

Optimization of CWP-Slag Blended Geopolymer Concrete using Taguchi Method

Ponalagappan Chokkalingam¹, Abdulkader Elmir¹, Hilal El-Hassan¹, Amr El-Dieb¹

¹United Arab Emirates University
Al Ain, United Arab Emirates

201770337@uaeu.ac.ae; abdelkader.elmir@hotmail.com; helhassan@uaeu.ac.ae; amr.eldieb@uaeu.ac.ae

Abstract - This paper aims to optimize the mixture proportions of geopolymer concrete prepared using a binary binder system composed of ceramic waste powder (CWP) and ground granulated blast furnace slag (or simply slag) for superior mechanical performance. The corresponding mixtures were proportioned, analyzed, and optimized by adopting the Taguchi approach. The binder content, CWP replacement rate by slag, alkali-activator solution-to-binder (AAS/B) ratio, sodium silicate-to-sodium hydroxide (SS/SH) ratio, and sodium hydroxide solution molarity were assigned as factors in the design phase. Each factor was characterized by four different levels, resulting in the establishment of an L_{16} orthogonal array. The target design property was the 28-day cylinder compressive strength. The analysis of variance showed that AAS/B ratio, CWP replacement rate by slag, and SS/SH ratio were key factors affecting the strength in geopolymer concrete, while SH molarity and binder content showed the least contributions. The blended geopolymer made with 40% CWP and 60% slag yielded the optimal compressive strength response with a binder content, AAS/B ratio, SS/SH ratio, and SH solution molarity of 450 kg/m³, 0.5, 1.5, and 10 M, respectively.

Keywords: Ceramic waste powder, slag, geopolymer, Taguchi method.

1. Introduction

The incorporation of powder wastes characterized with silicate and/or aluminate compounds have shown promising results during the manufacture of mortar/concrete. Fly ash, metakaolin, ground granulated blast furnace slag (or simply slag), ladle slag, expanded perlite, and ceramic waste powder (CWP) are considered as by-product wastes that could partially or completely replace Portland cement to achieve concrete with adequate strength properties and durability [1-9]. Such practice combines the advantages of enhancing the performance of concrete while mitigating its ecological footprint [10]. For instance, the global production of ceramics generates around 22 billion tons of CWP, leading to serious environmental and disposal concerns, as CWP contains toxic metals such as barium, cadmium, and copper that could degrade soil fertility and contaminate groundwater [11].

The use of CWP as a partial substitute of cement and/or natural fine aggregates is well documented in the literature [12-14]. AlArab et al. [15] reported that the combined use of CWP and slag as binding materials led to remarkably enhanced concrete properties. The ternary binder system containing 50% cement, 35% slag, and 15% CWP exhibited strength and durability characteristics comparable to the equivalent control mixture containing 100% cement. Zegardło et al. [16] demonstrated that the incorporation of recycled ceramic aggregate is suitable for the production of ultra-high strength concrete, with f'_c greater than 120 MPa. El-Dieb et al. [14] reported that the incorporation of CWP (i.e., 40% as a cement substitute) could remarkably improve the resistance against chloride ion permeability compared to control mixtures. Similarly, Mohammadhosseini et al. [17] found that the chloride penetration response was remarkably lower in mortar containing CWP compared to that of mortar made with 100% cement.

Limited studies have examined the use of CWP as a precursor binder in the production of alkali-activated concrete. Huseien et al. [18] reported that environmental-friendly alkali-activated mortars having f'_c values higher than 70 MPa can be successfully produced while blending 50% CWP and 50% slag as precursor binders. The corresponding SEM images showed a dense surface, reflecting the production of C-S-H and C-A-S-H gels [18]. Mahmoodi et al. [19] explored the development of binary geopolymer system composed of CWP and concrete waste. The mixture containing 45% slag, 35% concrete waste, and 20% CWP exhibited a significant strength response of 101 MPa after 28 days under high curing temperature of 100°C [19].

Based on the literature, the mixture proportions of CWP geopolymer concrete have not been optimized for superior mechanical properties yet. In fact, this process is quite complex, as several factors must be considered simultaneously

during the design phase, which may lead to an extensive number of experimental tests. Taguchi method proved to be efficient in achieving concrete with superior performance while limiting the number of test methods [20-22]. Hence, this approach can be considered reliable in identifying the optimum mixture proportions to optimize the performance of geopolymer concrete prepared with different CWP rates.

This study aims to optimize the mixture proportions of geopolymer concrete made with CWP for superior 28-day compressive strength. The Taguchi method for design of experiments was adopted for this optimization. The geopolymer concrete mixtures were proportioned with various combinations of binder content, GGBFS replacement rate by CWP, alkali-activator solution-to-binder (AAS/B) ratio, sodium silicate-to-sodium hydroxide (SS/SH) ratio, and sodium hydroxide solutions molarity. The findings of this study could pave the way for the utilization of CWP in geopolymer concrete while reducing the harmful environmental effects associated with its disposal.

2. Experimental program

2.1. Materials

Slag and CWP served as the precursor binding materials. The slag was sourced from Emirates Cement and used in its as-received conditions. Its particle size distribution, physical properties, and chemical composition can be found in other work [23]. Conversely, the ceramic waste was supplied by Exeed Industries. It was ground to a mean particle size of 5-10 μm and collected after oven-drying. The final product was denoted as CWP. Its SiO_2 and CaO contents were 68.6 and 1.7, respectively, while its Blaine fineness was $5550 \text{ cm}^2/\text{g}$. Crushed dolomitic limestone and dune sand served as aggregates. Their particle size distribution and properties are reported elsewhere [24].

The alkaline activator solution was composed of sodium hydroxide (SH) and sodium silicate (SS). The SH solution was prepared by dissolving 97-98% of SH flakes in water to obtain molarities of 8, 10, 12, and 14 M. The SS solution had SiO_2 , Na_2O , and H_2O compositions of 26.3, 10.3, and 63.4%, respectively. A commercially available high-range water reducer (HRWR) was employed to guarantee adequate consistency of the concrete mixtures.

2.2. Mix design

The Taguchi method was adopted to seek the optimum mixture conditions and significant factors while minimizing the number of experiments. This approach is based on an orthogonal array, which is converted into a signal-noise (S/N) ratio to compute the variation between the targeted values and the experimental ones. A Taguchi orthogonal array is composed of a defined number of factors. Herein, five factors (binder content, CWP replacement rate by slag, AAS/B ratio, SS/SH ratio, and SH molarity) and their corresponding levels investigated in this study are summarized in Table 1. A total of 16 mixtures were developed based on an L_{16} orthogonal Taguchi array and are summarized in Table 2.

The analysis of signal-to-noise (S/N) ratio is commonly carried out on three responses, namely, the nominal-the-better, the smaller-the-better, and the larger-the-better. The latter is employed in this study since the required response (i.e.: compressive strength) is to be maximized. The corresponding performance characteristic is given in Eq. (1).

$$\frac{S}{N} = -10 \log_{10} \left(\frac{1}{n} \sum_{i=0}^n \frac{1}{X_i^2} \right) \quad (1)$$

Where X_i is the measurements of the results for the evaluated responses and n is the number of repetitions.

Table 1: Defined factors and their corresponding levels.

Factors	Level-1	Level-2	Level-3	Level-4
A: Binder content (kg/m^3)	400	450	500	550
B: CWP replacement by slag (%)	20	40	60	80
C: AAS/B ratio	0.50	0.55	0.60	0.65
D: SS/SH ratio	1.0	1.5	2.0	2.5
E: SH solution (M)	8	10	12	14

Table 2: Mixture proportions of the geopolymer prepared for the L16 Taguchi array.

Mix ID	Binder content (kg/m ³)	CWP replacement by slag (%)	AAS/B	SS/SH	SH (M)
G1	400	20	0.50	1.0	8
G2	400	40	0.55	1.5	10
G3	400	60	0.60	2.0	12
G4	400	80	0.65	2.5	14
G5	450	20	0.55	2.0	14
G6	450	40	0.50	2.5	12
G7	450	60	0.65	1.0	10
G8	450	80	0.60	1.5	8
G9	500	20	0.60	2.5	10
G10	500	40	0.65	2.0	8
G11	500	60	0.50	1.5	14
G12	500	80	0.55	1.0	12
G13	550	20	0.65	1.5	12
G14	550	40	0.60	1.0	14
G15	550	60	0.55	2.5	8
G16	550	80	0.50	2.0	10

2.3. Mixing Sequences and Testing Methods

The mixing sequence consisted of homogenizing dune sand, saturated surface dry coarse aggregates, slag, and CWP in a pan for 5 min, then gradually introducing the alkaline activator solution over 3 minutes along with the HRWR. Following the end of mixing, the fresh concrete was cast into 150-mm cube and 150 x 300 mm (diameter x height) moulds for compressive strength testing. The specimens were demoulded after 24 hours and conserved in a room where the ambient temperature and relative humidity were kept within 25±2°C and 50±5%, respectively. The compressive strength of investigated mixtures was determined on cubic (f_{cu}) and cylindrical specimens (f'_c) as per BS EN-12390-3 and ASTM C39, respectively [25, 26]. Cube specimens were tested after 1, 7, and 28 days, while cylinders were only tested at 28 days.

3. Results

3.1. Compressive strength

Figure 1 plots the cube compressive strength (f_{cu}) responses of investigated mixtures determined after 1, 7, and 28 days. It can be observed that mixtures G5, G2, G11, and G16, made with 20, 40, 60, and 80% slag yielded the highest 28-day compressive strength responses of 53.7, 61.5, 78.5, and 64 MPa as per each slag category, respectively. The corresponding binder content, AAS/B ratio, SH/SS ratio, and SH concentration seem to be linked to the ranges of 400-550 kg/m³, 0.50-0.55, 1.5-2.0, and 10-14 M, respectively. It is interesting to note that the strength responses of mixture G13 degraded when using high binder content of 550 kg/m³ and AAS/B ratio of 0.65, respectively, reflecting the importance of determining the optimum binder content since any further addition would not positively affect the strength. Meanwhile, mixture G11 made with low binder content and AAS/B ratio exhibited higher strength responses, signifying the importance of AAS content on the performance.

The development of the compressive strength responses over curing time can be evaluated from Fig. 1. Among all mixes, mix G9 exhibited the highest strength increase of 220% between 1 and 7 days. This can be associated to the slow activation reaction of CWP under ambient conditions. In fact, mixes with 1-day strength below 10 MPa experienced the highest increase over the first 7 days. However, such rapid strength development was noticeably less pronounced when 20% CWP was replaced by slag. In fact, mixtures G1, G5, G9, and G13, made with slag replacement rate of 20%, exhibited the highest strength development between 7 and 28 days (41 to 74%). This can be attributed to the rich silica compound in CWP, leading to production of N-A-S-H gels at later ages when cured in ambient conditions [18]. Similar findings have been reported in geopolymer mixes incorporating fly ash [9, 27].

The 28-day cylinder compressive strength (f'_c) followed a similar trend as its cube counterpart. The values ranged between 10.6 and 56.6 MPa, as illustrated in Fig. 1. Accordingly, a relationship between 28-day f_{cu} and f'_c was developed in the form of Eq. (2). Figure 2 shows a strong correlation between the two mechanical properties with a correlation coefficient R^2 of 0.96.

$$f'_c = 0.69f_{cu} \tag{2}$$

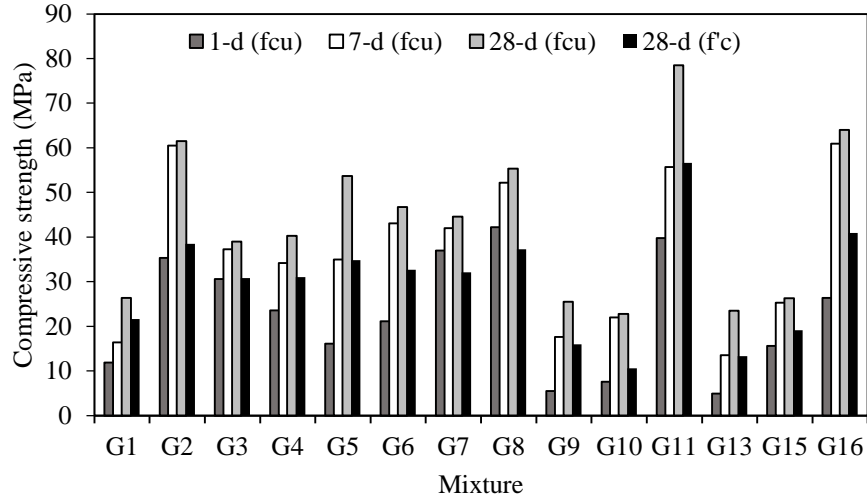


Fig. 1: compressive strength responses at the ages of 1, 7, and 28 days.

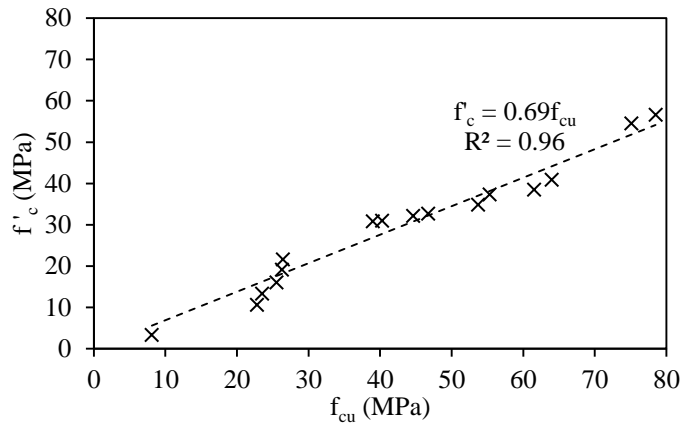


Fig. 2: Relationship between 28-day f'_c and f_{cu} .

The ANOVA analysis was carried out to identify the most significant factors on the 28-day cylinder compressive strength response. As shown in Fig. 2, AAS/B ratio, CWP replacement by slag, and SS/SH ratio were the most influencing factors with contribution responses of 35, 27, and 20%, respectively. However, the binder content and SH solution factors showed the lowest contribution response of 9% each. Hence, results reflect the complexity in identifying the single effect of each factor when evaluating the compressive strength of CWP geopolymers.

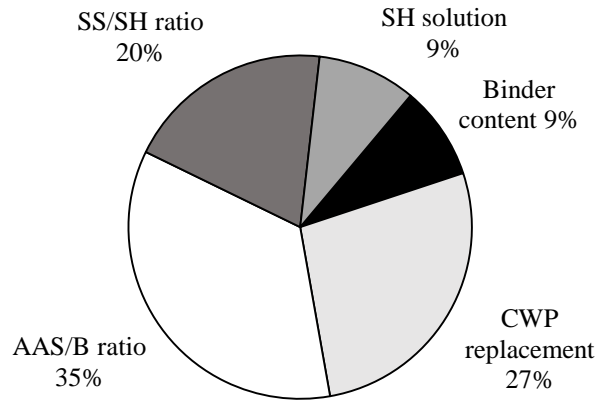


Fig. 3: ANOVA contribution factors for f'_c .

3.2. Analysis of the signal-to-noise (S/N)

The variation in the levels of factors was evaluated by employing the (S/N) ratios. The target response for geopolymer concrete mixtures designed using L_{16} orthogonal array was maximum 28-day cylinder compressive strength. Accordingly, the “larger-is-better” statistic performance [Eq. (1)] was adopted to compute the S/N ratios. As shown in Fig. 4, the maximum compressive strength was characterized by a binder content, CWP replacement rate by slag, AAS/B ratio, SS/SH ratio, and SH solution of 450 kg/m^3 , 60%, 0.5, 1.5, and 10M, respectively. To validate the optimization process, the optimum mix was cast and tested for 28-day cylinder compressive strength. Experimental results showed that the f'_c was indeed maximized with a value of 80.3 MPa.

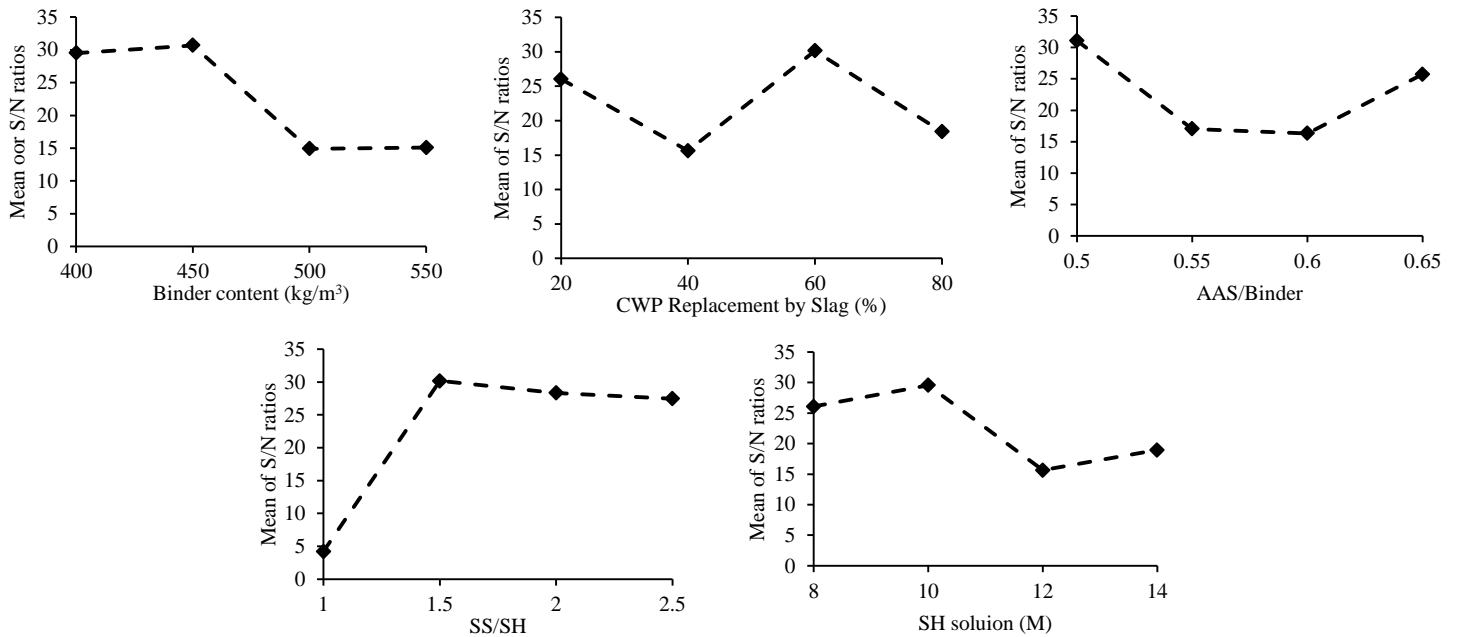


Fig. 4: Mean of S/N ratios of compressive strength.

4. Conclusions

The Taguchi method was adopted to optimize the mixture proportions of CWP-slag blended geopolymer concrete for maximum compressive strength. An L_{16} orthogonal array was established by varying the levels of five different factors, including the binder content, CWP replacement percentage by slag, alkali-activator solution-to-binder ratio (AAS/B), sodium silicate-to-sodium hydroxide ratio (SS/SH), and sodium hydroxide solution molarity. The results revealed that AAS/B ratio, CWP replacement percentage by slag, and SS/SH ratio were the most influencing factors affecting the strength in geopolymer concrete while SH solution and binder content were least influential. The binary binding system composed of 40% CWP and 60% slag exhibited the highest strength among the sixteen geopolymer concrete mixes. The optimum mix with highest 28-day cylinder compressive strength was made with a binder content, AAS/B ratio, SS/SH ratio, and SH solution molarity of 450 kg/m³, 0.5, 1.5, and 10 M, respectively.

Acknowledgements

The authors gratefully acknowledge the United Arab Emirates University (UAEU) for supporting the research reported in this paper by Grant No. 31N453. Also, the assistance of the lab staff and engineers at the civil engineering laboratory of UAEU in the preparations and testing of specimens is much appreciated.

References

1. Awoyera, P.O., Akinmusuru, J.O., and Ndambuki, J.M., 2016, "Green concrete production with ceramic wastes and laterite", *Constr Build Mater* 117, 29-36.
2. El Mir, A., Nehme, S.G., and Assaad, J.J., 2020, "Durability of self-consolidating concrete containing natural waste perlite powders", *Heliyon* 6(1), e03165.
3. Najm, O., El-Hassan, H., and El-Dieb, A., 2021, "Ladle slag characteristics and use in mortar and concrete: A comprehensive review", *J Clean Prod* 288, 125584.
4. Hwalla, J., Saba, M., and Assaad, J.J., 2020, "Suitability of metakaolin-based geopolymers for underwater applications", *Mater Struct* 53(5), 119.
5. Ismail, N. and El-Hassan, H., 2018, "Development and Characterization of Fly Ash/Slag-Blended Geopolymer Mortar and Lightweight Concrete", *J Mater Civ Eng* 30(4),
6. El-Hassan, H. and Ismail, N., 2018, "Effect of process parameters on the performance of fly ash/GGBS blended geopolymer composites", *J Sustain Cem Mater* 7(2), 122-140.
7. El-Hassan, H., Shehab, E., and Al-Sallamin, A., 2018, "Influence of Different Curing Regimes on the Performance and Microstructure of Alkali-Activated Slag Concrete", *J Mater Civ Eng* 30(9), 04018230.
8. El-Hassan, H. and Elkholy, S., 2021, "Enhancing the performance of Alkali-Activated Slag-Fly ash blended concrete through hybrid steel fiber reinforcement", *Constr Build Mater* 311, 125313.
9. El-Hassan, H., Shehab, E., and Al-Sallamin, A., 2021, "Effect of Curing Regime on the Performance and Microstructure Characteristics of Alkali-Activated Slag-Fly Ash Blended Concrete", *J Sustain Cem Mater* 10(5), 289-317.
10. Benhelal, E., Zahedi, G., Shamsaei, E., and Bahadori, A., 2013, "Global strategies and potentials to curb CO₂ emissions in cement industry", *J Clean Prod* 51, 142-161.
11. El-Dieb, A.S. and Kanaan, D.M., 2018, "Ceramic waste powder an alternative cement replacement – Characterization and evaluation", *Sustainable Materials and Technologies* 17, e00063.
12. Senthamarai, R.M., Manoharan, P.D., and Gobinath, D., 2011, "Concrete made from ceramic industry waste: Durability properties", *Constr Build Mater* 25(5), 2413-2419.
13. Kannan, D.M., Aboubakr, S.H., El-Dieb, A.S., and Reda Taha, M.M., 2017, "High performance concrete incorporating ceramic waste powder as large partial replacement of Portland cement", *Constr Build Mater* 144, 35-41.
14. El-Dieb, A., Taha, M.R., Kanaan, D., and Aly, S.T., 2018, "Ceramic waste powder: from landfill to sustainable concretes", *Proceedings of the Institution of Civil Engineers - Construction Materials* 171(3), 109-116.
15. AlArab, A., Hamad, B., and Assaad, J.J., 2022, "Strength and Durability of Concrete Containing Ceramic Waste Powder and Blast Furnace Slag", *J Mater Civ Eng* 34(1), 04021392.

16. Zegardło, B., Szeląg, M., and Ogrodnik, P., 2016, "Ultra-high strength concrete made with recycled aggregate from sanitary ceramic wastes – The method of production and the interfacial transition zone", *Constr Build Mater* 122, 736-742.
17. Mohammadhosseini, H., Lim, N.H.A.S., Tahir, M.M., Alyousef, R., Alabduljabbar, H., and Samadi, M., 2019, "Enhanced performance of green mortar comprising high volume of ceramic waste in aggressive environments", *Constr Build Mater* 212, 607-617.
18. Huseien, G.F., Sam, A.R.M., Shah, K.W., Asaad, M.A., Tahir, M.M., and Mirza, J., 2019, "Properties of ceramic tile waste based alkali-activated mortars incorporating GBFS and fly ash", *Constr Build Mater* 214, 355-368.
19. Mahmoodi, O., Siad, H., Lachemi, M., Dadsetan, S., and Sahmaran, M., 2021, "Development and characterization of binary recycled ceramic tile and brick wastes-based geopolymers at ambient and high temperatures", *Constr Build Mater* 301, 124138.
20. Najm, O., El-Hassan, H., and El-Dieb, A., 2022, "Optimization of alkali-activated ladle slag composites mix design using taguchi-based TOPSIS method", *Constr Build Mater* 327, 126946.
21. Nasir, M., Johari, M.A.M., Adesina, A., Maslehuddin, M., Yusuf, M.O., Mijarsh, M.J.A., Ibrahim, M., and Najamuddin, S.K., 2021, "Evolution of room-cured alkali-activated silicomanganese fume-based green mortar designed using Taguchi method", *Constr Build Mater* 307, 124970.
22. Mehta, A., Siddique, R., Ozbakkaloglu, T., Uddin Ahmed Shaikh, F., and Belarbi, R., 2020, "Fly ash and ground granulated blast furnace slag-based alkali-activated concrete: Mechanical, transport and microstructural properties", *Constr Build Mater* 257, 119548.
23. El-Hassan, H., Hussein, A., Medlly, J., and El-Maaddawy, T., 2021, "Performance of Steel Fiber-Reinforced Alkali-Activated Slag-Fly Ash Blended Concrete Incorporating Recycled Concrete Aggregates and Dune Sand", *Buildings* 11(8), 327.
24. El-Hassan, H., Medlly, J., and El-Maaddawy, T., 2021, "Properties of Steel Fiber-Reinforced Alkali-Activated Slag Concrete Made with Recycled Concrete Aggregates and Dune Sand", *Sustainability* 13(14), 8017.
25. British Standard, Testing hardened concrete - Compressive strength of test specimens, in *BS EN 12390-3*. British Standard: London, UK (2009).
26. ASTM, Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens, in *C39*. ASTM: West Conshohocken, PA (2015).
27. El-Hassan, H. and Elkholy, S., 2019, "Performance Evaluation and Microstructure Characterization of Steel Fiber-Reinforced Alkali-Activated Slag Concrete Incorporating Fly Ash", *J Mater Civ Eng* 31(10), 04019223.