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Optimizing the Mix Design of Cementitious Composites Incorporating Volcanic Ash by Taguchi-TOPSIS Method

Jad Bawab[1], Hilal El-Hassan[1], Amr El-Dieb[1], Jamal Khatib[2]
[1]United Arab Emirates University
Al Ain, United Arab Emirates
202190186@uaeu.ac.ae; helhassan@uaeu.ac; amr.eldieb@uaeu.ac.ae.
[2]Beirut Arab University
Beirut, Lebanon
j.khatib@bau.edu.lb

***Abstract* –** This research investigates the potential of using volcanic ash (VA) as a partial replacement for cement in construction applications that require superior early-age properties. The study employs Taguchi methodology to optimize the cementitious composites by varying key factors, including binder content, water-to-binder ratio, dune sand substitution rate of natural sand, VA substitution rate of cement, and superplasticizer (SP) dosage. As each factor had four corresponding levels, an L16 orthogonal array was developed. Testing included the flow, setting time, and 1-day compressive strength. The Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS) was employed to optimize for the three factors simultaneously. The results indicate that the optimum mix consisted of a binder content of 400 kg/m^3, a water-to-binder ratio of 0.5, a dune sand substitution of 20%, a VA substitution of 20%, and an SP dosage of 0.75%. Accordingly, the optimum mix achieved a flow of 115 mm, a final setting time of 350 min, and a 1-day compressive strength of 18.1 MPa. This study demonstrates the optimum use of volcanic ash as a sustainable alternative to cement in construction applications with time-critical requirements.

***Keywords*:** volcanic ash, dune sand, cementitious composites, Taguchi method, TOPSIS, flow, setting time, compressive strength.

1. Introduction

Among many strategies to promote the sustainability of cementitious composites, using supplementary cementitious materials (SCMs) to replace a given amount of Portland cement is the most widely investigated and practiced in the industry [1]. Indeed, the clinker portion in cement has significantly decreased from 85% in 2003 to 77% in 2010, with estimations predicting a continuous reduction to reach 71% only [2]. This strategy offers reduced CO_2 emissions and concentrated resource consumption since Portland cement is the main contributor to energy consumption and CO_2 emissions in cementitious composites [3]. Currently, only three materials compose the vast majority of SCMs, namely fly ash, slag, and limestone [4].

Volcanic ash (VA) is an SCM with abundant availability worldwide but confined to active and dormant volcanoes' locations [4]. Found in regions such as Europe, Central America, Southeast Asia, and East and Central Africa, among others [5], the mineralogy and reactivity of VA are influenced by various types of magma from which they originate [6]. However, it is generally composed of (SiO_2), (Al_2O_3), and (Fe_2O_3) along with traces of other minerals [5]. Thus, owing to its pozzolanic properties, VA is a desirable addition to cementitious composites, and several studies investigated its use in concrete. Hossain and Lachemi [7] reported that the flowability of concrete increased when using VA by up to 20% by cement mass and then decreased beyond this substitution level. Celik et al. [8] reported that the initial and final setting times of cement mortar were remarkably extended due to the incorporation of VA as Portland cement replacement by up to 50% by mass. Meanwhile, Ghrici et al. [9] observed that the strength of mortar slightly decreased with the inclusion of VA by up to 30% by mass. Nevertheless, limiting the VA content to 10-20% by mass rendered a strength comparable to the reference mortar.

In general, previous investigations have shown that using VA as a partial replacement for Portland cement is viable. In fact, a few million tons of VA are already in use as a cement substitute at this time [4]. However, as further research is essential, several mix design parameters should be considered to achieve the cementitious composite's required performance.

This would include the binder content, water-to-binder ratio, type of aggregates, inclusion of chemical admixtures, etc. The presence of various affecting parameters would demand experimenting with a large number of mixes to identify the effect of each factor. Instead, Taguchi's optimization method can create orthogonal arrays that cut research and development costs by assessing a plurality of parameters with a minimal number of runs. Taguchi's method was effectively implemented in previous investigations to assess the properties of building materials considering several mix design parameters [10]–[13]. However, Taguchi's method is only applicable to one response at a time. Therefore, a few studies used the technique for order of preference by similarity to the ideal solution (TOPSIS) to convert the optimization problem from a single criterion to multiple criteria [14]–[16].

This study aims to find the optimum mix design parameters of cement mortar for high early-age performance. The parameters considered are the binder content, water-to-binder ratio, dune sand substitution rate of natural sand, VA substitution rate of cement, and superplasticizer (SP) dosage. For the evaluation of the performance of the cement mortar under study, its flow, final setting time, and 1-day compressive strength were tested. The results were utilized to assess the influence of each parameter on the mortar's properties. The experimental outcomes were also implemented using the Taguchi-TOPSIS method to find the optimum mortar mix that satisfied the three performance criteria. The optimum mortar was then validated. This study would contribute to the understanding of factors affecting the early-age properties of cementitious composites containing SCMs, in particular VA.

2. Materials and Testing

2.1. Materials

Type I ordinary Portland cement (OPC) was used as the main binder in this study. The OPC conformed to ASTM C150 [17]. Meanwhile, VA was used as a partial replacement of cement at 5, 10, 20, and 40% by mass. The VA was used without any processing. The VA can be classified as Class N pozzolan according to ASTM C618 [18], as more than 70% of its chemical composition is made up of SiO_2, Al_2O_3, and Fe_2O_3, as detected by X-ray fluorescence (XRF). Further, OPC had a specific gravity of 3.16 and Blaine fineness of 335 m^2/kg, whereas the VA had a specific gravity of 3.05 and Blaine fineness of 380 m^2/kg.

Two types of fine aggregates were used in the study: crushed sand and dune sand. The nominal particle size for both types is 2.36 mm [No. 8]. The crushed sand is crushed dolomitic limestone, having a specific gravity of 2.67 and a fineness modulus of 2.55. On the other hand, the dune sand had a specific gravity of 2.77 and a fineness modulus of 1.45. The dune sand was used as a partial substitution for the crushed sand at 5, 10, 15, and 20% replacement levels. It was used for sustainability reasons, as it is available in abundant quantities in different parts of the world. The crushed sand is well-graded, while the dune sand is narrow-graded, as reported earlier [19]. Additionally, a polycarboxylate ether-based SP was utilized to achieve acceptable flowability of the cement mortar mixes. The SP was used at dosages of 0.25, 0.50, 0.75, and 1.00% by the total binder mass. Tap water was also used.

2.2. Mix design

Taguchi's method was utilized to design cement mortar mixes. Five factors, each having four levels, were studied: the binder content at 350, 400, 450, and 500 kg/m^3, the water-to-binder ratio at 0.450, 0.475, 0.500, and 0.525, dune sand substitution rate of natural sand at 5, 10, 15, 20%, VA substitution rate of cement at 10, 20, 30, and 40%, and superplasticizer (SP) dosage at 0.25, 0.50, 0.75, and 1.00% by the total binder mass. Consequently, an L16 type B orthogonal array was developed, where all factors and levels are covered by sixteen cement mortar mixes only. The proportions of the developed mortar mixes are shown in Table 1. After detecting the levels of factors in each mix, the absolute volume method was utilized to develop the proportions of the mortar mixes.

Table 1: Cement mortar mixture proportions

Mix ID	Binder (kg/m^3)	w/b ratio	Dune sand (%)	VA (%)	SP (%)
1	350	0.450	5	5	0.25
2	350	0.475	10	10	0.50
3	350	0.500	15	20	0.75
4	350	0.525	20	40	1.00
5	400	0.450	10	20	1.00
6	400	0.475	5	40	0.75
7	400	0.500	20	5	0.50
8	400	0.525	15	10	0.25
9	450	0.450	15	40	0.50
10	450	0.475	20	20	0.25
11	450	0.500	5	10	1.00
12	450	0.525	10	5	0.75
13	500	0.450	20	10	0.75
14	500	0.475	15	5	1.00
15	500	0.500	10	40	0.25
16	500	0.525	5	20	0.50

The mixing and batching of the mortar mixes were performed in laboratory conditions having a temperature of 24 ± 3℃ and a relative humidity of 50 ± 5%. The dry materials were mixed for 1 minute in a pan mixer at low speed before adding half the amount of water, where mixing continued for another 1 minute. After that, the other half of the water, along with the SP, was gradually added to the mix, and mixing continued for another 1 minute at medium speed. After ensuring the mix was homogenous, a part was tested for flow and setting time, whereas the remaining quantity was poured into 50-mm cubic molds for strength testing. The cast specimens were left in laboratory conditions for 24 hours and covered with plastic sheets.

2.3. Testing methods

The flowability of the mortar mixes was determined following ASTM C1437 [20]. A mini-slump test apparatus was accordingly used to test the freshly mixed mortar for flow. The final setting time of the cement mortar mixes was tested according to ASTM 403 [21]. For the penetration resistance test, freshly mixed mortar was cast into 150-mm cubes, where at least ten readings were taken for each mix. The final setting time was the duration that elapsed between adding water to the dry materials and reaching a pressure of 27.5 MPa. Additionally, the compressive strength of mortars was performed on 50-mm cubes after 24 hours of mixing. For the test, ASTM C109 [22] was followed, and the average of three cubic specimens represented the compressive strength value of a mix.

3. Results

3.1. Flow

Figure 1 presents the flow of the cement mortar mixes. The flow ranged between 100 and 210 mm. Mixes 1, 2, 3, 4, 7, and 9 had the lowest flow. On the other hand, mixes 14 and 16 had the highest flow. In general, achieving a high flowability of more than 200 mm would require the mortar mix to contain a binder content of 450-500 kg/m^3, a water-to-binder ratio of 0.475-0.525, dune sand substitution rate of natural sand of 5-15%, VA substitution rate of OPC of 5-20%, and an SP dosage of 0.50-1.00% by binder mass. Using a high binder content and high water-to-binder ratio increased the flow. Generally, this would facilitate the availability of more paste and free water in the mix, thus contributing to inter-particle lubrication [23]. Nevertheless, limiting the dune sand content between 5 and 15% was necessary to maintain high flow. It was previously reported that dune sand restricts water mobility and thus decreases the flowability of the associated mortar mix [24]. Similar to the dune sand content, the VA content was to be constrained to 20%. This is in agreement with a different investigation that observed a decrease in the flow when VA content exceeded 20% OPC mass [7]. Further, setting the dosage of SP at 0.50-1.00% effectively reduced the water demand of the mortar mixes and consequently increased their flow. The significant role of SP in the flow of cementitious composites was reported earlier in another study [25].

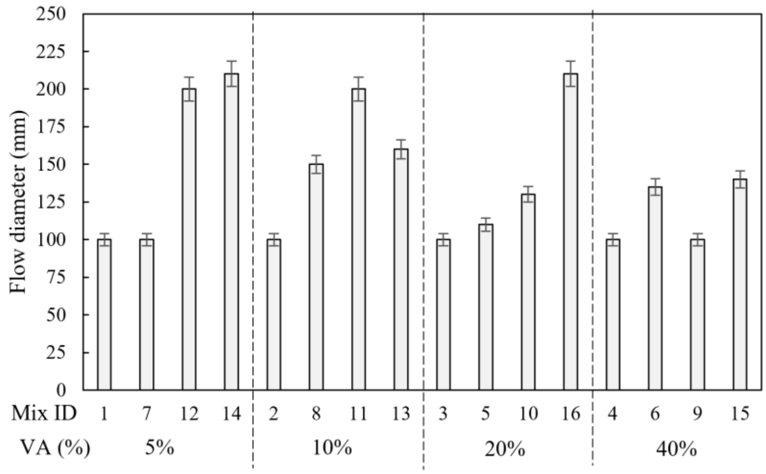

Fig. 1: Flow diameter of the cement mortar mixes.

3.2. Final setting time

The final setting time of the mortar mixes is illustrated in Figure 2. Mix 2 had the shortest final setting time at 250 min, while mix 14 had the longest final setting time at 800 min. Achieving a final setting time of less than 400 min would require a binder content of 350-500 kg/m^3, a water-to-binder ratio of 0.450-0.525, a dune sand content of 5-20%, VA content of 5-40%, and SP dosage of 0.25-0.75%. Therefore, an adequate final setting time of less than 400 min is achievable at any condition except for a high SP dosage of 1.00%. Indeed, a lower binder content and water-to-binder ratio would shorten the final setting time due to their influence on the hydration kinetics of the cement mortar [26]. Limiting the SP content also mitigates its effect on the final setting time, as SP retards the hydration of the binder [27]. On the contrary, the increase in VA seemed to have shortened the final setting time. This might be due to the high Al_2O_3 content that leads to an accelerated hydration process [28]. The effect of dune sand on the final setting time was marginal.

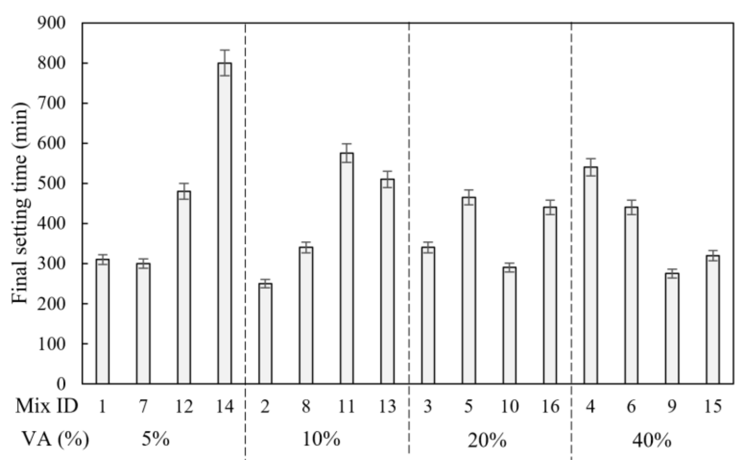

Fig. 2: Final setting time of the cement mortar mixes.

3.3. Compressive strength

Figure 3 illustrates the 1-day compressive strength of cement mortar mixes sorted by the VA substitution rate of OPC. OPC. The strength ranged between 2.4 and 14.1 MPa. Mix 1 achieved the lowest strength, whereas the highest strength was was recorded for mix 13. In general, a high 1-day strength exceeding 10 MPa was associated with a binder content of 400-400-500 kg/m^3, a water-to-binder ratio of 0.450-0.500, dune sand substitution rate of crushed sand of 10-20%, a VA substitution rate of OPC of 5-20%, and an SP dosage of 0.25-1.00%. An increase in the binder content would contribute to to enhanced strength development, mainly due to the formation of more hydration products and consequently lowering the porosity of the cement mortar [28]. Limiting the water-to-binder ratio to less than 0.500 was essential for achieving high strength, also due to the higher porosity at higher water-to-binder ratios [29]. Additionally, an improved strength was observed at higher dune sand contents. Dune sand was previously reported to increase the strength by improving the particle packing of the mortar mixture [30]. Limiting the VA content to a maximum of 20% was essential for early high strength. Celik et al. [8] reported a substantial loss in 1-day strength of concrete incorporating VA. Furthermore, the pozzolanic reaction affects strength at longer curing periods [31]. SP seemed to have limited effect on the 1-day strength.

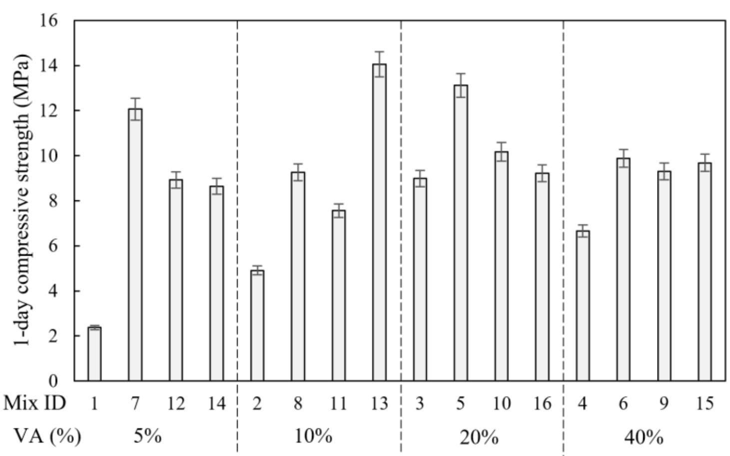

Fig. 3: 1-day compressive strength of the cement mortar mixes.

3.4. Optimization

Taguchi's method involves creating orthogonal arrays for the evaluation of a set of factors on a certain response. The evaluation is performed using a loss function, later converted into a signal-to-noise (S/N) ratio. Consequently, the S/N ratios for each cement mortar mixture at each response were calculated. In line with the experimental results, the higher S/N ratio indicates a better performance of the mortar mix in the corresponding response. Accordingly, to optimize for all responses simultaneously, the S/N ratios were implemented in TOPSIS. The method is detailed in previous work [14]. The results are shown in Figure 4. The level with the highest S/N ratio at each factor is the optimum level. Consequently, the cement mortar mix with optimum early age performance would have a binder content of 400 kg/m^3, a water-to-binder ratio of 0.500, a dune sand substitution rate of crushed sand of 20%, a VA substitution rate of cement of 20%, and an SP dosage of 0.75%.

The optimum mortar mix was cast and evaluated for validation. The same performance criteria, i.e., flow, final setting time, and 1-day compressive strength, were tested. While the optimum mix might not have the best value in every performance criterion, it will possess the best overall performance in all responses based on weights assigned in TOPSIS. The experimental results are listed in Table 2. The optimum mortar mix achieved a flow of 115 mm, a final setting time of 350 min, and a 1-day compressive strength of 18.1 MPa. The optimum mix achieved a 1-day strength substantially higher than that of other mixes while also reaching an adequate flow and final setting time. Additionally, the regression models developed using Taguchi's optimization were used to obtain theoretical results (Table 2). When compared with those obtained experimentally, it was evident that the regression models had high accuracy.

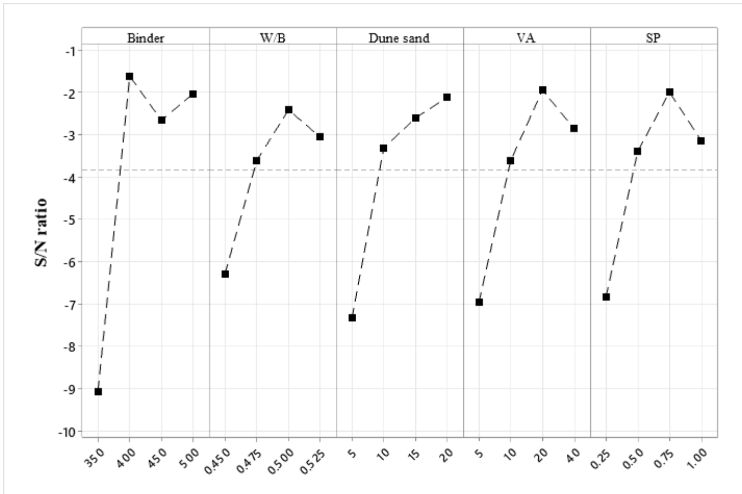

Fig. 4: Optimized mix for all responses.

Table 2: Comparison of experimental and theoretical results of the optimum mix

	Flow (mm)	Final setting time (min)	1-day compressive strength (MPa)
Experimental	115	350	18.1
Theoretical	106	338	16.0
Error (%)	8.49	3.55	13.12

4. Conclusion

The main aim of this study was to optimize the mix design parameters of cement mortar for superior early-age performance. Taguchi's optimization method was used to develop orthogonal arrays, and 16 mortar mixes were cast and tested for flow, final setting time, and 1-day compressive strength. TOPSIS was also integrated to optimize for the three responses simultaneously. The following are the key findings of the study:

- Attaining a flowability higher than 200 mm was associated with mortar mixes having a binder content of 450-500 kg/m^3, a water-to-binder ratio of 0.475-0.525, dune sand substitution rate of natural sand of 5-15%, VA substitution rate of OPC of 5-20%, and an SP dosage of 0.50-1.00% by binder mass.
- A final setting time of less than 400 min was achievable at all conditions (i.e., all levels of all factors) except for the SP dosage; the SP dosage was to be limited to 0.75% of total binder mass.
- An adequate 1-day compressive strength of at least 10 MPa was achieved by setting the mix design parameters as follows: a binder content of 400-500 kg/m^3, a water-to-binder ratio of 0.450-0.500, dune sand substitution rate of crushed sand of 10-20%, VA substitution rate of OPC of 5-20%, and an SP dosage of 0.25-1.00%.
- Taguchi's method and TOPSIS were used to obtain the optimum cement mortar mix that would achieve the best performance in flow, final setting time, and 1-day compressive strength. The mix consisted of a binder content of 400 kg/m^3, a water-to-binder ratio of 0.500, a dune sand substitution rate of crushed sand of 20%, VA substitution rate of cement of 20%, and an SP dosage of 0.75% by binder mass.
- The optimum mix was validated experimentally and achieved a flow of 115 mm, a final setting time of 350 min, and a 1-day compressive strength of 18.1 MPa. The regression models from Taguchi's method were used to theoretically approximate the performance of the cement mortar mix and showed great accuracy.

While this study demonstrates the potential of volcanic ash in early-age cementitious composites, certain limitations should be acknowledged. The experimental results may be influenced by variability in raw material properties, particularly the chemical composition and fineness of volcanic ash. Measures were taken to minimize this error, such as sourcing the materials from consistent batches. Also, it is essential to note that Taguchi optimization is constrained by the predefined factor levels, where potentially better combinations outside these ranges cannot be detected. However, factors and levels were rigorously selected to mitigate this based on preliminary trials and material characteristics. Future research could explore machine learning-based models to uncover potential optima beyond the tested range.

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