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Design of Alkali-Activated Ladle Slag Mortar Using Taguchi Method

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Abstract – This study investigates the feasibility of utilizing ladle furnace slag as a sole binder in alkali-activated mortar. The Taguchi method for design of experiments was used to design, analyze, and optimize the mixture proportions of alkali-activated ladle slag mortars. The five factors considered in the design included ladle slag content (LS), alkali-activator solution-to-binder ratio (AAS/B), sodium silicate-to-sodium hydroxide ratio (SS/SH), sodium hydroxide solution molarity (M), and crushed sand-to-dune sand replacement ratio (CSR). With four design levels, a corresponding L_{16} orthogonal design matrix was developed. The targeted design criteria were the 7-day compressive and tensile strength, workability, and initial setting time. Analysis of variance results showed that mechanical properties were equally impacted by LS, SS/SH, and CSR. Conversely, the LS, AAS/B, and SS/SH contributed the most to the workability and setting time. Using Taguchi method, three mixes were proportioned based on the analyzed data to optimize each of the design criteria. Validation of the optimized mixes provided evidence of the applicability of the Taguchi method to design alkali-activated ladle slag mortars with a margin of error of less than 9%.

Keywords**:** ladle slag; alkali-activated; mortar; performance evaluation; Taguchi method.

1. Introduction

Ladle slag is an industrial by-product of the secondary refinery treatment of steel. The steel production process generates around 80 kg of ladle slag per ton of refined steel produced [1]. With the ever-increasing global demand for steel, the current annual generation of ladle slag is approximately 150 Mt [2]. As it is typically stockpiled or landfilled, its disposal is considered a major environmental concern. To mitigate its adverse impact on the environment, various studies have been conducted to evaluate the applicability of using it as a construction material. After being slowly air-cooled, the primary mineral phase formed in ladle slag is γ-C_2S [3]. The low hydraulic property of ladle slag and stability of its hydration products have been investigated in the past [4]. Its use as a sole precursor in alkali-activated mortars has resulted in impressive mechanical performance [5, 6]. In turn, Wang et al. [7] examined the effect of the alkali agent concentration and alkaline-to-binder ratio on the mechanical properties of alkaline activated ladle slag mortars. An enhancement in shrinkage resistance and strength development was noted when saturated limewater was used as a curing regime.

Despite the promising results of alkali-activated ladle slag mortars in the literature, the effect of mix design parameters on the performance of these mortars has received little attention. Indeed, the complex integration of various constraints in the design of experiments can lead to thousands of extensive experimental runs to capture the effect of each factor for a particular response. However, it has been proven that a robust statistical approach, such as a Bayesian network, could predict the behavior and define the critical mix design parameters [8]. Also, the statistical approach of Taguchi design of experiment can be employed efficiently. This fractional factorial method uses predefined orthogonal arrays to accommodates all the required levels of the investigated parameters within a limited number of experimental runs [9, 10]. Yoyok Setyo and Sabarudin Bin [11] investigated the applicability of the Taguchi method in designing and analyzing the properties of self-compacting concrete. Their investigation included six control factors with three levels to optimize four primary responses: slump flow, flow time, V-funnel, L-box, and segregation resistance. Similarly, Sharifi, et al. [12] found a substantial improvement in the performance of self-compacting concrete when optimizing the mix design using Taguchi method. Other studies were also able to optimize the rheological and mechanical properties of alkali-activated mortars [10, 13].

This study adopts the Taguchi method for design of experiments in designing and optimizing the mixture proportions of alkali-activated ladle slag mortars. The optimization was carried out based on superior workability, setting time, compressive strength, and splitting tensile strength. It is anticipated that the Taguchi method will develop an in-depth understanding of different design parameters on the properties of the alkali-activated ladle slag mortar as a means of promoting the use of ladle slag to endorse sustainable construction.

2. Experimental Work
2.1. Materials
The as-received ladle slag (LS) was locally sourced from Emirates Steel. It is classified as a high reactive material as the calculated basicity using oxide ratios of $(CaO + MgO)/(SiO_2 + Al_2O_3)$ was found to be 1.66. It has an average particle diameter of 122 µm, surface area of 648.9 m^2/kg, and D_{10}, D_{50}, and D_{90} of 4.472, 30.56, and 232 µm, respectively. Natural crushed sand passing sieve #16 (in compliance with ASTM C778 [14]) served as the conventional fine aggregate. It was replaced with different percentages of locally abundant desert dune sand to promote sustainability. For this reason, all mixes had some replacement of crushed sand by dune sand. The properties and microstructure of these aggregates can be found in other work [15].

A combination of sodium hydroxide (NaOH, SH) and sodium silicate (Na_2SiO_3, SS) was used to formulate the alkaline activator solution (AAS). The NaOH was in the form of flakes with 99.5% purity and 1610 kg/m^3 density, whereas the sodium silicate was classified as grade N with Na_2O:SiO_2:H_2O mass ratio of 8.9:28.6:62.5 and density of 1380 kg/m^3. A commercially obtained superplasticizer (polycarboxylic ether polymer) with a specific gravity of 1.115 was used to maintain adequate flow of the mortar.

2.2. Taguchi Method
The experimental method sets predefined factors and levels to design the minimum experimental runs required to capture the levels' effects on the preferred quality criteria. The factors and levels are used in Eq. (1) to evaluate the degree of freedom (DOF) by which the minimum required Taguchi orthogonal array will be defined. As a total of five factors and four levels are considered in this study, the DOF obtained is 15. As such, the L_{16} orthogonal array was employed to define the mixing proportions in this study.

$$\mathrm{DOF} = (\mathrm{L} - 1)\mathrm{F} \tag{1}$$

Where L is the number of levels and F is the number of Factors.

In the early 1950s, Dr. Genichi Taguchi established a method based on Gauss's quadratic function for quality estimation. The essence of this method is that it eliminates the sources of error that could affect the responses by considering them noise factors. The optimization technique of the Taguchi method is based on reducing the noise factor affecting specific design responses through the calculation of signal-to-noise ratios (S/N). Three S/N ratios are used to optimize the design responses, larger-is-better, smaller-is-better, and nominal-is-the-best. As the required responses are to be maximized, Eq. (2) conforming larger-is-better S/N ratio was selected in this investigation. Taguchi predicted results are calculated through Eq. (3).

$$S/N = -10\times \log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Y_i^2}\right) \tag{2}$$

Where Y_i represents the responses to be maximized.

$$\eta = \eta_m + \sum_{i=1}^{f}(\eta_i - \eta_m) \tag{3}$$

Where η_m is the total mean value of S/N ratios representing all experimental results, f is the factor number, and η_i is the mean of S/N ratio conforming to targeted factor levels.

After defining the factors of interest in generating the mortar mixes, trials were conducted to define the four levels of each factor, as listed in Table (1). The factors are ladle slag content (A), molarity of sodium hydroxide solution (B), AAS-to-binder ratio (C), SS-to-SH ratio (D), and crushed sand replacement (CSR, E). The final design matrix was generated by mapping Table (1) into the orthogonal array. As a result, 16 experimental runs were created and are listed in Table (2).

Table 1: Factors and levels for mixture proportioning of alkali-activated ladle slag mortar.

Factors	Level-1	Level-2	Level-3	Level-4
A: Binder content (kg/m^3)	500	550	600	650
B: Molarity of NaOH solution (M)	8	10	12	14
C: AAS/binder ratio	0.45	0.50	0.55	0.60
D: SS/SH ratio	1.0	1.5	2.0	2.5
E: Crushed sand replacement (CSR) (%)	25	50	75	100

Table 2: Mixture proportions of alkali-activated ladle slag mortar mixes.

Mix No.	LS (kg/m^3)	NaOH (M)	AAS/Binder	SS/SH	CSR (%)
CM-1	500	8	0.45	1.0	25
CM-2	500	10	0.50	1.5	50
CM-3	500	12	0.55	2.0	75
CM-4	500	14	0.60	2.5	100
CM-5	550	8	0.50	2.0	100
CM-6	550	10	0.45	2.5	75
CM-7	550	12	0.60	1.0	50
CM-8	550	14	0.55	1.5	25
CM-9	600	8	0.55	2.5	50
CM-10	600	10	0.60	2.0	25
CM-11	600	12	0.45	1.5	100
CM-12	600	14	0.50	1.0	75
CM-13	650	8	0.60	1.5	75
CM-14	650	10	0.55	1.0	100
CM-15	650	12	0.50	2.5	25
CM-16	650	14	0.45	2.0	50

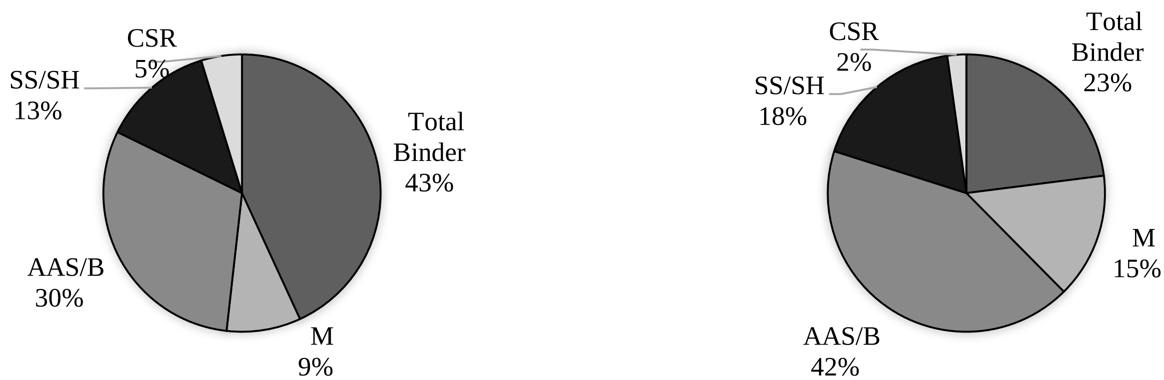
2.3. Sample Preparation and Testing Methods

The alkaline activator solution was prepared by mixing NaOH flakes with a specific amount of water to obtain the required molarity. The cooled NaOH solution was then mixed with Na_2SiO_3 and left for up to 24 hours until the acquired AAS reached ambient temperature. To minimize the work and energy required to process the as-received ladle slag, it was only sieved to pass a 150-μm sieve; no further crushing or magnate separation was carried out. Mixing was conducted under ambient conditions (23±1°C temperature and 50±5% relative humidity), whereby the ladle slag and fine aggregates were mixed for 2-3 minutes followed by the gradual addition of the AAS. The fresh mortar properties, including flow table and initial setting time, were conducted as per ASTM C1437 [16] and ASTM C191 [17], respectively. The produced mortar was cast in 50x50x50 mm cubes and cylinders of 100x200 mm for compressive and splitting tensile strength. Samples were covered with a plastic sheet for the first 24 hours and then demoulded and cured in ambient conditions until the required testing age. Compressive and tensile strength were performed at the age of 7 days in compliance with ASTM C109 [18] and ASTM C496 [19].

3. Results

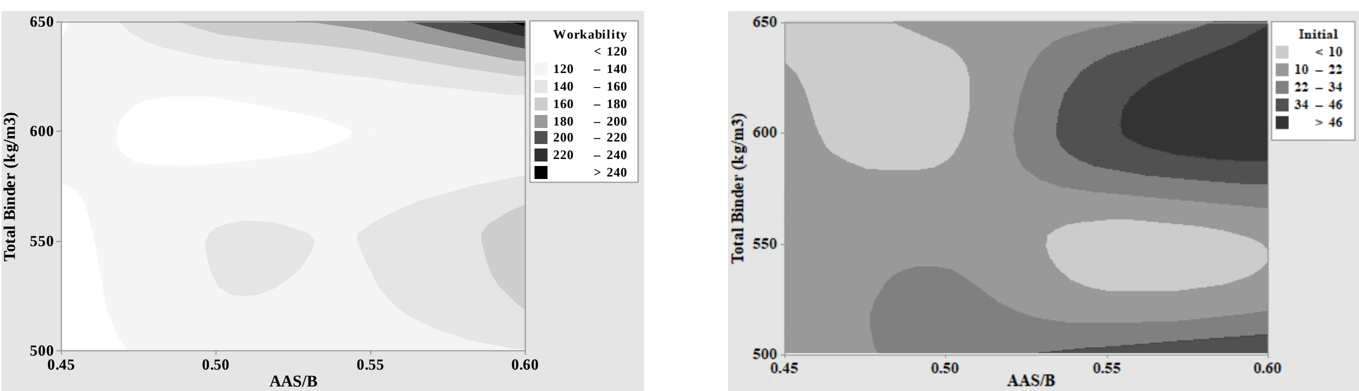
3.1. Fresh Properties

Analysis of variance (ANOVA) was implemented to define the contribution percentile of each factor on the required response. As illustrated in Fig. (1-a), the main contributors to the workability property, characterized by the flow table test, were the total binder with 43% and AAS/B with 30%. Conversely, the least influential factors were the SH molarity (M) and the CSR with 9% and 5%, respectively. In the meantime, the SS/SH ratio showed a moderate contribution of 13%. The main contributors to the workability were further utilized to generate contour plots shown in Fig. (2-a). The results show that the increase in binder content and AAS/B ratio led to an increase in the workability of the alkali-activated ladle slag mortar. Similar findings were reported for alkali-activated ground granulated blast furnace slag mortars [20].



(a) (b)

Fig 1: ANOVA contribution factors for (a) workability and (b) initial setting time.



(a) (b)

Fig 2: Contour plots of the total binder content against AAS/B ratio for (a) workability and (b) initial setting time.

Moreover, for the initial setting time in Fig. (1-b), the most contributing factors were the AAS/B ratio with 42% and binder content with 23%. Meanwhile, the SH molarity (M) and SS/SH ratio proved to be more impactful on the initial setting time than the workability. Yet, a similar trend to the workability was noticed in Fig. (2-b), where the increase in binder content and AAS/B ratio increased the initial setting time. The relatively low surface area and large average diameter size of the used ladle slag affected the reactivity, particle packing, and dissolution in the mix, leading to an increase in the initial setting time [21]. Nevertheless, the setting time values were comparable to those of alkali-activated ground granulated blast furnace slag counterparts, owing to the similar morphologies of the precursor materials [22, 23].

3.2. Mechanical Properties

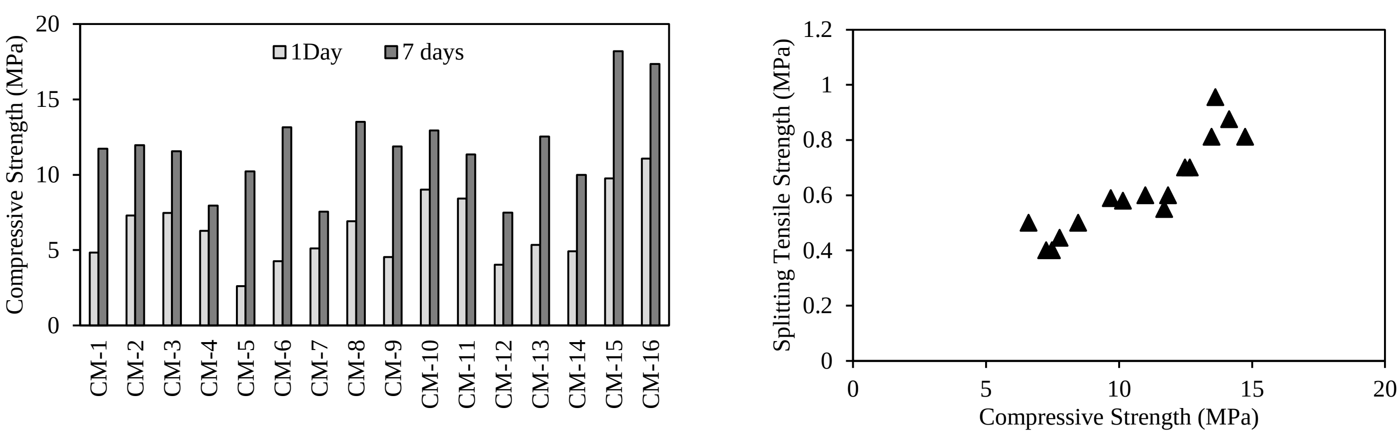
The main contributor to the mechanical properties was the total binder, SS/SH, and CSR, all with nearly 28% contribution percentile, as per the ANOVA analysis summarized in Table (3). Contrarily, the contributions of the AAS/B and SH molarity (M) factors were about 15 and 1%, respectively. This shows that the AAS/B and SH molarity (M) had limited impact on the mechanical properties.

Table 3: ANOVA contribution factors for compressive strength.

Factor	Contribution
Total Binder	28.4%
NaOH Molarity (M)	0.8%
AAS/B	14.7%
SS/SH	28.2%
Crushed Sand Replacement	27.9%
Error	---
Total	100.0%

As presented in Fig. (3-a), the compressive strength exhibited a significant increase from 1 to 7 days, owing to the high degree of reactivity of the ladle slag. A similar trend was noted in other work [24]. The highest compressive strength of 18.2 MPa (CM-15) corresponds to a total binder of 650 kg/m^3, SS/SH ratio of 2.5, AAS/binder of 0.50, SH molarity of 12 M, and CSR of 25%. For all alkali-activated ladle slag mortar mixes, the standard deviation and coefficient of variation were in the ranges of 0.6-2.1 MPa and 2.0-9.1%, respectively.

Furthermore, the splitting tensile strength experienced a similar pattern to that of the compressive strength, as shown in Fig. (3-b). For mixes having a compressive strength less than 12 MPa, the splitting tensile strength did not exceed 0.6 MPa, while mixes with higher compressive strength could reach 1 MPa. The increase in the binder content and silicates content promotes higher dissolution of ladle slag, i.e., calcium ions, and improves the reaction efficiency, thereby increasing the strength [25]. In the meantime, maximizing the crushed sand content, i.e., minimizing the CSR, resulted in higher strength due to the angular shape of the crushed sand, which provides a better bond within the alkali-activated matrix. The standard deviation of these mixes ranged between 0.06 and 0.15 MPa, while the coefficient of variation was in the range of 2.1-9.5%. These values provide evidence to the relatively low dispersion and high precision of the results, while also being lower than the recommended margins of error of 10 and 20% for reliable statistical analysis of the splitting tensile and compressive strengths [26,27].

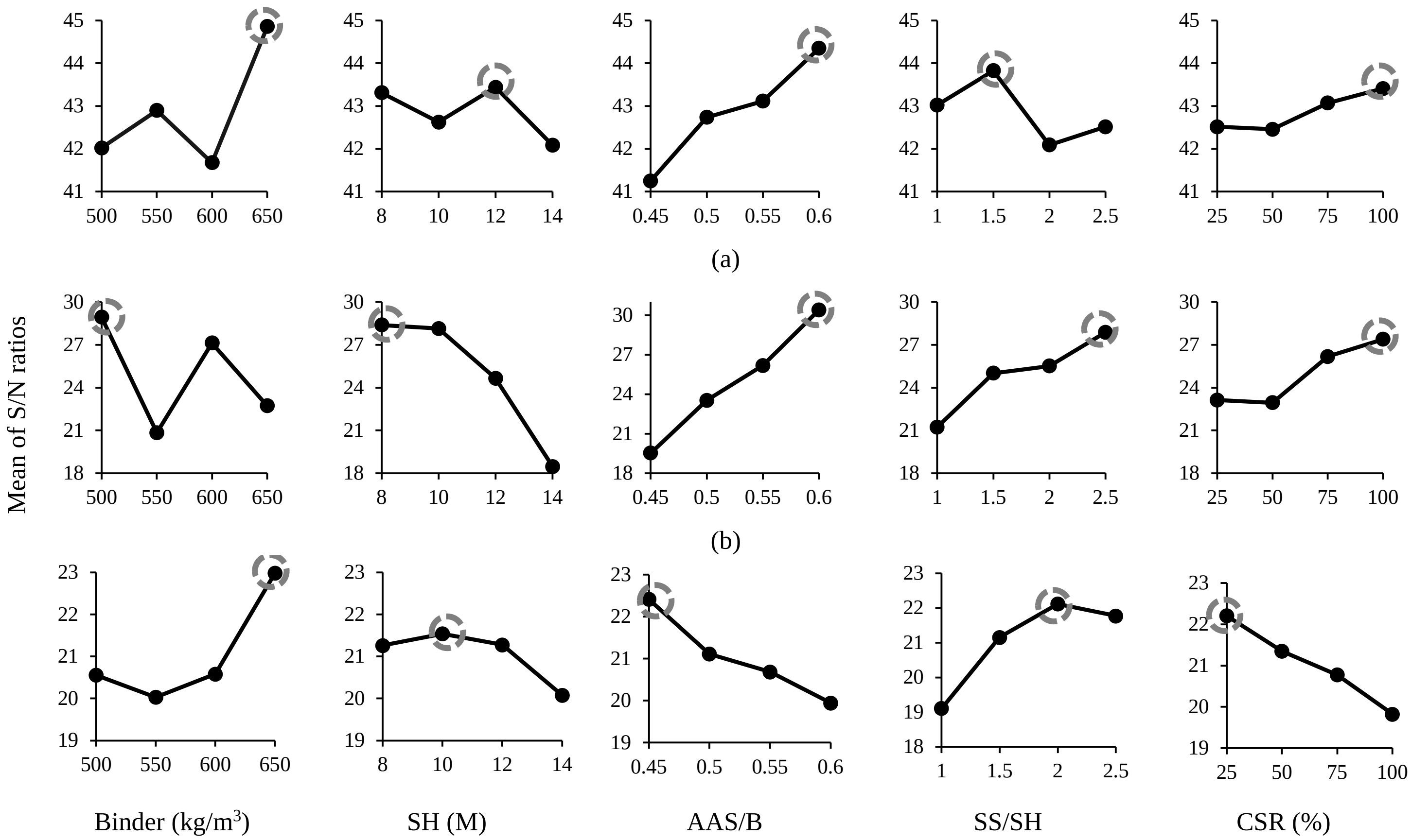


(a) (b)

Fig 3: (a) Compressive strength results and (b) splitting tensile strength as a function of compressive strength of alkali-activated ladle slag mortar.

3.3. Taguchi Optimization Results

Corresponding to Eq. (2), three different models were developed to optimize the mechanical properties, workability, and initial setting time of alkali-activated ladle slag mortar. The maximum mean S/N ratios calculated for each response provide the optimized proportion for the given quality criterion. As illustrated in Fig. (4-a), the mean S/N ratios for the workability was 650 kg/m^3 of ladle slag, 12 M of SH molarity, 0.6 for AAS/binder ratio, 1.5 for SS/SH ratio, and 100% of CSR. As for optimizing the initial setting time in Fig. (4-b), the total binder content, SH molarity, AAS/binder ratio, SS/SH ratio, and CSR were 500 kg/m^3, 8M, 0.6, 2.5, and 100%, respectively. Conversely, in Fig. (4-c) the maximum strength per Taguchi optimization process corresponded to 650 kg/m^3 of binder content, 10 M of SH molarity, 0.45 for AAS/binder ratio, 2.0 for SS/SH ratio, and 25% of CSR.



(c)

Fig 4: Mean of S/N ratios of (a) workability, (b) initial setting time and (c) compressive strength.

Table (4) summarizes all the optimum mixes results, comparing the actual and predicted values. Analyzing the results of the first optimum mix for compressive and splitting tensile strength showed that the Taguchi prediction tool could accurately predict the actual values of these two properties. Indeed, the actual values varied by about 8.6% relative to the predicted counterparts. The prediction accuracy of the workability property was relatively higher, i.e., lower error, for the second optimum mix. In fact, the Taguchi tool predicted a flow diameter of 243 mm while that of the experimental test was 230 mm. However, the initial setting time elapsed longer than the predicted value of 60 minutes. Nevertheless, the third optimum mix, which aimed to prolong the initial setting time, offered the longest initial setting time among the tested mixes.

Table 4: Comparison between actual and predicted results.

Optimum mix	Designation	Actual	Predicted	Error (%)
1	Compressive Strength (MPa)	17.9	19.6	1.7 (8.6)
1	Tensile Strength (MPa)	1.27	1.16	0.11 (8.6)
2	Workability (mm)	230	243	13 (5.6)
3	Initial setting (min)	>60	60	-

4. Conclusions

Taguchi design of experiments was successfully implemented to design the mixture proportions of alkali-activated ladle slag mortar for optimum workability, setting, and mechanical performance. A total of five parameters, each with four different levels, were used to develop the experimental runs by using the Taguchi method. A total of sixteen mixes were utilized to capture the effect of each factor on the fresh and hardened mortar properties. The results showed that the binder content and the AAS-to-binder ratio were most impactful on the workability and initial setting time, as per the analysis of variance, while the CSR percentage showed the lowest contribution. On the other hand, the mechanical strengths were affected mainly by binder content, SS/SH, and CSR with a 28% contribution for each factor. Conversely, the molarity of SH did not have any effect on the mechanical strength. The Taguchi optimization process based on the average S/N ratios successfully designed and predicted the performance of optimal mixes with corresponding responses. The results proved that the Taguchi method is a robust model that could efficiently design and optimize alkali-activated mortar using ladle slag as the sole binder.

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