio in the mixture, and the porosity and CS decrease with the increase of the silicate module [40, 44]. Shoaie et al. [32] stated that the CS increases with the increase of curing temperature in geopolymer mortars produced with WPC. They stated that the 28-day CS of the samples cured at 105 °C increased from 23 MPa to 26.4 MPa with the increase in W/B ratio from 0.40 to 0.50. Regarding the effect of alkaline solution-binder (S/B) ratio, S/B increased values improve CS. They found that it reached its maximum value at S/B = 0.6. They also stated that 28-day samples had good strength within the age groups of the samples. Similarly, a certain amount of water increase in the CS as in the FS positively affects the strength [44, 48]. Generally, the strength development in ceramic powder based geopolymers is not very high compared to BFS and FA origin geopolymers due to the chemical composition of the ceramic powder [35, 50–51]. Rashad et al. [34] found that the compressive strength increases with the increase in the amount of WPC in the mixture in alkali activated pastes produced with ceramic powder and slag. They stated that with the increase of the amount of WPC in the mixture to 50 %, the 28-day CS increased 1.53 times. In the literature, it is stated that temperature curing increases the strength as well as the durability [52]. The increase in the NaOH/Na₂SiO₃ ratio causes a decrease in the CS [4, 53–54]. When the NaOH/Na₂SiO₃ ratio is exceeded the optimum value, the strength is negatively affected [19, 40].

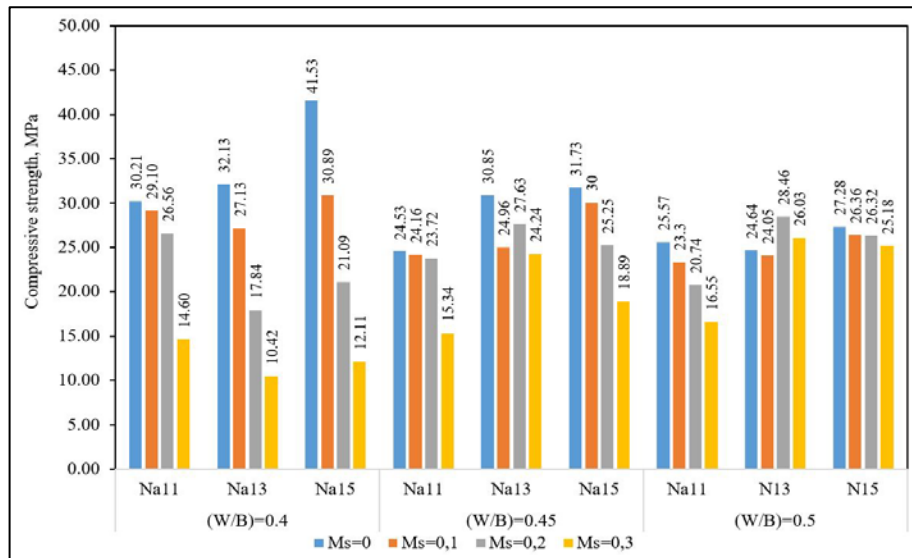


Figure 9. CS of the samples.

3.6. Relationships between some properties

The relationship between UPV-CS and AP-CS of samples are given in Fig. 10. All correlations in graphs are given exponential. It is observed that UPV values increase and AP values decrease with the increase of CS in all sodium ratios. It was determined that there is a strong relationship between UPV and CS with $R^2 = 0.96$. Hwang et al. [45]. found a correlation of $R^2 = 0.91$ between UPV and CS in alkali activated waste tile and ceramic powder pastes. It is seen that the CS increases with the decrease of AP. The correlation between AP and CS was determined as $R^2 = 0.79$. The relationship between UW and CS of samples are given in Fig. 11. The correlation between UW and CS was determined as $R^2 = 0.94$. It was observed that the CS increased with the increase in UW. The correlation between FS and CS of all samples are given in Fig. 12. In the correlation between FS and CS of all samples, a relation such as $R^2 = 0.83$ was observed. Huseien et al. [35] found a correlation of $R^2 = 0.9435$ between FS and CS for 28 days in their geopolymer studies with waste ceramic additives.

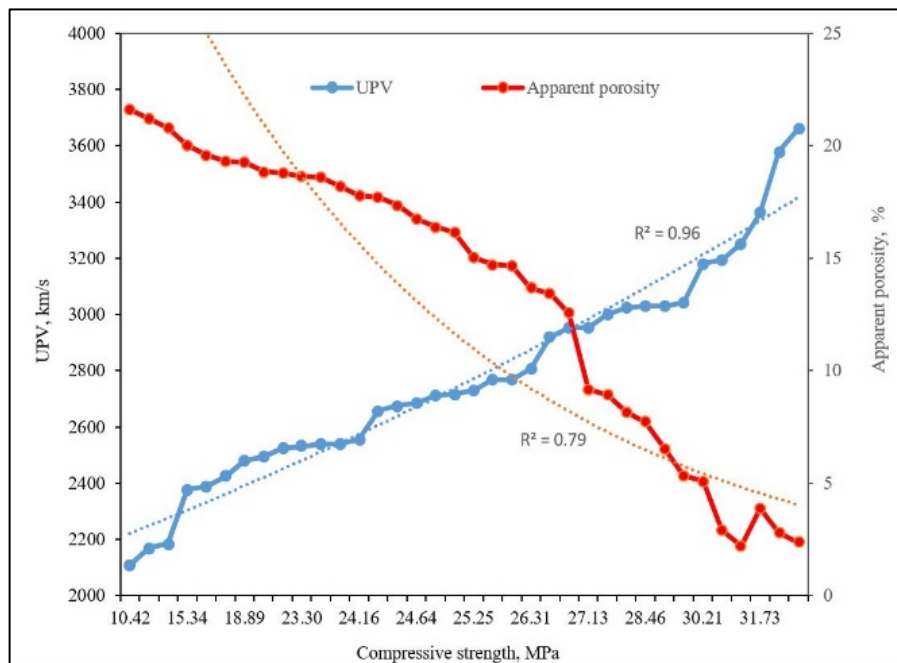


Figure 10. Relationship between UPV-CS and AP-CS of the samples.

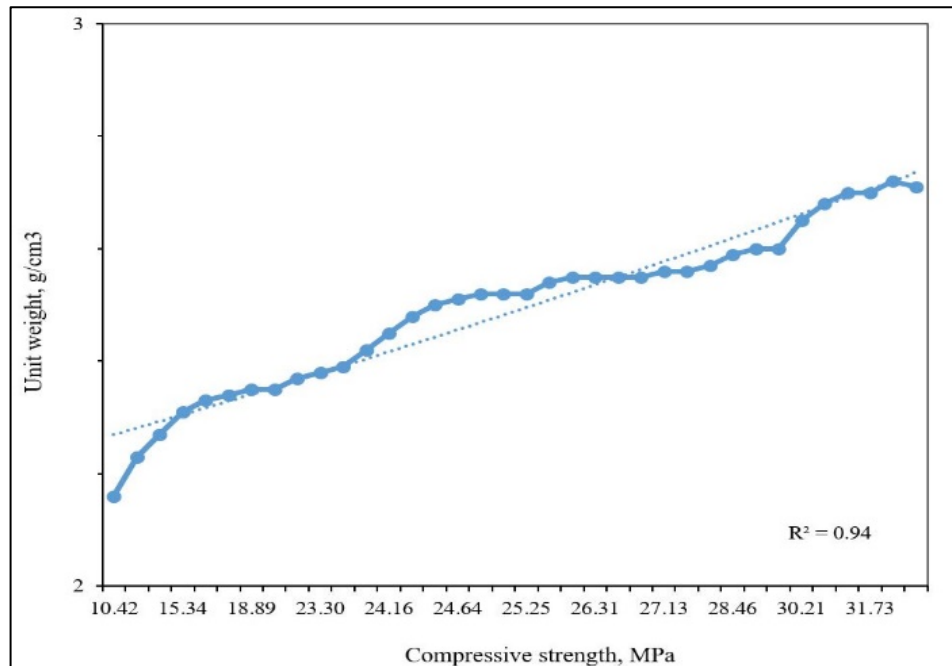


Figure 11. Relationship between UW-CS of the samples.

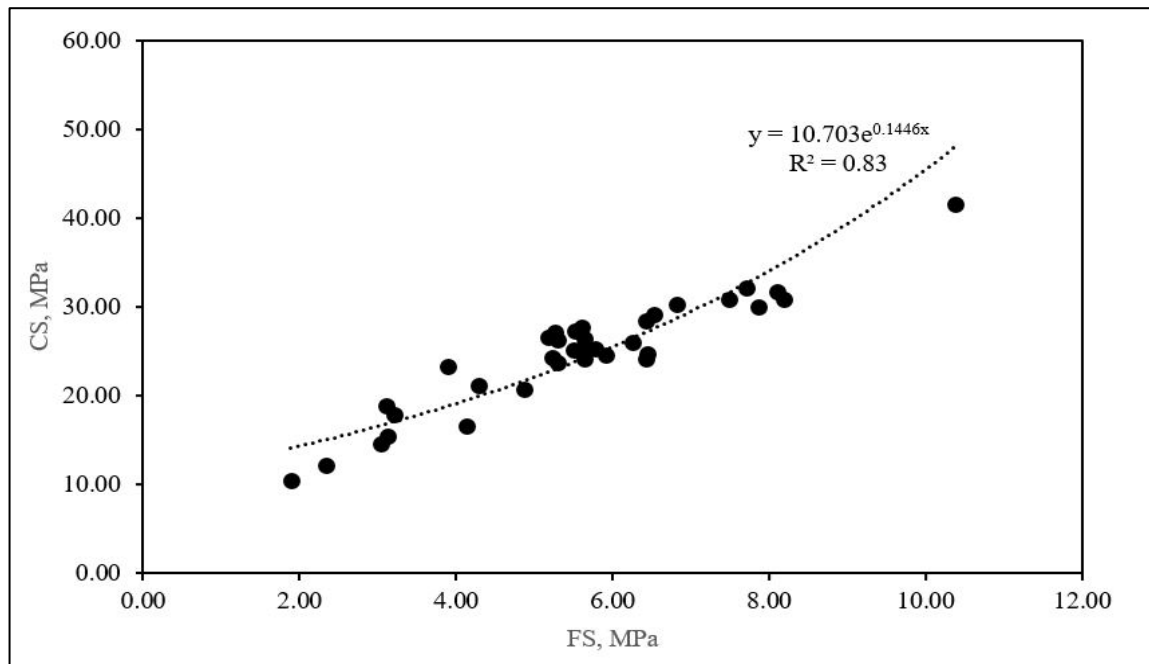


Figure 12. Correlation between CS and FS of all samples.

3.7. Experimental Design and Optimization

Response surface method (RSM) is a method consisting of mathematical and statistical techniques. Empirical models and first presented by Box and Wilson⁴⁴ in 1951. Then RSM, It has been used in modeling numerical experiments [55–57]. In this study, flexural strength (FS) and compressive strength (CS) test results are designed with RSM. Water/binder W/B, activator/binder Na (%), silicate module is coded with Ms. FS codes were used for flexural strength and CS codes for compressive strength. The independent variables in this study are W/B, Na (%) and Ms values. In this study, optimum values were determined by response surface method by using 11 %, 13 % and 15 % Na ratio, 0, 0.1, 0.2, 0.3 Ms ratio and 0.40, 0.45, 0.50 W/B independent variables. In order to obtain a response surface, 27 data for each response were used for variance analysis. The response surfaces of FS and CS as a function of Na%, W/B ratio and Ms values are given in Fig. 13 and Fig. 14, respectively. Equations obtained from regression models for FS and CS are given in Equation (1) and Equation (2). Regression coefficient was found as $R^2 = 0.77$ for FS and $R^2 = 0.73$ for CS.

$$FS = 27.98 - 0.45(Na) - 64.37(W/B) - 81.49(Ms) + 2.35[(Na \times W/B)] - 1.71(Na \times Ms) + 211.25[(W/B) \times Ms], \quad (1)$$

$$CS = 167.69 - 6.66978(Na) - 360.19(W/B) - 236.37(Ms) + 18.89[Na \times (W/B)] - 6.21(Na \times Ms) + 637.08[(W/B) \times Ms] \quad (2)$$

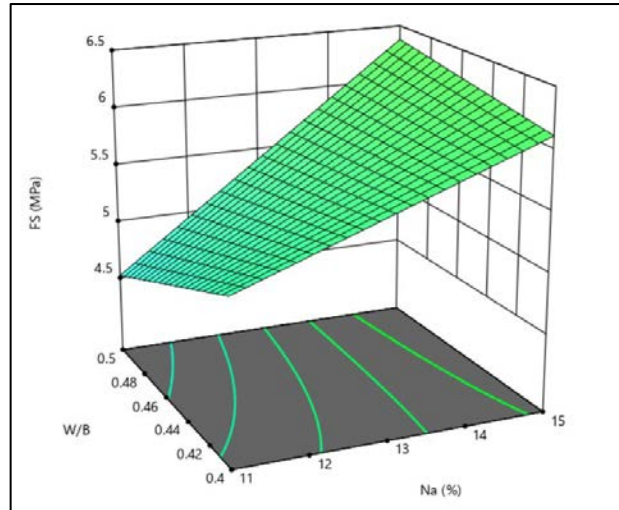


Figure 13. Respose surface for FS.

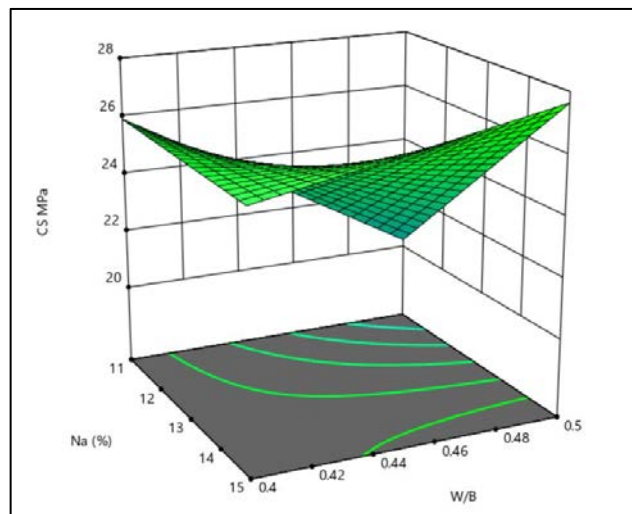


Figure 14. Respose surface for compressive strength (CS).

The numerical optimization technique proposed by Derringer and Suich [58] to simultaneously optimize the responses for a desirability is given as functions (d_i). A desirability function for each answer ranges from $0 \leq d_i \leq 1$. A multipurpose optimization problem the single compound given in Equation 3 is resolved using the response or general desirability (Dis) which is the geometric mean of individual desirabilities.

$$Dis = (d_1 \times d_2 \times d_3 \times \dots \times d_n)^{(1/n)}, \quad (3)$$

n represents the number of response surfaces in the optimization. Suitable regions used in this study are:

$$\begin{aligned} \%11 &\leq Na \leq \%15, \\ 0.40 &\leq (W/B) \leq 0.50, \\ 0 &\leq Ms \leq 0.30. \end{aligned}$$

In order to obtain high strength, FS and CS strength must be high. Activator is seen as an important cost element in the production of geopolymer. Therefore, $n = 4$ in the equation to minimize the activator while maximizing flexural and compressive strength.

$$Dis = (d1 \times d2 \times d3 \times d4)^{(1/4)}$$

The optimum solution for this modeling result is given in Table 5.

Table 5. The optimum solution for this modeling result.

		Constraints		
		Lower Limit	Upper Limit	Optimum Solution
Na (%)	Minimum	11	15	12.67
W/B	Minimum	0.4	0.5	0.425
Ms	Minimum	0	0.3	0.12
FS (MPa)	Maximum	2.34	10.38	5.97
CS (MPa)	Maximum	12.11	41.53	26.75

The desirability level resulting from the modeling of the samples and the optimum flexural (FS) and compressive strength (CS) values are shown in Fig. 15. In Fig. 13, with the increase of Na ratio, an increase in flexural strength is observed. With the increase of W/B at low Na ratios, the flexural strength decreased. With the increase of W/B at high Na ratios, the flexural strength increased. Fig. 14 shows an increase in compressive strength with the increase of Na ratio. Similar to the flexural strength, the compressive strength decreased with the increase of W/B at low Na ratios, while it increased significantly at high Na ratios. The optimum solution was determined as 12.67 % Na, 0.425 W/B 0.12 Ms, and the flexural strength as 5.97 MPa and 26.75 MPa. Mermerdas et al. [59] In their optimization study for geopolymer produced with blast furnace slag and fly ash, determined that the optimum curing temperature was between 9 and 24 hours at 60 °C. Zhang and Yue [60] made use of the optimization technique in their study where they produced glass powder doped slag based geopolymer. Regarding the flexural and compressive strengths, they found that the optimal values were 14.57 % glass powder content for 8.31 % Na₂O. They stated that the Response Surface methodology (RSM) is an efficient and useful method for optimizing design parameters of the following.

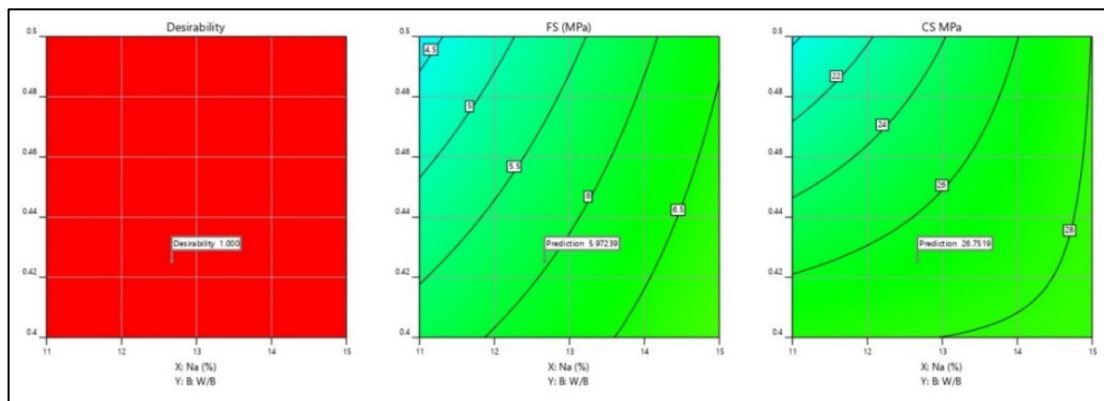


Figure 15. Desirability, optimum FS and CS.

4. Conclusions

The following results have been obtained from the experimental studies:

- 28-day flexural strength of mortar samples produced by alkali activation of ceramic powder varies between 2.34–10.38 MPa and compressive strength between 10.42–41.53 MPa. The highest compressive strength was determined as 41.53 MPa for samples containing 40 % water/binder, 15 % Na and Ms = 0.
- With the increase of silicate modulus in all samples, decreases in flexural and compressive strength were detected. Using only NaOH for alkali activation of ceramic powder gives good results in terms of strength.

- With the increase in the Na ratio by weight in the samples with low water content, the flexural and compressive strengths increased significantly. On the other hand, this increase in strength is not evident in samples with high water content. In samples with W/B ratios of 0.40 and 0.45, significant decreases in compressive strength are observed with the increase of the silicate modulus. The difference in compressive strength is not significant for samples with a ratio of W/B 0.50.
- As a result of the optimization, it has been determined that the optimum mixture of 12.67 % Na, 0.425 W/B and 0.12 Ms can achieve a flexural strength of 5.97 MPa and a compressive strength of 26.75 MPa.

High strength geopolymer can be produced by alkali activation of ceramic powder. It is thought that this geopolymer material can be used in the production of high strength ceramics.

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