Karaelmas Fen ve Müh. Derg. 10(2):177-185, 2020

Karaelmas Science and Engineering Journal
Journal home page: https://dergipark.org.tr/tr/pub/karaelmasfen
DOI: 10.7212/zkufbd.v10i2.1611

Research Article
Received / Geliş tarihi : 16.04.2020
Accepted / Kabul tarihi : 02.06.2020

Effect of Alkali Content and Activator Modulus on Mechanical Properties of Alkali Activated Mortars

Alkali İçeriği ve Aktivatör Modülünün Alkali Aktive Edilmiş Harçların Mekanik Özellikleri Üzerindeki Etkisi

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Abstract

Alkali activation is a promising method of reducing cement production considerably without further significant investments. Utilization of these materials are of extreme importance when environmental aspects such as CO_2 emissions and energy consumptions are considered. In this article, effect of activator modulus and alkali content on fresh behavior and mechanical properties were investigated. 12 M NaOH solution and sodium silicate solution with a modulus of 3 were used. Water-to-binder ratio was selected as 0.5 for all specimens where binder content was calculated as the total of slag content plus activator solids. Slag content was 500 kg/m^3 for all mixtures. Mechanical tests were conducted on 40 x 40 x 160 mm prismatic specimens. Results indicate that mechanical properties of alkali activated mixtures are highly dependent on both parameters hence proper optimization of these two parameters becomes compulsory. In this study, it is concluded that alkali content (Na_2O/slag) should be selected 9% and whereas activator modulus should be leveled near a value of 1.5. Flexural properties were also highly affected from optimization of these parameters and similar findings are deduced. Early strength evaluations are highly dependent on activator modulus. Workability of the mixture was also assessed and it should be noted that flowability of mixture was adversely affected with the increasing activator solids content.

Keywords: Activator modulus, Alkali activated slag, Alkali content, Sodium silicate

Öz

Alkali aktivasyonu çok büyük yatırımlar yapılmadan dahi çimento üretimini önemli seviyede indirgeyebilecek umut verici bir yöntem olarak görülmektedir. Atık olarak görülen cürufların bu şekilde endüstriye kazandırılması ile CO2 emisyonlarının ve enerji tüketiminin azalması, bu malzemelerin sürdürelebilirlik kapsamında önemli katkılar sağlayabileceğini işaret etmektedir. Bu çalışmada, alkali içeriği ve alkali aktivatör modülü değerlerinin alkali aktivasyonu sürecinde harç numunelerin akışkanlık ve mekanik özellikleri üzerindeki özellikleri araştırılmıştır. Aktivatör olarak 12 M NaOH çözeltisi ve 3 modüllü sodyum silikat kullanılmıştır. Su-çimento oranı 0.5 olarak belirlenmiş ve bağlayıcı miktarı karışımda kullanılan curuf ve katı aktivatör miktarlarının toplamı olarak alınmıştır. Harç numuneler hazırlanırken cüruf miktarı 500kg/m^3 olarak tercih edilmiş ve 40 x 40 x 160 mm prizmatik kalıplara dökülerek 7 ve 28 gün boyunca kürde tutulmuşlardır. Harç numunelerin mekanik özelliklerinin kullanılan her iki parametreye daha fazlasıyla bağımlı olduğu ve parametrelerin optimizasyonunun çok önemli olduğu gözlenmiştir. Çalışmanın özelinde kullanılan değerlerle mekanik özellikler açısından en iyi parametre seçimi olarak aktivasyon modülünün 9% ve aktivatör modülünün 2 olduğu seçenek olarak bulunmuştur. Erken dayanım sonuçlarının ağırlık olarak aktivatör modülü değerlerinden fazlasıyla etkilendiği gözlenmiştir. Harç karışımlaranın işlenebilirlik sonuçlarının özellikle katı aktivatör miktarının artmasıyla olumsuz etkilendiği gözlenmiştir.

Anahtar Kelimeler: Aktivatör modülü, Alkali aktive edilmiş cüruf, Alkali miktarı, Sodyum silikat

1. Introduction

Alkali activated slag composites display great potential to be used as replacement for cementitious materials. As an industrial by-product, blast furnace slag is formed within the process of producing iron and has glassy structure rich with calcium and silicon (Gao et al. 2015).

Many rich deposits have been depleted all around the world with the production of ordinary Portland cement which also contributes to 8% of man-made CO_2 emissions (Luukkonen et al. 2018). As the construction sector and accordingly demand for concrete is growing, cement production has to be

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optimized to reduce energy consumption and gas emissions (El-Hassan et al. 2018). In recent years, researchers focus on alternative methods to decrease cement production (Biricik and Karapinar 2018). Partial replacement of cement with supplementary cementitious materials is developed and added to regulations and standards. However, these efforts were not satisfactory for reduction in gas emissions thus any prospect of advancement in this concept gained utmost significance.

Many studies have been conducted to understand the influence of mixture proportions and microstructure of alkali activated slag mixtures (Jiao et al. 2018, Aydin and Baradan 2014). High alkaline solutions were implemented as activator by combining sodium hydroxide pellets and sodium silicate solutions. Na and Si contents were decided and used in mixture proportioning. Combination of sodium silicate and sodium hydroxide was found to be significantly effective when increasing the reactivity of the slag particles. Development of mechanical and rheological properties were monitored with several parameters including activator modulus, Na_2O content, amount of slag, slag fineness, curing method and vice versa (Jiao et al. 2018, Jimenez et al. 2003, Shi 1996, Li et al. 2018).

Due to findings in several studies alkali activated slag composites have several advantages such as low hydration temperature, high mechanical performance, good micro-structural integrity, low porosity and several good durability features. And comparatively, they have several disadvantages such as susceptibility to high shrinkage and rapid setting (Jiao et al. 2018, Gao et al. 2015, Akcaozoglu et al. 2017)

Alkali activated slag composites may be attributed as a great prospect to replace ordinary Portland cement (Taghvayi et al. 2018, Babaee and Castel 2018). Currently its applicability is not easy to implement in construction industry since parameters governing both early and ultimate mechanical properties are interrelated. This may be also concluded from several studies, there is not a net conclusion derived to establish some strict rules for alkali activated concrete mix design. This study aims to fill these gaps with the findings derived from the 2 most significant parameters (Na_2O content and activator modulus) and give insight about the most effective parameter combinations.

2. Materials and Methods

2.1. Materials

In this study ground blast furnace slag was acquired from Erdemir steel factory as waste of steel production and transported to Akcansa cement factory and grinded to a Blaine specific surface area of 5550 cm^2/gr with a specific weight of 2.75 gr/cm^3. Distilled water was used as mixture water. Chemical oxide compositions and physical properties of slag are shown in Table 1. Natural river sand with a maximum diameter size of 4 mm was used. Specific gravity of the sand was 2.71 gr/cm^3. Sodium hydroxide and sodium silicate were used as activators. Sodium hydroxide used was in powder form whereas sodium silicate was used in aqueous form and detailed properties of the sodium silicate activator were provided in Table 2.

2.2. Specimen Preparation

2.2.1. Preparation of activator solutions

NaOH in powder form was mixed with water and 12 M NaOH solution was formed at least 48 hours before mortar mixing. Alkali activator solution was formed prior to mortar casting by combining mixture water with 12M NaOH solution and sodium silicate with proper stirring since sodium silicate was in a more viscous state. Alkali activator solution was left for at least for 24 hours in laboratory conditions.

2.2.2. Mixture proportion configurations

Mixture proportions were designed for the given parameter configurations. Liquid to binder ratio and ground granulated

Table 1. Composition of ground granulated blast furnace slag.

Composition (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
GGBS	40.21	12.64	1.20	36.42	5.50	0.13	0.81	0.64	0.11

Table 2. Composition of sodium silicate activator solution.

Activator	Specific gravity (g/cm^3)	Baume	Na_2O (%)	SiO_2 (%)	Modulus (SiO_2/Na_2O)
Sodium Silicate	1.40	41.25	9.44	28.28	3.00

blast furnace slag (GGBS) dosage were held constant at 0.5 and 500 kg/m^3, respectively. Na_2O amount originated from 12 M NaOH solution and sodium solution were summed and calculated as total Na_2O content of the mix. Total Na_2O and SiO_2 contents were summed and noted as activator solids. Binder content was calculated as the sum of slag and activator solids. Total mixture water amount was calculated as the total water used in 12 M NaOH solution plus the water in the sodium silicate and the extra water added to supply the liquid to binder ratio of 0.5.

Sixteen different mortar mixtures were formed with 4 different Na_2O/slag ratios (3, 6, 9, 12%) and 4 different moduli (SiO_2 / Na_2O) ratios (M=0.5, 1, 1.5, 2). Mixture proportions and several calculation details are given in Table 3. In this study, a constant sand to slag ratio of 2.70 is used. Sodium silicate, 12 M NaOH and water amounts are adjusted for 16 different mixture types so that activator modulus and Na_2O / slag ratios are satisfied. Finally, water amounts are adjusted to have a liquid binder ratio of 0.5 for all mixes.

2.2.3. Preparations of specimens

Dry mixture was formed by mixing slag with aggregates and alkali activator solution was added gradually in 3 minutes time and mixing was continued for one more minute. Alkali activator solution was formed 24 hours prior to mortar casting by adding NaOH in powder form, sodium silicate solution and also the mixing water.

Mixture compositions are given in Table 3. 40x40x160 mm specimens were casted from all sixteen mixtures. Specimens were placed in water tanks which were held at constant temperature of 20±1 °C. All specimens were casted in steel moulds and held in laboratory conditions. Subsequently specimens were demolded 48 hours after mixing and placed in curing chambers.

2.3. Testing Method

2.3.1. Flowability of mortar mixtures

Flowability tests were conducted to determine the effect of alkali activated slag mortars. There were no coarse aggregates in mixtures so that the workability of the mixtures was tested according to ASTM C1437-15 which is the standard for flow analysis of hydraulic cement mortar (ASTM International 2015). Tests were conducted on a jumping table with a steel ring mold that was filled with two layers of mortar mixture to its full height of 50 mm and both layers were tamped 20 times. When mixture was released by

Table 3. Mixture proportions and parameters.

		NS3M05	NS3M10	NS3M15	NS3M20	NS6M05	NS6M10	NS6M15	NS6M20	NS9M05	NS9M10	NS9M15	NS9M20	NS12M05	NS12M10	NS12M15	NS12M20
Mix Proportions (kg/m^3)	Slag	500	500	500	500	500	500	500	500	500	500	500	500	500	500	500	500
	Water	222	216	207	199	198	181	164	147	171	146	120	94	145	111	76	42
	Sodium Silicate	27	53	80	107	53	106	159	212	80	160	239	319	105	212	320	425
	12 M NaOH Solution	50	40	30	20	100	80	60	40	149	119	89	59	199	159	119	79
	Natural Sand	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350
Calculated Values (kg/m^3)	Na_2O (from sodium silicate)	2.55	5.01	7.56	10.12	5.01	10.02	15.03	20.04	7.56	15.13	22.60	30.16	9.93	20.04	30.26	40.18
	Na_2O (from NaOH solution)	12.57	9.93	7.54	5.03	25.01	19.98	14.96	9.93	37.45	29.91	22.37	14.83	50.02	39.96	29.91	19.86
	Na_2O (total)	15.12	14.94	15.10	15.14	30.02	30.00	29.99	29.97	45.02	45.04	44.97	44.99	59.95	60.01	60.17	60.04
	SiO_2	7.63	14.99	22.62	30.25	14.99	29.97	44.96	59.94	22.62	45.24	67.58	90.20	29.69	59.94	90.48	120.17
	Total water	272.10	275.69	277.09	279.14	298.23	300.72	302.71	305.20	321.49	326.04	328.96	332.51	344.84	350.44	355.67	360.03
	Activator Solids	22.75	29.93	37.72	45.40	45.01	59.98	74.95	89.92	67.64	90.28	112.54	135.19	89.64	119.95	150.65	180.21
	Total Binder	522.75	529.93	537.72	545.40	545.01	559.98	574.95	589.92	567.64	590.28	612.54	635.19	589.64	619.95	650.65	680.21
Parameters	Na_2O/slag	3.0%	3.0%	3.0%	3.0%	6.0%	6.0%	6.0%	6.0%	9.0%	9.0%	9.0%	9.0%	12.0%	12.0%	12.0%	12.0%
	Liquid/ binder	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50	0.50
	Modulus	0.50	1.00	1.50	2.00	0.50	1.00	1.50	2.00	0.50	1.00	1.50	2.00	0.50	1.00	1.50	2.00

removing the mold, jumping table was dropped for 25 times in a period of 15 seconds. Two sets of flowability tests were conducted for all specimens. Diameters of the mortar flows were measured from two vertical directions and average of these values were calculated. Flow of the mixtures were determined by dividing the findings by the initial diameter of the mold and expressed in percentage form.

All mortar specimens were tested under both compressive and flexural loads. Three-point loading procedure was applied according to ASTM C 348-20 with a span length of 100 mm with a displacement controlled loading rate at a speed of 0.5 mm/min at different curing intervals of 7 and 28 days (ASTM International 2020). Subsequent to three point loadings, specimens were cracked at midspan and separated into two pieces where both pieces were used for compressive strength tests according to ASTM C 349-18 (ASTM International 2018). Compressive strength tests were applied at a compressive loading test device which was force controlled at a rate of 0.5 MPa/sec. Appropriate loading fixture was implemented for compressive testing to apply accurate loading on the 40x40 mm surfaces.

3. Results

3.1. Flowability Results of Mortar Mixtures

Mixtures were diagnosed by means of workability when being casted into steel molds and it has been seen that all mixtures were in highly workable state hence there were no need for superplasticizers. Also workability conditions were measured by flowability tests. Although all mixtures were diagnosed as workable, some differences were observed from flowability results which are illustrated in Figure 1. Flowability values were calculated as the percentage to initial diameter size of 100 mm.

According to test results, highest flowability result was 154% (NS3M10) whereas lowest value was 133% (NS12M20). It is observed that low alkali content specimens had better workability results.

3.2. Compressive Strength Results

The compressive strength results were obtained from the two halves of the specimens that were cracked under flexural loadings. Since there were 3 specimens from each set tested under flexure, six specimens were tested for compressive strength results. Therefore, a good set of results were obtained and given in Table 4.

The highest compressive strength was obtained for specimens with 9% Na_2O content where activator modulus is highest (M=2). Second best result from these findings were found for 6% Na_2O specimens with a modulus of 2 whereas specimens with 9% Na_2O content and modulus of 1.5 was very close to these results. So these results show that

Table 4. Compressive and flexural strength results.

	Compressive Strength (MPa)		Flexural Strength (MPa)	
	7-day	28-day	7-day	28-day
NS3M05	24.13	32.61	3.38	5.24
NS6M05	32.14	43.44	4.06	6.63
NS9M05	39.76	55.22	4.68	7.87
NS12M05	39.60	55.00	4.26	7.43
NS3M10	29.63	46.30	4.06	7.27
NS6M10	37.25	55.60	4.58	8.28
NS9M10	43.82	63.50	5.26	9.23
NS12M10	43.56	60.50	5.03	8.46
NS3M15	23.81	44.10	3.00	6.42
NS6M15	43.01	66.16	5.11	9.51
NS9M15	51.12	73.03	6.34	10.97
NS12M15	48.11	67.76	5.63	9.59
NS3M20	20.69	40.57	2.58	5.71
NS6M20	44.80	73.44	5.36	10.64
NS9M20	55.48	78.14	6.94	11.84
NS12M20	49.80	66.40	6.05	9.77

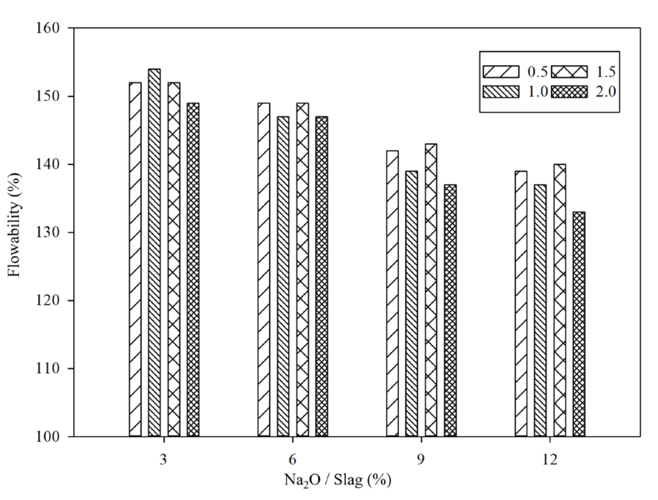

Figure 1. Flowability test results with different Na_2O content and activator modulus.

there is a net combined effect of both Na_2O/slag ratio and activator modulus.

Lowest compressive strength results were recorded for NS3M05 mixtures which have the lowest Na_2O/slag ratio and lowest modulus. NS3M05 specimens have shown very low strength with respect to all other mixtures (Figure 2). Specimens with 3% Na_2O/slag ratios showed generally the lowest results. For these specimens, it can be seen that increasing the activator modulus have little effect on the compressive strength except NS3M05 specimens which is most probably because of the low Na_2O content. When 6% and 9% Na_2O / slag ratio specimens were investigated, it is seen that compressive strength increased with the increasing activator modulus with a linear good fit. However, this behavior was not seen for specimens with 3 and 12% Na_2O content.

When exact values of 7-day results were compared, specimens with the lowest Na_2O content and highest modulus (NS3M20) were found to be weaker than all the other mixtures which contradicts with the 28-day results where weakest mixtures were found to be NS3M05 (Figure 2). When all mixtures are compared, effect of modulus contradicted only specimens with 3% of Na_2O content. For all the other mixture types, increasing modulus content increased the 7-day compressive strength results. Maximum 7-day compressive strength was obtained for specimens with 9% Na_2O content with an activator modulus of 2.

When analyzing the early strength results, 7-day results are normalized with the 28-day results and given in Table 5. Since specimens had quite different 28-day results so that comparing their 7-day results are found to be more expressive when analyzed with this point of view.

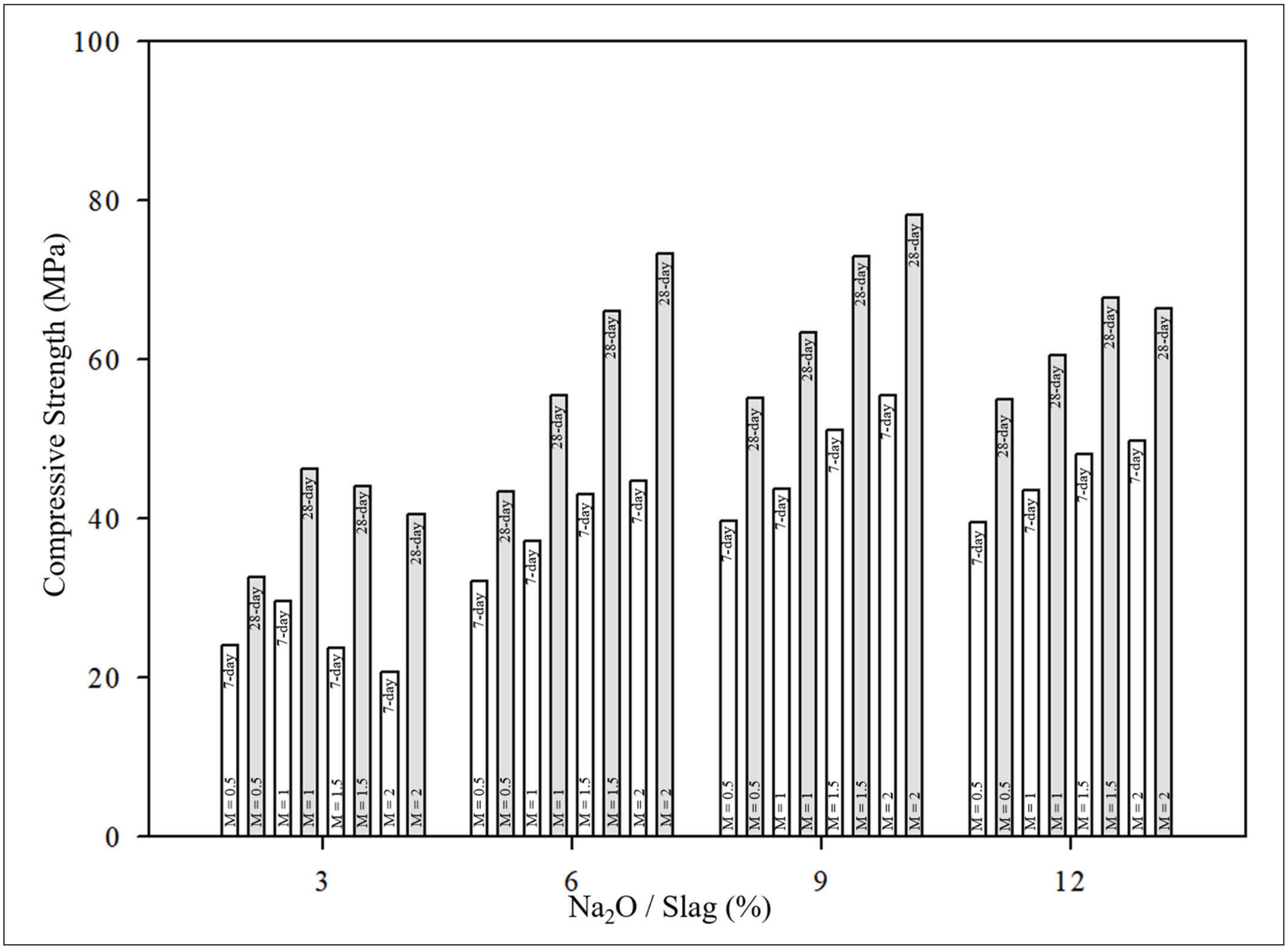

Figure 2. Comparison of 7 and 28-day compressive strength results for all mixtures.

Table 5. 7-day results normalized with 28-day results.

7-day / 28-day ratios		Activator Modulus (Si_2O/Na_2O)			
		0.5	**1**	**1.5**	**2**
Na_2O / Slag (%)	3	0.74	0.64	0.54	0.51
	6	0.74	0.67	0.65	0.61
	9	0.72	0.69	0.70	0.71
	12	0.72	0.72	0.71	0.75

Highest difference between 7-day and 28-day results were recognized for mixtures with higher modulus (M_s=2). For mixtures with modulus of 0.5, there was no important difference increase between early and 28-day strengths. Importance of the difference between the early and 28-day strength increases with the increasing activator modulus. Accordingly, it can be concluded that early strength evolution is found to be limited for higher modulus mixtures. And also it may be added that for higher percentages of Na_2O content, early strength results are more stable for various modulus levels.

Increase in compressive strength results with the increasing activator modulus can be directly related to SiO_2 content increasing and especially it is observed that SiO_2 content becomes more meaningful when alkali level (Na_2O content) of the mixture is higher.

3.3. Flexural Strength Analysis

Flexural strength tests are made on triplicates of 40x40x160 mm specimens loaded under three-point loadings. Flexural strength results are given in Table 4 and also depicted in Figure 3.

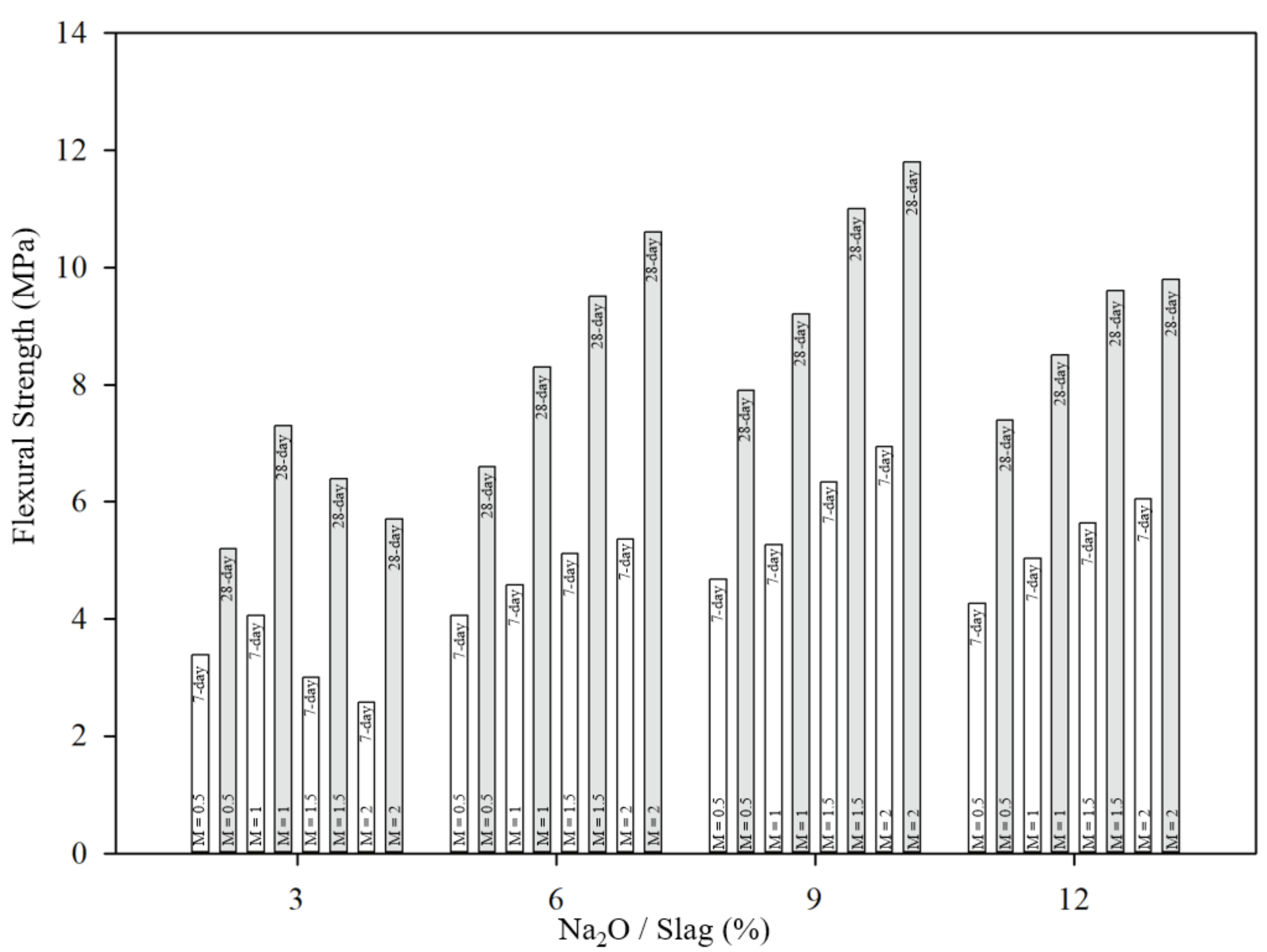

Figure 3. Comparison of 7 and 28-day flexural strength results for all mixtures.

When 28-day results are investigated, it can be seen that NS3M20 specimens showed the worst flexural performance. Similar results were also seen for compressive strength specimens. Best flexural performance was seen for mixtures with 9% Na_2O content with higher modulus values (1.5 and 2). When best performances are considered, it is seen that at higher Na_2O contents, advantage of using higher modulus activator diminishes hence NS12M15 is very close to NS12M20 results. Moreover, it is seen that flexural performance for all Na_2O contents other than 3%, increases with the increasing activator modulus. However, mixtures containing 12% Na_2O are relatively limited in flexural performance.

When 7-day results are observed it may be deduced that performance of 3% Na_2O content specimens are highly different with respect to 28-day results. Effect of modulus is different for 7-day results with respect to 28-day results. All other findings are very close to 7-day compressive strength results.

4. Discussion

Alkali activation of slag mortars is a highly applicable solution to replace cement in concrete applications for a sustainable approach. Optimization of the alkali activators are significantly important when designing the alkali activated mix proportions. In this study, two types of activators are used together with different proportions to satisfy the predefined parameters: (i) activator modulus and (ii) Na_2O/slag ratio. Four different moduli (SiO_2/Na_2O) (0.5, 1, 1.5, 2) and also and 4 different Na_2O / slag ratios (3, 6, 9, 12%) were selected to conclude on the effects of these parameters on mechanical properties of alkali activated slag mortars. From the flowability results it may be deduced that workability of specimens were not much affected with varying parameters however difference between results may be analyzed nevertheless. Specimens with higher alkali content was found to be adversely affected in workability. Higher amount of sodium silicates provides a higher early strength thus decreasing workability with quick reaction and rapid hardening (Bakharev et al. 2000). Slag particles were observed to affect the workability quicker when activation progress is more efficient (Gao et al. 2015). In this study, activation progress is assessed to be more related to activator solids content rather than just alkali content. Correspondingly, modulus of the specimens should be optimized for proper workability assessments. When the averages of specimens with same Na_2O content are compared with the averages of mixtures with same modulus, it may be noticed that effect of Na_2O content is more pronounced whereas effect of modulus seems more impotent. Moreover, for specimens with modulus of 1.5 it is seen that flowability results were found to be more close to each other for all Na_2O contents which may be concluded as a more robust design value for alkali activated slag mixtures in workability concept.

When mechanical properties were considered, flexural and compressive strength results were found to have similar trends. However, in a study authors declared that flexural strength is more prone to cracks with respect to compressive strength. They reported that there was not a remarkable change in flexural strength with the increasing M_s values. Same authors also declared that early strength values for both compression and flexural strength were observed to higher for lower modulus specimens as in accord with the findings of this study (Aydin and Baradan 2014). In another study, researchers reported higher early strength with lower modulus specimens and added that maximum strength results at later ages were found to be better for higher modulus specimens (Bakharev et al. 1999). Alkali activation process without sodium silicate activator was halted by a fast initial reaction of NaOH activated slag thus creating a dense CSH formation with smaller amount of chemically bound water. This phenomenon causes higher porosity and accordingly lower mechanical strength. Use of two activators simultaneously were significant and increasing amount of solid activators were dominant in mechanical strength evolution. Single NaOH activation produced a microstructure constructed with dense particles with connected pores whereas simultaneous use of activators supplied better homogeneity. (Ben Haha et al. 2011). Mechanical strength of the alkali activate mortars is altered by the pore volume and hydration products. Microcracks in the formation of the mortars were also significant when mechanical properties were considered (Zhang et al. 2020).

Specimens with higher alkali content attributed better results and activator modulus should be optimized by adjusting the sodium silicate amount in the mix design. Both Na_2O and soluble Si contents act an important part in the development mechanical properties of alkali activated mixtures (Zhang et al. 2020). Compressive strength results increased with the increasing modulus values which may be correlated to decreasing Ca/Si ratio thus binding ability of C-S-H particles was improved. Therefore, more compact inner structure was formed with the increasing modulus (Aydin and Baradan 2014, Bakharev et al. 2000).

Earlier strength values for both compressive and flexural results were found to be higher for low modulus specimens in contrast to later ages as was also reported by different studies (Aydin and Baradan 2014, Bakharev et al. 1999).

NaOH activated alkali reactions can be parted into three segments: First stage consists of the breaking of the bonds of Ca-O, Si-O-Si and Al-O-Al and also Al-O-Si bonds in the binder material. In the second stage, polycondensation occurs which is a results of the precipitation of the products in the solution. Existence of higher soluble Si amount create a difference of hydration products till exhausted (Zhang et al. 2020).

Conclusions derived from this study may be summarized as:

- Mechanical properties were found to be affected from both parameters (Na_2O/slag ratio and activator modulus). Both parameters are found to be significantly related to both compressive and flexural strength results.
- Best combination of these two parameters were found to be 9% alkali content with a modulus of 2.
- Mechanical properties except 3% alkali content specimens were found to be improved with the increasing activator modulus. However, this effect started to diminish for 12% alkali content specimens which is the maximum alkali level used in this study.
- Early strength values were better for lower modulus specimens with respect to 28-day strength values.
- Flow characteristics of mixtures at the fresh state were adversely affected by the increasing the content of the activator solids although liquid to binder ratios were the same for all mixtures.

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