Journal of Cleaner Production 310 (2021) 127350

Contents lists available at ScienceDirect

Journal of Cleaner Production

journal homepage: www.elsevier.com/locate/jclepro

Mechanical and energy performance of variably cured effective microorganisms cementitious composite designed via Taguchi

Nafeesa Shaheen [a], Syed Ali Rizwan [b], Rao Arsalan Khushnood [a,*], Thomas A. Bier [c]

[a] NUST Institute of Civil Engineering (NICE), National University of Sciences and Technology (NUST), Sector H-12, Islamabad, Pakistan
[b] Civil Engineering Department, NUCES (FAST), Faisal Town Lahore, Pakistan
[c] Chair Construction Materials Technology, IKGB, TUBAF, Germany

ARTICLE INFO

Handling editor: Prof. Jiri Jaromir Klemeš

Keywords:
Bio-inspired
EMC
EM1
EM ceramics Powder
MIP
Compressive strength
Thermal gravimetric analysis
Taguchi design (9)

ABSTRACT

Cement is a source of anthropogenic emissions ensuing environmental concerns and susceptible to crack under stresses. For mitigating the durability concerns, a bio-influenced effective microorganisms technology (EMs) has been employed in the cementitious composites. In the present research, the effect of EMs on the properties of self-compacting and conventional cementitious composites was explored using three types of EMs (EM1®, EMC® and EMX-Ceramics®) with three different of cements (CEMI, CEMIII and Calcium Aluminate cement). Taguchi approach of experimental design was implemented for optimization of formulations. L18 ($2^1 \times 3^7$) orthogonal array was selected based on influencing factors namely w/c ratio, cement type, EMs type and EMs percentage replacement. Specimens were casted and cured in air, moisture and desiccator. Forensic inspections were performed to monitor the growth of hydration products and precipitation of bio-calcite precipitate. Energy performance of EMs modified composites was also analyzed using ECOTECT. Experimental results were analyzed statistically using analysis of variance (ANOVA). The analysis revealed that w/c and EM % replacements as the most governing parameters in response modification of cement composites. The 4% EM replacement was determined to be the optimal percentage in refining the fresh and hardened state response of cementitious systems. Forensics endorsed the microstructural refinements as a result of bio-precipitate, contributing in the increase of mechanical properties and reduction in 27% cooling loads. Moreover, theoretical equations were proposed for prediction and optimization of mix design based on Taguchi approach using extensive regressions.

1. Introduction

In recent decades, a scientific shift has been observed toward the application of different types of admixtures in cementitious composites for the improvement of durability and aesthetics of the structures. Admixtures are added into cementitious systems for miscellaneous intended applications (Cordeiro et al., 2008). A variety of materials has been investigated as admixture including inert, reactive, synthesized carbonaceous particles in addition to chemical, mineral, fibrous and polymers to improve envisioned properties (Collepardi, 2005; De Schutter and Luo, 2004; Myrdal, 2007). The revolutionary transformation in the behavior of cementitious composites urged the scientists to explore new materials i.e. bio-influenced admixtures to transform the cementitious composites into living habitat (Sato et al., 2003) (Nathaniel et al., 2020). Effective microorganisms (EMs) and alkaline calcifying microbes have been applied to cementitious composites for improving durability and mitigating the cracks development consequent to drying shrinkage and stresses (Andrew et al., 2012; Siddique and Chahal, 2011). Primarily, EMs were investigated to Japan to improve the performance of soil for augmenting the plantation rate (Higa, 1991; Iriti et al., 2019). Later, they were employed for wastewater treatment and recycling of natural resources (Szymanski and Patterson, 2003). EMs insertion in soil eliminated the fertilization demand of crops resulting into 45% reduction in ammonia emissions. Moreover, CO_2 inhaling aptitude of EMs making them proactive aspirant for other industries (Kumar and Gopal, 2015). The sustainable attributes of EMS compelled the researchers to investigate their feasibility into cementitious systems as admixtures. EMs are consortia of beneficial probiotic microbes including lactic acid bacteria (LAB), photosynthetic bacteria, yeast and fungi (Higa, 1991; Iriti et al., 2019; SI, 2016). Initially, Sato et al. used EM1, EM3, EMX and EM

* Corresponding author.
E-mail addresses: nshaheen.phd16@nice.nust.edu.pk (N. Shaheen), syed.ali@nu.edu.pk (S.A. Rizwan), arsalan.khushnood@nice.nust.edu.pk (R.A. Khushnood), thomas.bier@ikgb.tu-freiberg.de (T.A. Bier).

https://doi.org/10.1016/j.jclepro.2021.127350
Received 12 January 2021; Received in revised form 3 April 2021; Accepted 28 April 2021
Available online 11 May 2021
0959-6526/

Table 1
Particle size of used powders.

	CEMI	CEMIII	Calcium Aluminate	EMX-ceramics
D50 (μm)	10.79	9.85	9.57	3.52

Table 2
Control factors and their different levels.

Variables	Level 1	Level 2	Level 3
W/C ratio	0.28+SP	0.4	–
Cement	CEM-I	CEM-III	Calcium Aluminate
EMs	EM1	EMC	EMX ceramic powder
EMs concentration	0	4%	11%

ceramics in replacement of 5%, 10% and 15% by weight of cement (Sato et al., 2003). EMs offered suppression of carbonation in addition to mechanical strength improvements (Yatim et al., 2011). Then, the carbonation of EMs modified concrete was tested under different environments comprising acidic and basic scenarios. Experimental outputs evidenced the carbonation resistance of EMs in all environments (Yatim et al., 2011). Andrew et al. optimized the concentration of EMs in concrete by formulating 11 trails and replacing water content by EMs from 5% to 50%. Their study concluded that a 5% replacement of EMs was optimum both mechanically and economically (Andrew et al., 2012). Whereas, another researcher reported 10% optimum replacement content of EMs complimented by 20% strength improvements due to denser microstructure (Ismail and Mohd Saman, 2014). The homogeneity and refinement of microstructure were also confirmed by nano-indentation study of calcium silicate hydrate phases of EMs modified concrete (Venkovic et al., 2014). Moreover, the pore size of EMs modified cement paste was lower than the reference cement paste (Ismail et al., 2017). Furthermore, EMs exhibited good thermal insulation characteristics when tested against thermal mass (Idris and Yusof, 2018). EMs modified self-compacting paste formulations exhibited higher strength values as compared to control due to viscosity modifications by insertion of EMs into cementitious systems (Rizwan et al., 2017).

EM1 has been reported as an effective material to modify the mechanical response of cementitious composites. However, its efficacy has never been tested on integration with different standard cement types subjected to varying curing conditions. Further, there is quite limited available research analyzing the potential of EMC and EMP types in conjunction to cementitious matrixes (Nathaniel et al., 2020). To explore above mentioned interaction multiple recipes of different w/c ratios and cement genera are designed while Taguchi approach is used for their optimization. Taguchi method, a partial factorial method of

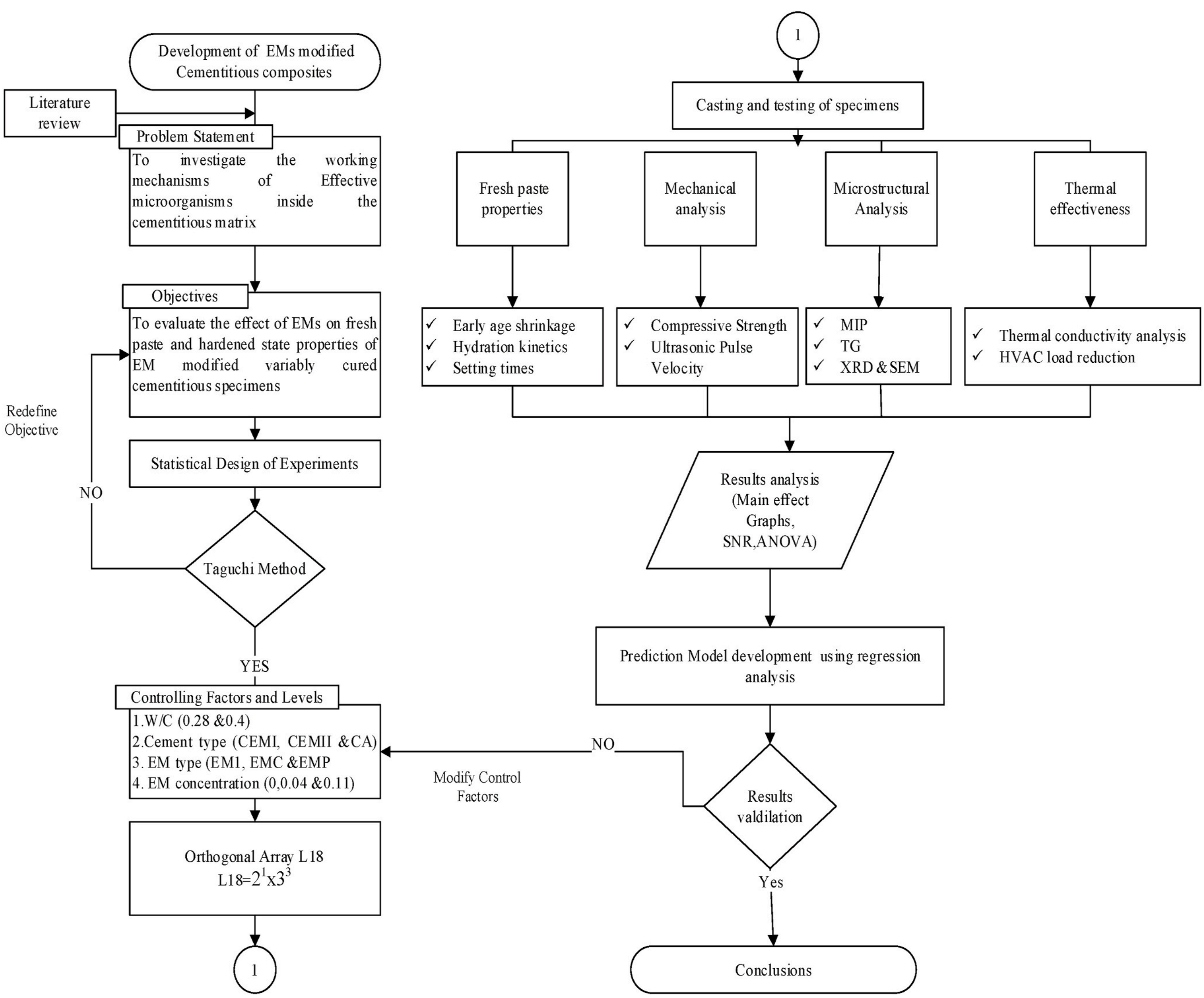


Fig. 1. Schematic diagram of investigated research designed via Taguchi Approach.

Table 3
The mix proportion of investigated formulations.

Sr.	Denotation	Cement	W/C ratio	Super plasticizer (g)	EMs	EMs percentage
1	C1-0-0.28	CEMI	0.28	2.72	–	0
2	C1-4EMC-0.28	CEMI	0.28	2.72	EMC	4
3	C1-11EMP-0.28	CEMI	0.28	2.72	EMX-ceramics powder	11
4	C3-0-0.28	CEMIII	0.28	1.2	–	0
5	C3-4EMC-0.28	CEMIII	0.28	1.2	EMC	4
6	C3-11EMP-0.28	CEMIII	0.28	1.2	EMX-ceramics powder	11
7	CA-4EM1-0.28	Calcium Aluminate	0.28	0.4	EM1	4
8	CA-11EMC-0.28	Calcium Aluminate	0.28	0.4	EMC	11
9	CA-0-0.28	Calcium Aluminate	0.28	0.4	–	0
10	C1-11EM1-0.4	CEMI	0.4	–	EM1	11
11	C1-0-0.4	CEMI	0.4	–	–	0
12	C1-4EMP-0.4	CEMI	0.4	–	EMX-ceramics powder	4
13	C3-4EM1-0.4	CEMIII	0.4	–	EM1	4
14	C3-11EMC-0.4	CEMIII	0.4	–	EMC	11
15	C3-0-0.4	CEMIII	0.4	–	–	0
16	CA-11EM1-0.4	Calcium Aluminate	0.4	–	EM1	11
17	CA-0-0.4	Calcium Aluminate	0.4	–	–	0
18	CA-4EMP-0.4	Calcium Aluminate	0.4	–	EMX-ceramics powder	4

[a]1500 g of cement was used on each batch.

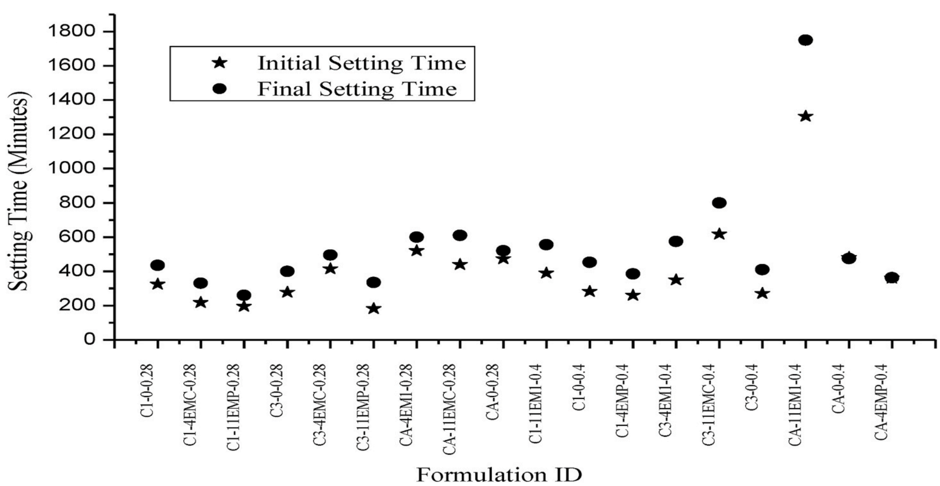


Fig. 2. Setting times of investigated formulations.

experimental design, is a combination of engineering and statistical techniques, which was devised in 1950 by Taguchi to reduce hectic, costly and laborious trials for design optimization (Hochstein et al., 1997; Panagiotopoulou et al., 2015). This method is successfully practiced in industries and accepted by the research community as well (Chen et al., 2017; Roy, 1990; Şimşek and Uygunoğlu, 2016). Hadi et al., used this model for optimization of mix proportion of geopolymer concrete whereas Jafari et al., employed this for non-destructive analysis of polymer concrete (Hadi et al., 2017; Jafari et al., 2018). Similarly, abrasion resistance of concrete, post-fire behavior and mix design of high strength concrete were evaluated using Taguchi method (Mohebi et al., 2015; Ozbay et al., 2009; Tanyıldızı, 2014). The promising results obtained from the previous studies encouraged us to use the Taguchi orthogonal arrays for optimization of mix proportions of EMs-modified cementitious systems (Kumar and Simha, 2012). For investigation of EMs influence on different types of cements, low heat of hydration cement (CEMIII) and Secar 51 self-leveling calcium aluminate cement were used in this research in addition to OPC (CEMI). Response of self-compacting and ordinary cementitious systems are analyzed to access any possible the interactions of super plasticizers with EMs. Similarly, to fully diagnosed the impact of curing on the hydration mechanisms of EMs, specimens are exposed to three different curing regimes; moist curing, desiccator curing without CO_2 and air curing having CO_2. Cementitious hydrate phases are quantified using thermal gravimetric technique (TG) While microstructure is examined via mercury intrusion porosimetry (MIP), scanning electron microscopy (SEM) and x-ray diffraction (XRD) techniques. Further, the energy conservation and carbon emissions reduction potential of EMs modified composites is calculated using ECOTECT.

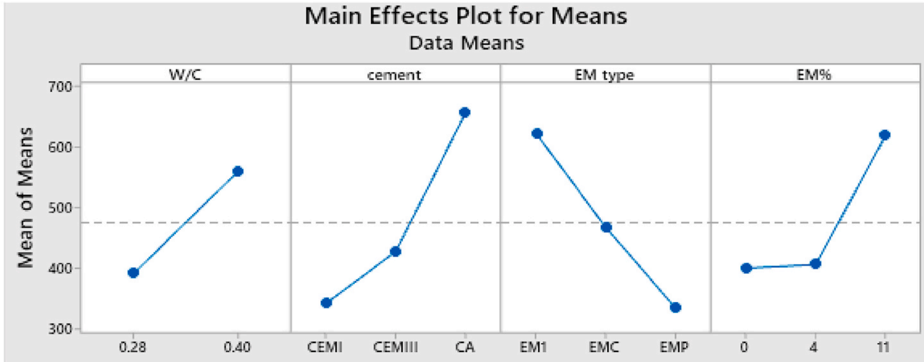
2. Materials and methods

2.1. Materials

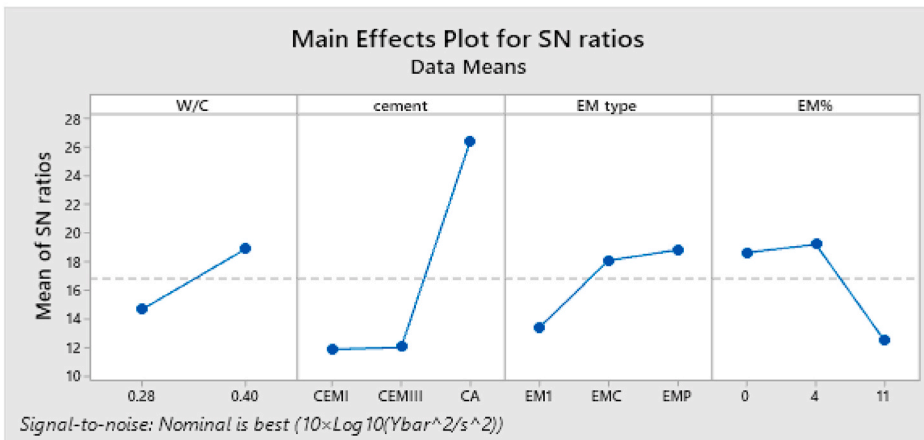
Ordinary portland CEM-I 42.5R and low heat of hydration CEM-III B 32.5N cement conforming to EN 197–1 are used (EN, 2000). To investigate the effect of EMs on aluminat phases, calcium aluminate (secar-51) cement is also utilized. The particle size analysis of all cement types are displayed in Table 1. For formulating self-compacting cementitious system, a PCE based Melflux 2651-F BASF powdered super-plasticizer was added for attaining the targetted flow of 31 ± 1 cm (Rizwan et al., 2017).

2.2. Effective microorganism

There are different types of EMs available in the market. For current experimental purposes, EM1® and EMC® liquids were selected in addition to EMX Ceramics ® powder. The mean particle size of EMX



(a) Main effect graphs of means



(b) Main effect graphs of Signal to Noise Ratio

Fig. 3. (a) Main effect graphs of means. Fig. 3 (b) Main effect graphs of Signal to Noise Ratio.

powder is mentioned in Table 1. Effective microorganism's consortia in liquid form were supplied by the Effective Microorganisms Research Organization (EMRO), Japan. The exact microbial composition of EM is kept confidential by the manufacturer (Schenck zu Schenck zu Schweinsberg-Mickan and Müller, 2009). EM1 is originally developed EM by Dr. Higa whereas EMC was developed for concrete (Andrew et al., 2012). The EMs liquids were stored in an airtight container at a cold place as the microorganisms in EM are anaerobic (Rashed and Massoud, 2015). Before using, it is ensured EMs had sweet-sour fermented smell and no foul odor.

2.3. Design of experiments (DOE)

It was intended to investigate the self-compacting cementitious systems along with the conventional cementitious systems to differentiate the effect of super plasticizer. Three different concentrations of EMs i.e. 0%, 4% and 11% by replacements of cement weight were used along with three different types of cement. A total of 54 formulations were required in the regular mix design approach. To cut down formulations number, design of experiments by Taguchi was used. He proposed different combinations of orthogonal arrays (Yang et al., 2007). Orthogonal arrays are consisted of noise and control factors (Ghosh et al., 2010). Noise factors are those factors which cannot control i-e environment (temperature, humidity) and machinery related factors (mix procedures and compaction procedures). Control factors are w/c ratios, cement type, EM type and EM%. Control factors and their variation levels are tabulated in Table 2. According to parameters and their variations levels L18 arrays was selected for mixed design of 2^1 and 3^3 parameters. L18 ($2^1 \times 3^7$) array is designed to solve a combination of 1–2 level and 3–7 level factors we (Kowalczyk, 2014). The formulations were reduced from 52 to 18. The key steps of Taguchi analysis are depicted in Fig. 1.

To calculate the target values three approaches has been proposed by the Taguchi (Ferdous et al., 2017).

- Smaller is better: choose when goal is to minimize the response. The SNR can be calculated as given in Equation (1)

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

- Larger is better: when goal is to maximize the response. The S/N ratio is calculated as given in Equation (2)

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

- Nominal is better: choose when goal is to target the response and it is required to base the S/N ratio on standard deviations and means. The S/N ratio is calculated as given in Equation (3)

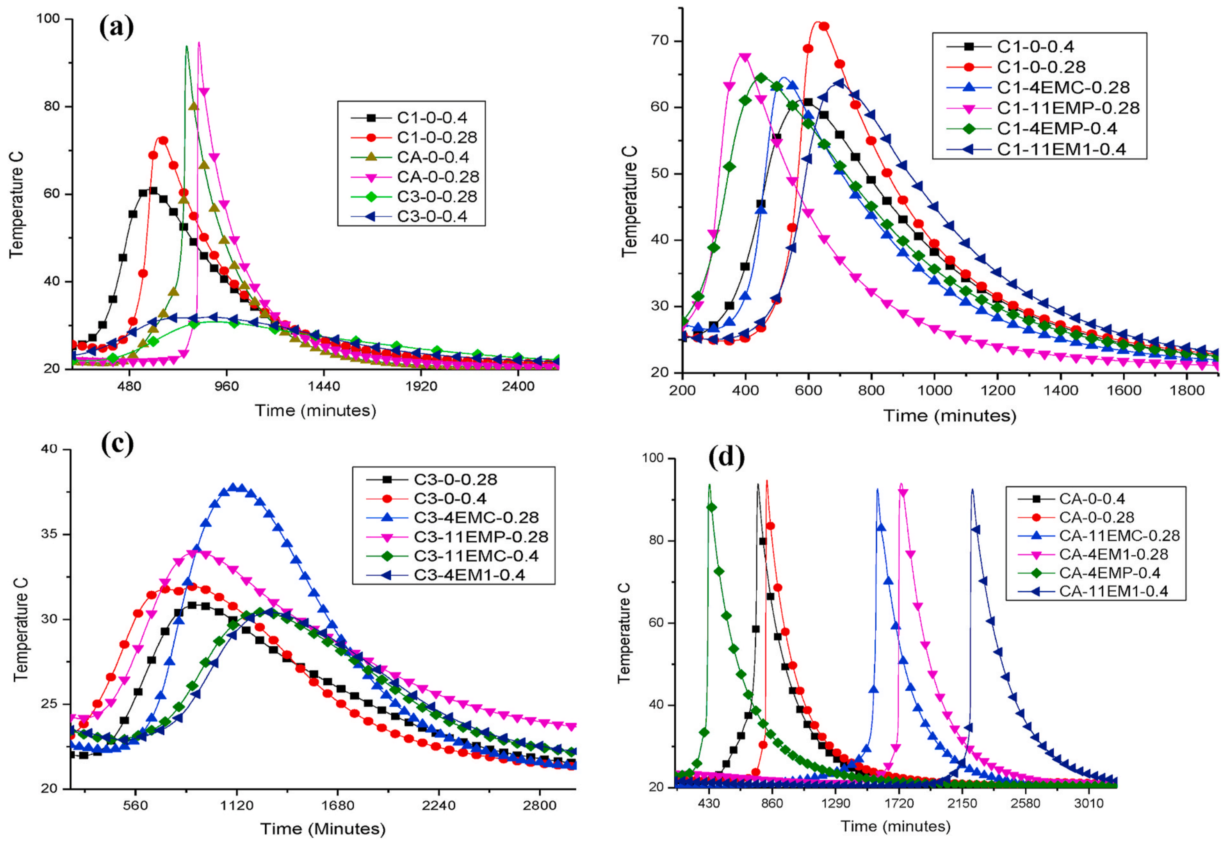


Fig. 4. Left to right (a) Hydration curves of control formulations (b) Hydration curves of EMs modified CEM-I formulations (c) Hydration curves of EMs modified CEM-III formulations (d) Hydration curves of EMs modified CA formulations.

$$S/_N = -10^{*}log\left(\frac{\overline{Y}_i^2}{\sigma_i^2}\right) \tag{3}$$

$$\overline{Y}_i = \frac{1}{n}\sum_{i=1}^{n} Y$$

$$\sigma_i^2 = \frac{1}{n-1}\sum_{i=1}^{n}\left(Y_i - \overline{Y}\right)$$

Where, Y is the experimental value of response, $\overline{Y}$ is the mean value of Y, σ is the variance of Y, n is the observation number (Manivel and Gandhinathan, 2016).

In the present study, Nominal is better loss function was used for Reducing Variability around a Target (Asiltürk and Akkuş, 2011; Varghese and Philip, 2016). Minitab software was used for calculations. Taguchi design of experiments has been recommended in the literature to optimize cost, time and resources. However, loss functions proposed by Taguchi are controversial yet because western statistician believes that sample mean and variance must be analyzed separately instead in loss functions (Maghsoodloo et al., 2004). The Taguchi method has been criticized in the literature for difficulty in accounting for interactions between parameters (Eşme, 2009). Moreover, it has limited number of arrays and can give idea of dominant parameters of the design but their impact values are relative.

Formulation's nomenclature is discussed in Table 3. CEMI, CEMII and Calcium aluminate cements are represented as C1, C3 and CA, respectively. EMX-ceramics powder is designated as EMP.

2.4. Casting and curing regimes

For casting of self-compacting paste formulations, super plasticizer quantity for each cement was determined by hit and trial method to achieve flow of 31 ± 1 cm via Hagerman cone (Rizwan and Bier, 2012). To ensure the quality standard, the mixing regime was kept constant during mixing procedure. To begin with, dry mixing of cement and super plasticizer for 30 s was done then water and EMs were added in the bowl of Hobart mixer and slow mixed (145 rpm) for 30s. After that edges of bowl were cleaned for 30s followed by fast mixing (285 rpm) for 150s. The total mixing time was maintained at 3 min (Rizwan et al., 2018). Mixing water was 28% of the weight of cement including the water available in consortia for self-compacting system. The temperature was kept at 18 ± 2 °C. For normal paste systems, mixing water was 40% by the weight of cement and same mixing regime was followed. For assessment of compression, standard prims 4x4x16 cm was casted for all mixes types and removed from molds after 24 h of casting and then placed for curing under controlled conditions. Tamping was carried out to achieve proper consolidation of the specimens. For normal paste systems compaction was done for 30 s using a vibrating table.

For thermal conductivity analysis, a square of 5 × 5 cm having thickness of 0.25 cm was casted according to ASTM C518 (EN, 2000).

Three different types of curing regimes were practiced for better insight into the working principle of EMs at different scenarios. First was moist curing at 100%RH and 25 °C, second was desiccator curing in which lime (CaO) was used to consume CO_2 and NaCl was used to maintain RH at 75% and third was air curing in presence of CO_2 at 75% RH and 25 °C.

2.5. Testing regimes

Testing regime was subdivided into four phases as illustrated in

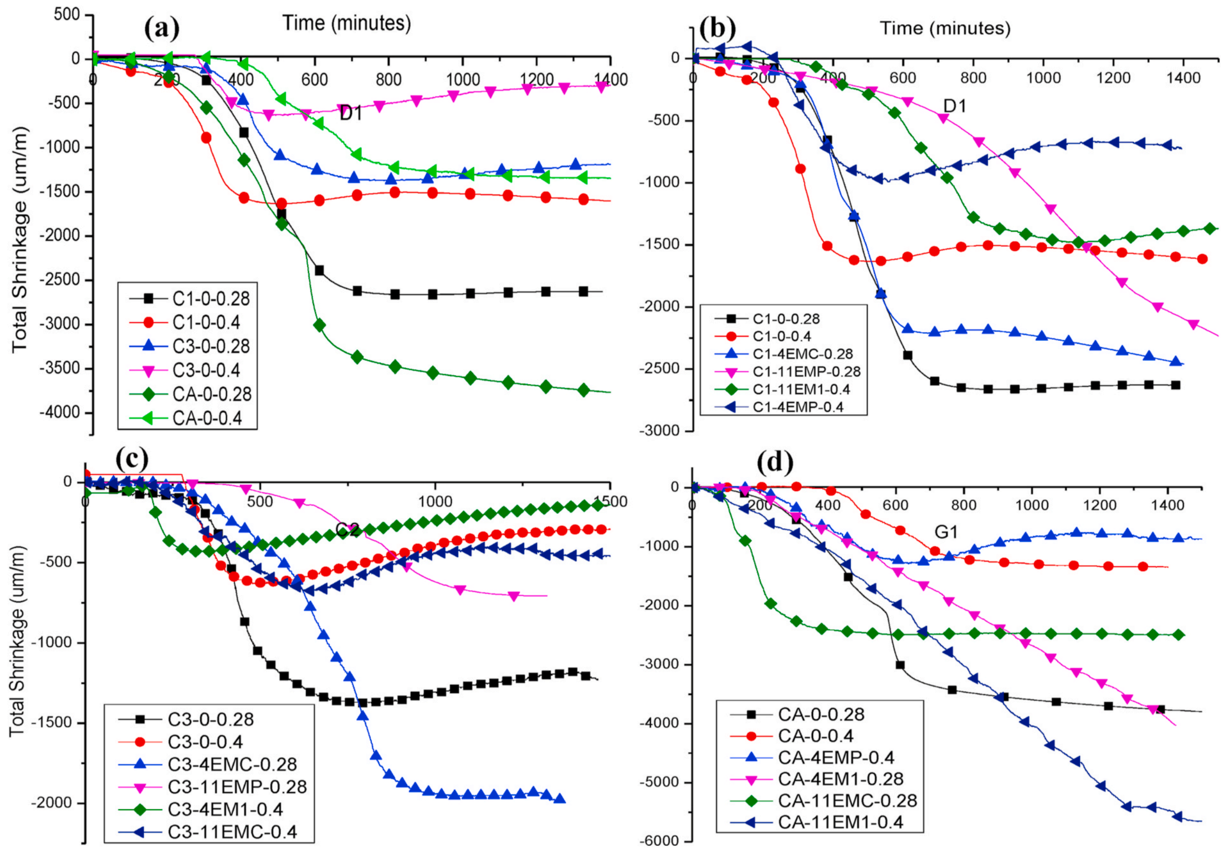


Fig. 5. Shrinkage response of all modified EMs formulations (a) Early age shrinkage response of control formulations (b) EMs modified CEM-I formulations (c) EMs modified CEM-III formulations (d) EMs modified CA formulations.

Fig. 1. In first phase, fresh state properties were determined. Setting times of cement pastes were determined using automatic Vicat apparatus corresponding to the standard DIN 196–1 (DIN, 2005). Hydration kinetics of EMs modified cementitious systems were investigated through Field calorimetry corresponding to ASTM C403 (Concrete and Aggregates, 2008). Early age shrinkage response of EMs-modified formulations was studied using German Schwindrine linear shrinkage apparatus (C157, 2006).

In second phase, Compressive strength and ultrasonic pulse velocity (UPV) were determined at the age of 2, 7 and 28 days. The compressive strength of all formulations was measured using compression testing machine conforming to ASTM test standards C-105 (C150, 2002). UPV was calculated according to BS EN 12504 standards (EN, 2004). Analysis of variance was performed and % contribution of parameters was calculated using following equation (Ghosh et al., 2010).

$$\%\ contribution\ of\ Factor\ A = \frac{SS_A}{SS_T} \tag{4}$$

Where SS_A is the sum of squared deviations for control parameter A and SS_T is total sum of squared deviation of the total response.

Regression model was developed to predict the values after experimental results' analysis for compressive strength only. To verify the accuracy of the model predicted and experimental values were compared and model was validated.

In third phase, microstructural inspection was performed using physical, micro-graphical and chemical means to find the impact of EMs on hydration of cementitious composites. For physical evaluation of pores sizes, Mercury Intrusion Porosimetry (MIP) test was conducted after 28 days of curing using ISO 9277:2010 (ISO, 2010). Scanning electron microscope (SEM) and x-ray Diffraction (XRD) and thermogravimetric (TG) was used for inspection of hydrates formation.

In last phase, thermal conductivity analysis of few specimens was performed using instrument DRX-I-PB Thermal conductivity tester (Guarded hot plate apparatus) according to standard ASTM C518 (EN, 2000). ECOTECT software was used for HVAC analysis.

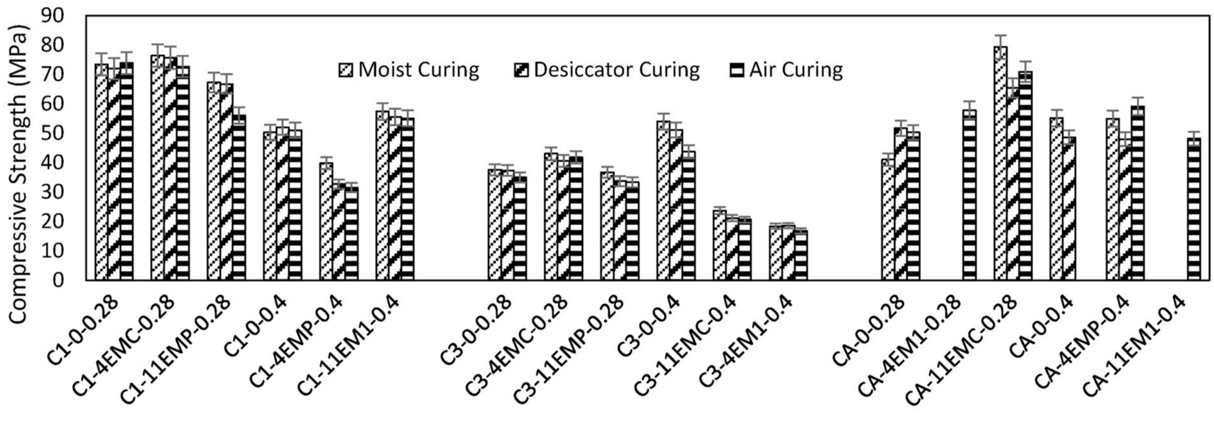
3. Results and discussions

3.1. Fresh paste properties

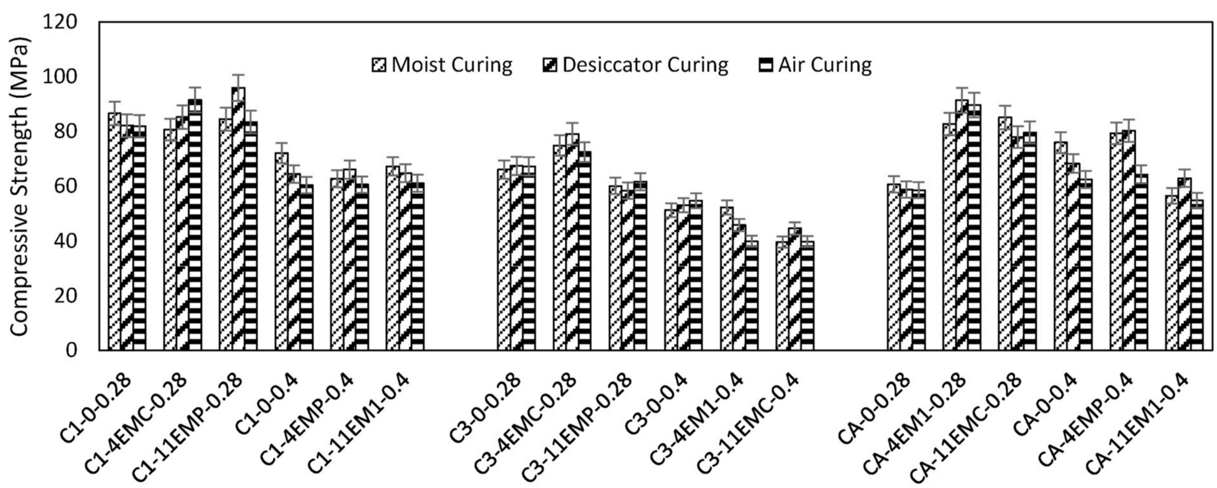
3.1.1. Setting times of formulations

Super plasticizer demand for self-compacting formulations was investigated for all three types of cement to achieve the targeted flow of 30 ± 1 cm. The SP demand of CEMI was higher followed by CEMIII and CA cements.

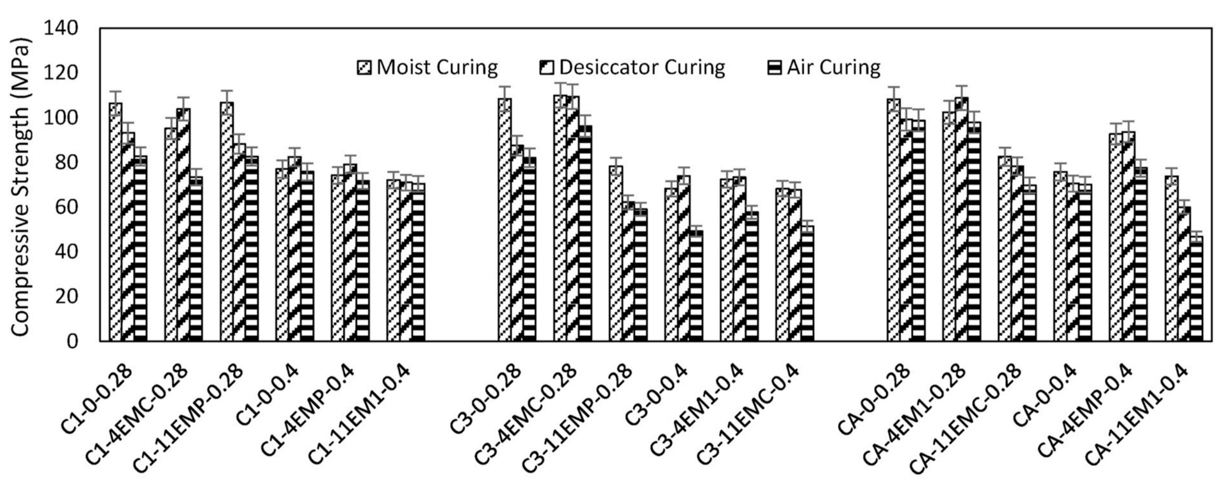
The results were quite interesting as they indicated the increase in the setting times on addition of EMs solutions. The retardation effect was because of molasses present in the EM solutions (Kavas et al., 2005). For both percentages of EM1 and EMC, setting times were generally higher for 11% replacement as compared to 4% replacement by the cement weight. This trend is much similar to the one reported by Rizwan et al. (2017) (M.Yatim et al., 2011a). However, a deviation in the trend was observed with formulation C1-4EMC-0.28 where EMC addition reduced setting time with CEMI only. Addition of EMP also reduced the setting times owing to reduced water to binder ratio because of its very fine powdered grains adding into exposed surface cover. In case of EMs solution water was balanced within water content of mix design. Trends of both initial and final setting times for all formulations are shown in Fig. 2. Addition of PCE based super-plasticizer further added into delayed setting times due to steric repulsion among the cement particle (Mohammed et al., 2016). CEMI setting times were lowest among all; followed by CEMIII and CA. In case of CA, the difference between initial



(a) 2 days compressive strength of EMs modified formulations at different curing conditions

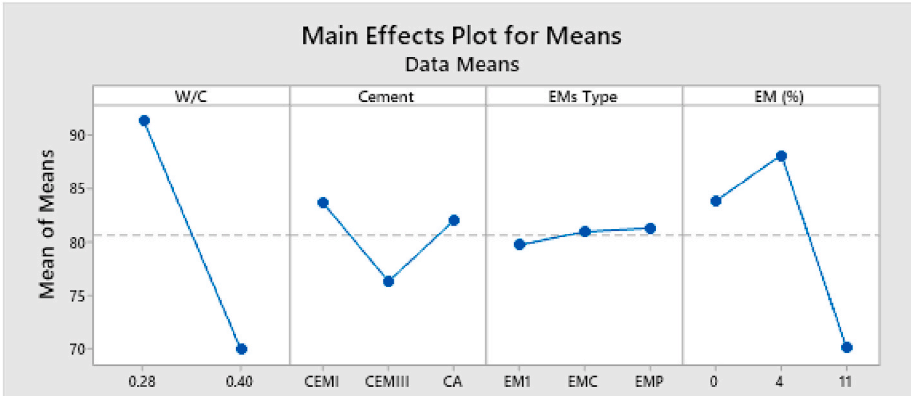


(b) 7 days compressive strength of EMs modified formulations at different curing conditions

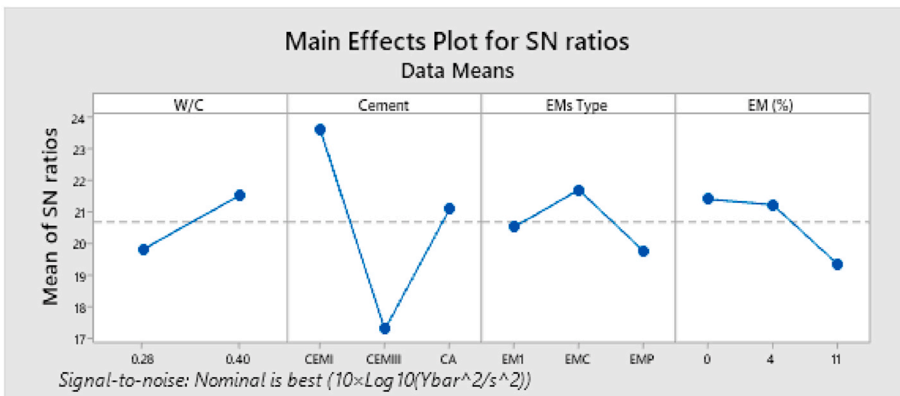


(c) 28 days compressive strength of EMs modified formulations at different curing conditions

Fig. 6. (a) 2 days compressive strength of EMs modified formulations at different curing conditions. Fig. 6 (b) 7 days compressive strength of EMs modified formulations at different curing conditions. Fig. 6 (c) 28 days compressive strength of EMs modified formulations at different curing conditions.



(a): Main effect graphs of Means with context to Compressive strength



(b): Main effect graphs of Signal to Noise Ratio with context to Compressive strength

Fig. 7. (a): Main effect graphs of Means with context to Compressive strength. Fig. 7 (b): Main effect graphs of Signal to Noise Ratio with context to Compressive strength.

and final setting time was relatively shorter as Aluminate content shows slow setting and rapid hardening owing to aluminate phases (Ukrainczyk and Rogina, 2010). Setting times of all EMs modified formulation were in permissible limits except CA-11EM1-0.4 having setting time up to 22 h.

Statistical analysis was performed on the experimental results to rule out the effect of different governing factors on the setting time of formulations. Taguchi results of means and SNR are depicted in Fig. 3. According to the statistical analysis EMC having 4% replacement is best among others as it is not adding much into the retardation. Generally, addition of EMs prolonged the setting times (Ismail et al., 2017). SNR values are around 18 to 24, these trends can be endorsed via literature (Ishrat et al., 2019).

3.1.2. Hydration kinetics

The output curves of field calorimetry of all formulations are illustrated in Fig. 4. Calorimetry curves of control formulations of all cement types are shown in Fig. 4(a). The timings and shape of temperature curves gave insight about different cement types. Lowest temperature was depicted by CEMIII (Clear, 2011) while the highest heat was measured by CA cement (Ukrainczyk and Matusinović, 2010). It is also recognized from the curves that addition of SP delayed the hydration process (Kong et al., 2016). However, heat generation was slightly higher in SP modified formulations. Acceleration-deacceleration period of CA formulations were the smallest. Fig. 4(b) illustrated the temperature curves of the modified formulation of CEMI with EMs. Addition of EMP with both w/c ratios accelerated the temperature while EM1 delayed the hydration process. Fig. 4(c) showed the temperature curves of the modified EMs formulation with CEM-III. EMC with 28% w/c ratio raised the overall temperature of the system. Fig. 4(d) depicted the EMs modified formulation of CA. Where, EMP accelerated the hydration process while other EMs solution delayed the hydration phase. Both liquid and powdered EMs seemed compatible with CEMI and CEMIII on the other hand liquid EMs adversely affected setting times of CA cements. Inclusively, EMs solution delayed the hydration process while the EMP accelerated the hydration rate.

3.1.3. Early age shrinkage

Addition of EMs and cement types play a significant role in the early age shrinkage as shown in Fig. 5. Fig. 5(a) displayed comparative response of all cement types with SP and without SP. Lowest shrinkage rate was observed in CEMIII as it is low heat of hydration cement. While highest rate was observed in case of CA cement. Moreover, addition of SP accelerated the shrinkage response of all cements. Fig. 5(b) depicted the shrinkage curves of CEMI with its EMs modified formulations. It was evident from the curves that addition of EMs reduced the shrinkage response of all SP and conventional formulations. Similar trend was recognized from the literature (Abd Rahman and Sam). EMP gave best shrinkage response with CEMI. Fig. 5(c) showed the trends of shrinkage in case of CEMIII. EM1 and EMP modified formulations showed the lesser shrinkage rate while EMC modified formulations stimulated the early age shrinkage. Fig. 5(d) showed the relative performance of EMs with CA. Contrary to the other two cements, EM1 accelerated the shrinkage rate while EMP and EMC lessened the rate in case of CA. This may be due to non-compatibility of EM1 with CA as this EM has delayed the setting times as well.

Overall, addition of EMs reduced the early age shrinkage of the cementitious systems. Self-compacting formulations depicted more

Table 4
Analysis of variance (ANOVA).

Parameter	Statistical parameters	Setting time	Compressive Strength			Ultra-Sonic Pulse		
			MC	DC	AC	MC	DC	AC
w/c	DF[a]	1	1	1	1	1	1	1
	SS[b]	89,465	2909.85	1527.4	2040.12	0.7743	0.3756	4.567
	MS[c]	89,465	2909.85	1527.44	2040.12	0.77432	0.3756	4.5669
	F[d]	2.33	29.54	18.55	10.12	0.42	0.28	2.21
	P[e]	0.158	0.000	0.002	0.01	0.533	0.608	0.168
	Contribution %	12	66.90	33.73	38.20	7.07	5.5	26.4
Cement	DF	2	2	2	2	2	2	2
	SS	331,994	72.54	186	334.32	6.1838	1.112	1.715
	MS	165,997	36.27	92.98	167.16	3.09191	0.556	0.8573
	F	4.32	0.37	1.13	0.83	1.67	0.42	0.41
	P	0.044	0.701	0.361	0.464	0.237	0.671	0.671
	Contribution %	47	3.93	4.11	6.04	31	16	9.9
EMs Type	DF	2	2	2	2	2	2	2
	SS	169,576	92.78	105.9	45.56	0.0479	1.4668	3.043
	MS	84,788	46.39	52.97	22.78	0.02395	0.7334	1.5217
	F	2.21	0.47	0.64	0.11	0.01	0.55	0.74
	P	0.161	0.638	0.546	0.894	0.987	0.594	0.503
	Contribution %	25	1.71	2.34	0.17	4.01	21	17.7
EM (%)	DF	2	2	2	2	2	2	2
	SS	114,714	421.19	1885.7	1280.47	8.3735	3.8601	7.912
	MS	57,357	210.6	942.84	640.23	4.18673	1.9301	3.956
	F	1.49	2.14	11.45	3.18	2.26	1.44	1.91
	P	0.271	0.169	2	0.086	0.155	0.281	0.198
	Contribution %	16.25	19.07	41.64	21.40	55	56	45

[a] DF Degree of freedom.
[b] SS sum of squares.
[c] MS mean square.
[d] F variance in means (Fisher value).
[e] P probability denoted significance of factor.

shrinkage than conventional ones. Highest shrinkage was observed in case of CA cement.

3.2. Mechanical analysis

3.2.1. Compressive strength

Compressive results of all modified formulations are displayed in Fig. 6. Fig. 6 (a) represents the 2 days compressive strength results of all modified formulations at three different curing conditions namely Moist curing (MC), Desiccator Curing (DC) and Air Curing (AC). CEMI modified formulations attained maximum strength at 2 days in comparison to other two cements. Moist curing (MC) represented the maximum strength gain and air curing (AC) the least and similar trend was reported by literature (Abd Rahman and Sam). In case of CA modified EM1 formulations, only air curing results were obtained as setting time was delayed for the other two formulations (see Fig. 2). Addition of EMs added into the compressive resistance of analyzed formulations at 2 & 7 days depicted in Fig. 6 (a&b). Addition of EMP improved the maximum strength in CEMI and CA cements while in case of CEMIII, EMC gave highest strength. As curing conditions are concerned, moist curing gave maximum strength while the least is attained with Air Curing. 28-days compressive strength values are summarized in Fig. 6 (c). CEMI is compatible with all EMs as no adverse action was noticed in compressive strengths of self-compacting and conventional cementitious formulations. In case of CEMIII, EMC and EM1 were compatible but EMP badly influenced the strength of the modified formulations. In case of CA, EMP and EM1 both gave good resulted in added compressive strength.

Overall, self-compacting paste formulations gave higher strength values than normal paste systems owing to deflocculating of cement particles. CEMI having 4%EMC showed 10% increment at DC condition and result are in lined with the published literature (Sam et al., 2019). CEMIII with 4% EMC gave highest compressive strength having 2%,20% and 17% increment at MC, DC and AC, respectively. CA formulations achieved a maximum of 22%, 32% and 10% strength improvement at MC, DC and AC having 4% EMP. EM1 exhibited lower strength gain rate in cementitious paste systems may be attributed to lower dosage of Lactobacillus as this strain absorbed the air contributing to development denser microstructure (Nathaniel et al., 2020).

Statistical analysis of experimental results revealed the impact of all contributing parameters on the compressive strength. Nominal is best loss function was best suited approach in the literature for cementitious systems (Kumar and Simha, 2012). Main effect graph of mean responses and SNR for all curing conditions are represented in Fig. 7. Variance of experimental compressive strength values at all curing conditions are mentioned in Table 4. W/C and EM % are however the main influencing parameters for the compressing strength as represented in Table 4. Cement types and EMs type are the least governing parameters. Variance analysis gave indication of all parameters influence on strength development (Tanyildizi and Şahin, 2015) Furthermore, response surface graphs of three curing conditions are shown in Fig. 8. These graphs are based on fraction of w/c (%) and EM (%).

The response may assist in designing for desired nominal strength of cementitious systems. Since, w/c ratios and EM% replacements are the dominating factors, therefore, Models predicting equations given as 5–7 are based on these two parameters.

$$MC = 167.8 - 211.9\ W/C - 0.946\ \%\ EM \quad (5)$$

$$DC = 143.6 - 153.5\ W/C - 1.669\%EM \quad (6)$$

$$AC = 140.3 - 177.4\ W/C - 1.632\%EM \quad (7)$$

Experimental and predicted compressive strength values based on the statistical model are compared in Fig. 9. Predicted values for the MC and DC curing are well in comparison to performed experiments while some difference is noticed in the results of air cured specimens. Ignoring outliers, the predictive statistical model corresponds with Air curing be regarded unbiased and appropriate for future use.

3.2.2. Ultrasonic pulse velocity test

Ultrasonic pulse velocity test is a non-destructive structural health

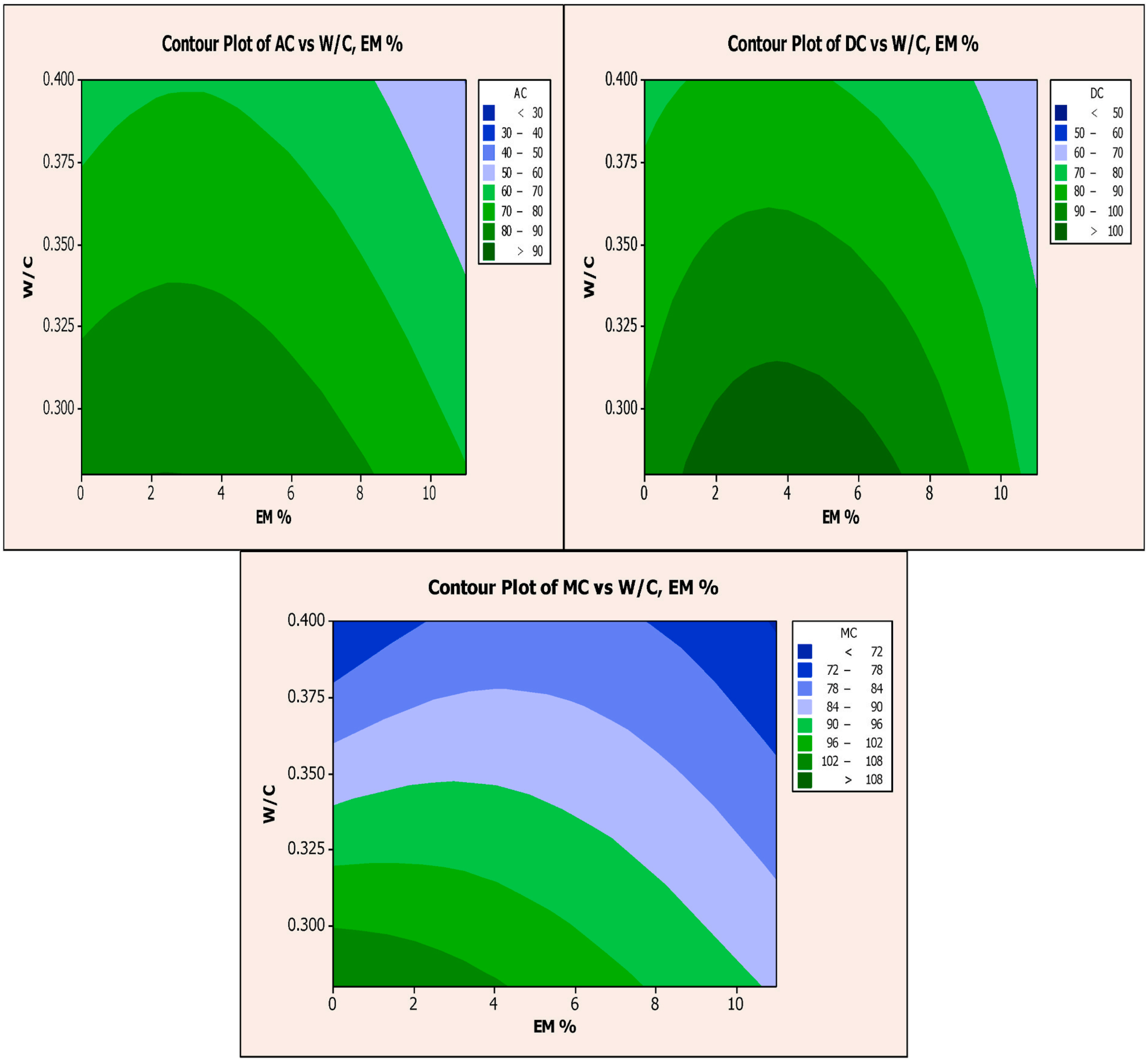


Fig. 8. Response surfaces for all curing conditions for designing and optimization of mix.

monitoring technique and used to evaluate the internal structure of cement composites. In this test, sound waves are transmitted through the sample and their travel speed is monitored. The denser the matrix is, lesser is travel time (Ozbay et al., 2009). Calculated ultrasonic pulse velocity values of EMs-modified formulations at 2, 7 and 28 days are shown in Fig. 10. At 2-days highest values of velocity were recorded by CEMIII composites while CA showed the lowest values. Self-compacting paste formulations resulted in higher values than the conventional formulations. At 7 days testing, EMs impact on cementitious matrix was visible as EMC and EMP improved the density of self-compacting paste systems.

At 28 days testing (Fig. 10(c)) EMC and EMP additions improved the matrix density of CEMI while EM1 reduced the density. In case of CEMIII, addition of EMC and EM1 reduced the densities. Similarly, EMP resulted in highest values on integration with CA cement. Both EMs types and EMs concentration affects the cementitious matrix density. As per curing conditions are concerned, Moist curing condition resulted in low values while the Air curing yielded highest values in density of resulting cement composites. Two researchers employed this test on EMs modified formulations and reported similar trends (Abd Rahman and Sam; Ozbay et al., 2009). The main effect graph for statistical analysis of pulse velocity output values is given in Fig. 11. The analysis of variance of UPV values is tabulated in Table 4. The lesser w/c (%) and 4% EMC & EMP replacement lead to denser microstructure development. The governing factors of density development are w/c ratios and EM percentage replacements.

3.3. Microstructural inspections

3.3.1. Mercury intrusion Porosimeter

Mercury intrusion porosimetry (MIP) test was conducted to find out pore diameter of the matrix. Intrusion volumes and pore diameters of some selected formulations cured under water are tabulated in Table 5 while pore size profiling is given in Table 6. In CEMI formulations, C1-0-0.4 showed largest porosity (average pore diameter 18.35 nm) while formulation C1-0-0.28 (average pore diameter 16.32 nm) gave the lower porosity owing to lesser w/c ratios. Addition of EMC & EMP reduced the porosity in all type of paste systems. Calcium Aluminate cements induced relatively high porosity 13.56% in refence formulation while 9.07% in EMs modified formulation. C3-4EMC-0.28 depicted the lowest porosity 1.08% while C1-4EMC-0.28 showed 2.26% porisity. Overall, lowest porosity is noticed in CEMIII composites. Generally, EMs shrink the pore diameters and that has been previously diagnosed by et al., (Ismail et al., 2017). This claim can further affirm from the pore

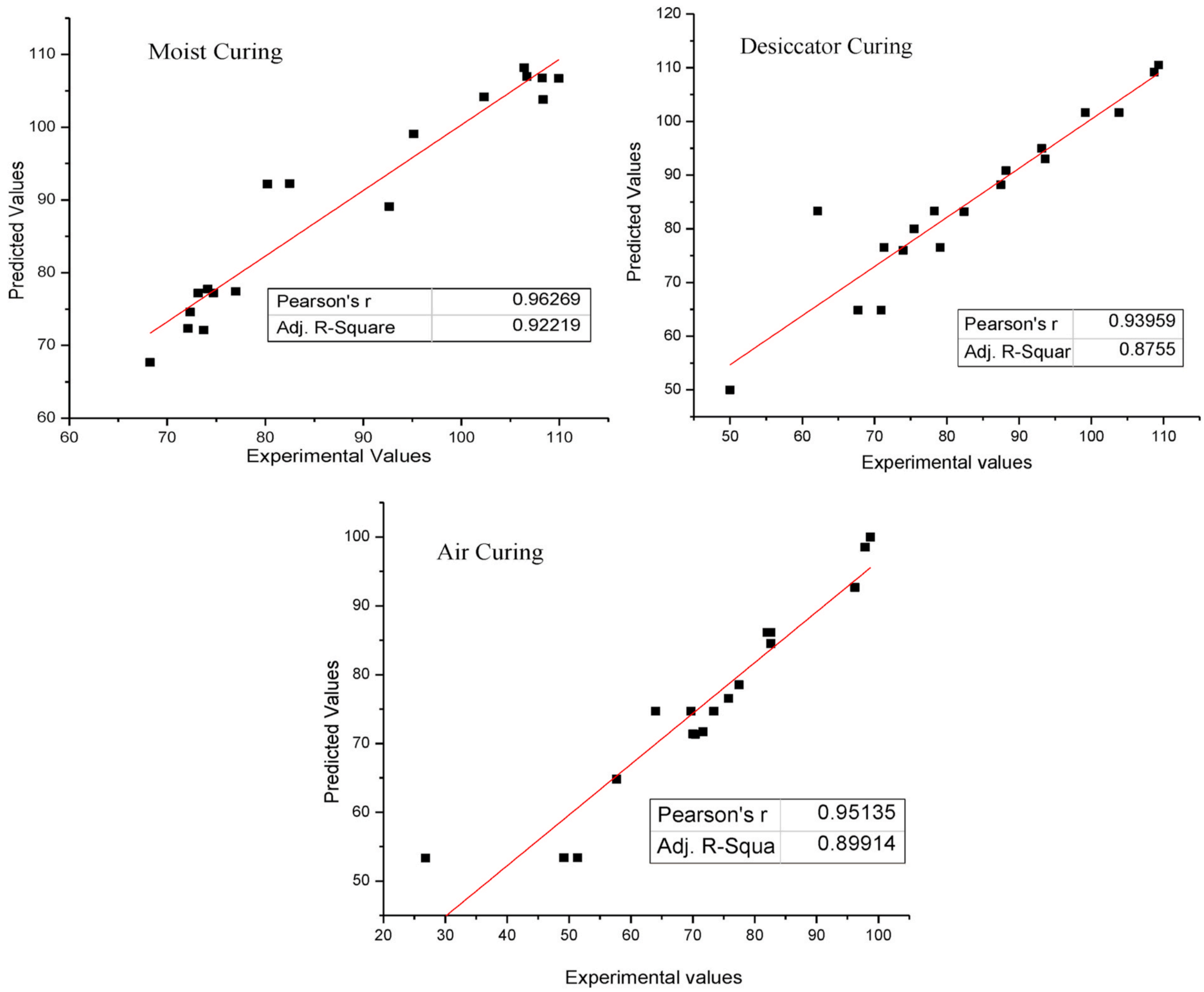


Fig. 9. Experimental and Predicted values of Compressive strength.

distribution (%) as in EMs modified formulation more pore distributed in the range lesser than 50 nm.

3.3.2. *Thermal gravimetric analysis of modified EMs cement paste*

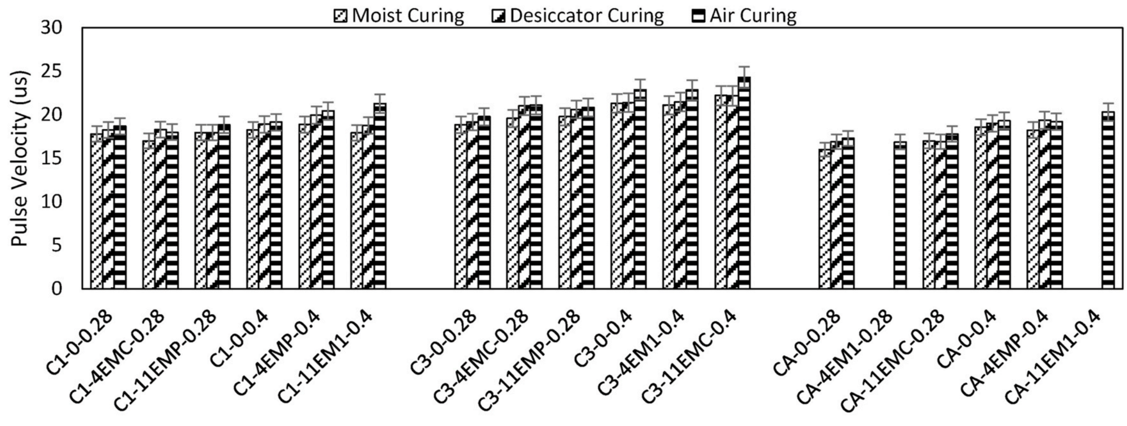
Thermo-gravimetric analysis (TG) is a mode of thermal analysis that was performed via thermo-gravimetric analyzer (Shaheen et al., 2019). Mass, time and temperature are considered basic measurements of TG. Analyzer continuously measures the mass of the substance while temperature of sample changes continuously at a constant rate (Shaheen and Khushnood, 2018). The powder of investigated formulations was subjected to temperature gradient of 30–1000 °C while heat flow rate was 10 ml/min. Nitrogen source was used for heating of furnace. TG curves of few investigated formulations are given in Fig. 12. Whereas, detailed analysis is tabulated in Table 7. The impact of curing conditions is evident from Fig. 12 (a), as more weight loss was observed in MC. However, the traces of $CaCO_3$ were higher in AC specimens. Fig. 12 (b) compared the self-compacting formulation with normal paste formulation of CEMI at two different ages. More weight loss was occurred for 28 days specimens' consequent to more hydrate phases formation at 28 days as compared to 2 days. Moreover, larger quantity of $Ca(OH)_2$ was observed for 28 days' specimens whereas $CaCO_3$ quantity is analogous for both testing ages. The TG curves depicted more C–S–H phases for SP formulation whereas weight loss was larger for normal formulations. In the same graph EMs modified formulation containing 11% EMP exhibited more hydrated phases as compared to control. Similar trend was observed for CEMIII control and EMs modified formulation as shown in Fig. 12 (c). Secar 51 cement only produced AFM phases (Fig. 12 (d)), whereas more weight loss was occurred for EMs modified formulation. TG results confirmed that EMs increased the hydration products as shown in Table 7. More hydration products were formed in case of Moist curing and least in air curing. Addition of EMs enhanced the $CaCO_3$ formation.

3.3.3. *X-ray diffraction analysis*

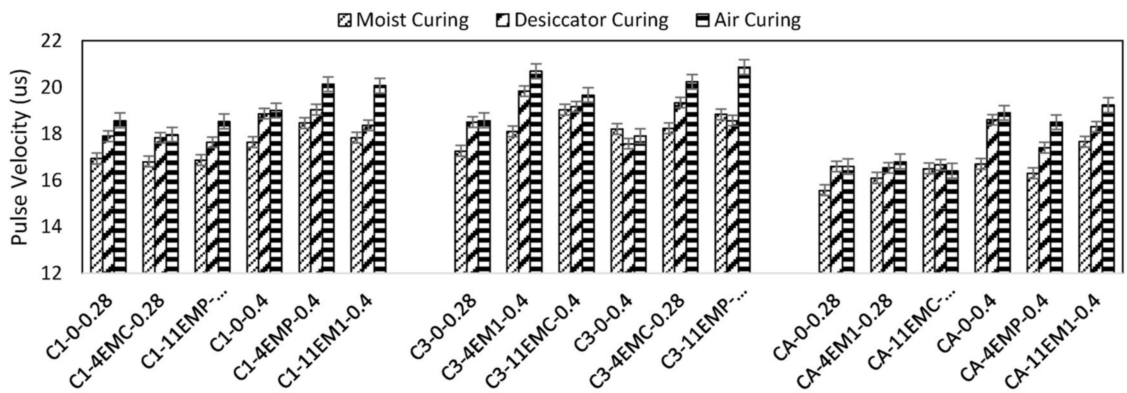
X-ray Diffraction (XRD) is used to investigate the chemical compounds and their crystallography. XRD patterns of selected formulations are shown in Fig. 13. XRD of selected formulations was performed at the age of 28 days of specimens. Generally, cementitious matrix contains calcium hydroxide, silicate hydrates, aluminate hydrates and calcium carbonate phases (Walenta and Füllmann, 2004). The formulation C1-0-0.28 was investigated for two curing conditions. In case of moist curing, calcium carbonate was abundant while in desiccator curing calcium hydroxide was abundant. Whereas addition of EMC in CEMI promoted the production of calcium carbonate (Ismail and Mohd Saman, 2014). In case of C3-4EMC-0.28-DC, silicate hydrates, calcium carbonate and calcium hydroxide were visible. Calcium aluminates phases were detected in CA-0-0.28 and CA-4EM1-0.28 formulations. Some traces of calcium carbonates were also identified in CA-4EM1-0.28 formulation.

3.3.4. *Scanning electron microscopy*

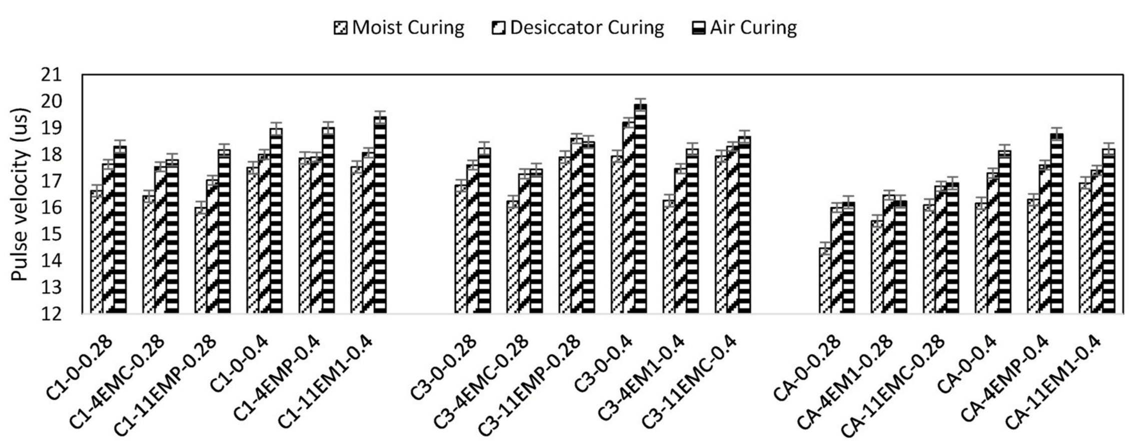
Scanning electron microscopy (SEM) is a micrographic technique for inspection of crystal shapes. Samples from selected formulations were collected at the age of 28 days and investigated via SEM. SEM



(a) 2 days Pulse velocity trends of EMs modified formulations at different curing conditions



(b) 7 days Pulse velocity trends of EMs modified formulations at different curing conditions

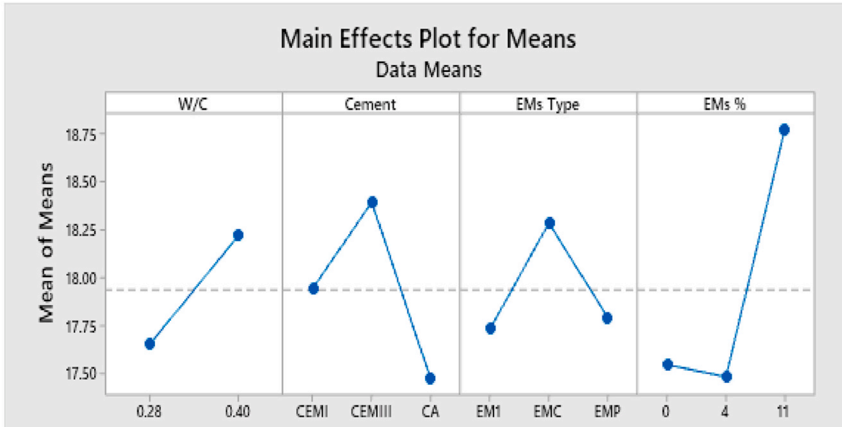


(c) 28 days Pulse velocity trends of EMs modified formulations at different curing conditions

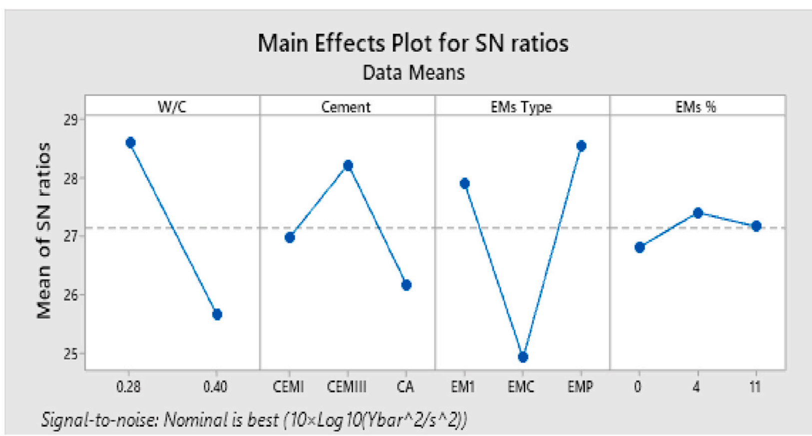
Fig. 10. (a) 2 days Pulse velocity trends of EMs modified formulations at different curing conditions. Fig. 10 (b) 7 days Pulse velocity trends of EMs modified formulations at different curing conditions. Fig. 10 (c) 28 days Pulse velocity trends of EMs modified formulations at different curing conditions.

micrographs of selected formulations are shown in Fig. 14.

In C1-0-0.28- MC (moist curing) formulations, only calcium silicate hydrates and portlandite crystals were visible. In C1-4EMC-0.28-MC, C–S–H gels and calcium carbonates crystals were seen while C–S–H gels, portlandite and calcium carbonates crystals were observed in C1-4EMC-0.28-DC. In SEM image of C3-0-0.28-MC continuous hydration gel was seen and similar type of gel is observed by Matalkah while investigating the microstructure of cementitious system (Matalkah and Soroushian, 2018; Şimşek and Uygunoğlu, 2016). C3-0-0.28-DC calcium hydroxide crystals were seen alongside CSH gels, which supports our claim of production of portlandite stimulated in the environment having no CO_2. When EMs were introduced to CEMIII, calcium carbonates crystals were seen alongside portlandite. Similar denser crystals were seen in another study (Ismail and Mohd Saman, 2014; Rizwan et al.,



(a) Optimum factor levels and graph of Means with context to Pulse Velocity Values



(b) Optimum factor levels and graph of signal to Noise ratios with context to Pulse Velocity Values

Fig. 11. (a) Optimum factor levels and graph of Means with context to Pulse Velocity Values. Fig. 11 (b) Optimum factor levels and graph of signal to Noise ratios with context to Pulse Velocity Values.

Table 5
Intrusion volume and pore diameters of selected EMs modified campsites (MC).

	Formulation ID	Total intrusion volume (mm^3/g)	Median pore diameter (volume) (nm)	Median pore diameter (Area) (nm)	Average pore diameter (nm)	Bulk density (g/mL)	Porosity (%)
1.	C1-0-0.28	80.8	31.91	6.07	16.37	1.9167	3.4%
2.	C1-4EMC-0.28	67.2	29.58	7.57	16.20	2.0877	2.26%
3.	C3-4EMC-0.28	79.2	8.46	6.49	8.31	1.9978	1.08%
4.	C1-0-0.4	94.4	48.1	9.59	18.15	2.0755	6.05%
5.	C1-4EMP-0.4	97.7	19.05	10.70	14.74	1.8723	2.16%
6.	CA-0-0.4	103.6	95.61	7.29	31.72	1.8613	13.58%
7.	CA-4EMP-0.4	90.4	58.19	6.11	19.95	1.9159	9.07%

2017). Monosulphoalumiate crystals were formed by calcium aluminate cement (Mindess, 1981). C-A-H of similar pattern are reported by Parr et al. (2005) (Parr et al., 2005). Few traces of $CaCO_3$ crystals were seen in EMs modified formulation reinforcing the claim of calcium carbonate precipitation in cements.

3.4. Thermal conductivity and CO_2 emissions

It is evident from Fig. 15 that addition of EMs transformed the thermal conductivity of the cementitious paste. The 4% and 11% replacement of EM1 resulted into 20% and 40% reduction in thermal conductivity values. Whereas, 4% and 11% replacement of EMC showed 14% increase and 4% decrease in thermal transmission, respectively. However, thermal conductivity values were reduced by increasing the EMs content and similar trend was reported by Idris et al., (Idris and Yusof, 2018). The reduction in thermal conductivity values by introducing EM1 may be attributed to air bubble entrapment during mixing and can be linked to low density values. Whereas, addition of EMC assembled more denser structure resulting into more thermal transmission.

Table 6
Pore distribution (%) in analyzed formulations.

	Formulation ID	>200 nm	100–200 nm	50–100 nm	20–50 nm	<20 nm
1.	C1-0-0.28	5.32	2.72	37.13	24.01	30.82
2.	C1-4EMC-0.28	6.85	1.93	9.82	47.47	33.93
3.	C3-4EMC-0.28	4.67	0.88	1.52	6.44	86.49
4.	C1-0-0.4	4.98	4.87	12.08	46.08	31.99
5.	C1-4EMP-0.4	4.40	2.15	6.14	33.78	53.53
6.	CA-0-0.4	24.32	23.75	24.03	13.22	14.67
7.	CA-4EMP-0.4	22.23	7.96	24.12	19.03	26.66

In order to investigate the impact of EMs on the heating ventilation and air-conditioning (HVAC) systems of buildings, a small house was selected for load calculation as shown in Fig. 16. ECOTECT software was acquired for HVAC analysis. HVAC consumption per annum of selected house is illustrated in Fig. 16. For reference formulation, commonly used material were employed as mention by Nathaniel et al. (2020) (Shaheen et al., 2016). In case of EM1, 14% and 27% reduction were observed in HVAC loading for 4% and 11% replacement. Whereas, 2.4% increase and 0.5% decrease were observed in case of 4% and 11% replacement of EMC. Hence, addition of EMs didn't escalate the cooling loads.

In order to demonstrate the energy effectiveness and sustainability of EMs, carbon emission were calculated as per IEA guidelines (Administration, 2011). According to IEA, CO_2 emissions of coal, natural gas, petroleum and hydropower electricity generation are (1000, 412.7, 966 and 18) g/kWh. In Pakistan, we have mixed electricity production according to Parr et al. (2005). So, a value of 363.5gCO^2-eq/kWh was selected based on electricity generation data provided by PEMRA (State of industry, 2020). Carbon emissions of al selected formulations are mentioned in Fig. 16. These Figs. are suggesting a significant reduction in emissions by application of EMs.

4. Conclusions

In this study, Taguchi design of experiments is employed to investigate the effect of four independent factors i.e. W/C ratio, cement type, EMs type and EM (%) replacement, on the properties of EMs modified self-compacting and conventional cementitious systems. Following conclusions are drawn from statistical evaluations of experimental results:

1. According to ANOVA analysis, W/C and EM (%) replacements are main contributing factors affecting the properties of EMs cementitious composites.
2. The setting times and hydration process of EMs formulations are delayed at higher w/c ratios and excess dosage of EM (%), however, the shrinkage is significantly reduced.
3. The compressive strength and density of system is optimized at lower water and EMs contents. Inclusively, 4% EM replacement enhances the compressive resistance and density of subsequent EMs composites by 10% and 20%. Moist curing attains maximum strength values followed by desiccator and air curing, respectively.
4. As for cement types are concerned, CEMI and CEMIII are harmonious with EMs while properties of CA formulations are adversely affected by EMs intrusions. Among EMs type, EMC & EMP performance is better relatively in terms of hardened matrix properties of cementitious composites, whereas EM1 only behaves well in shrinkage response.
5. SEM micrographs evidence more calcite precipitation and denser microstructure of the specimens in the EMs modified formulations.

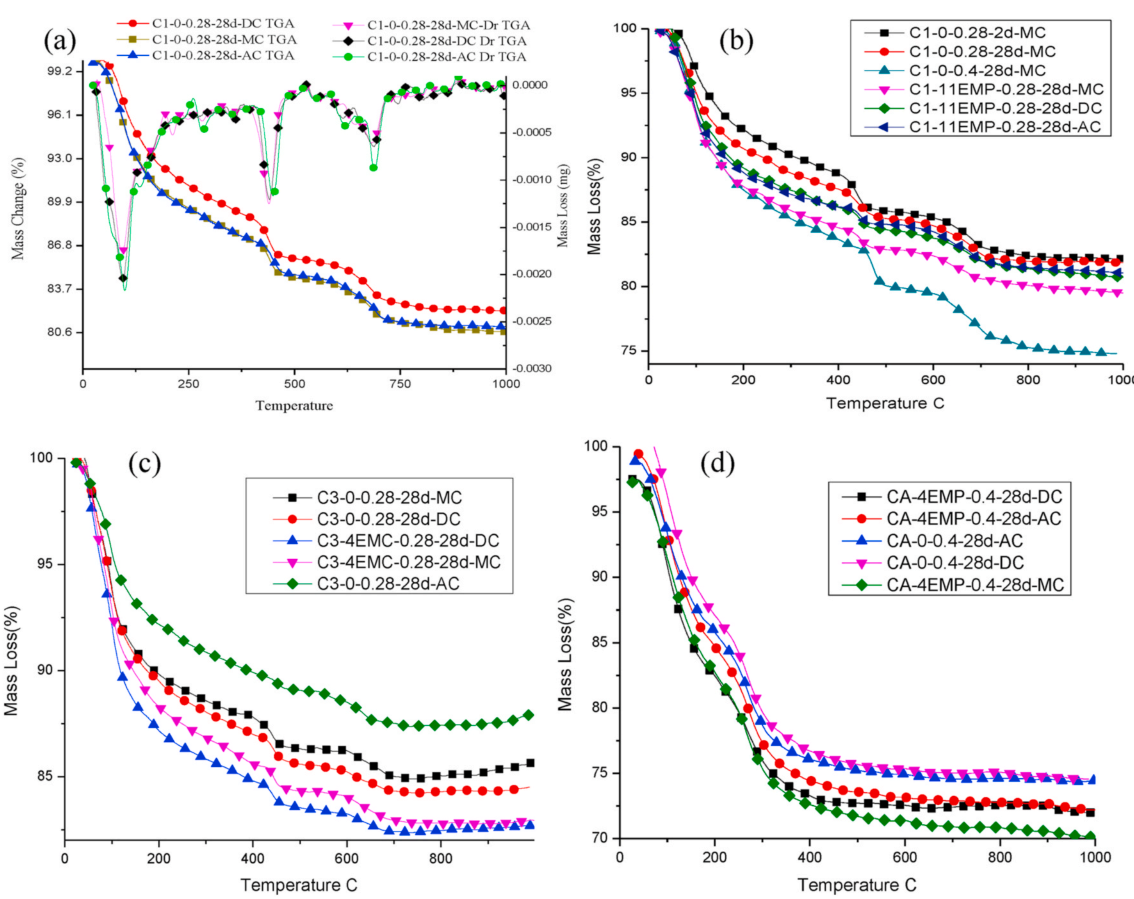


Fig. 12. TGA curves of modified EMs cementitious composites.

Table 7
Thermal Gravimetric Analysis of modified EMs cement systems.

Hydrates	Temperature °C	Moist Curing	Desiccator Curing	Air Curing	Moist Curing	Desiccator Curing	Air Curing	Moist Curing	Desiccator Curing	Air Curing	Moist Curing	Desiccator Curing	Air Curing
		C1-0-0.28-2d			**C1-0-0.28–28d**			**C1-11EMP-0.28–28d**			**C1-0-0.4–28d**		
CSH + AFm	50–200	7.39	8.39	7.4	7.87	9	9.08	10.99	10.52	9.81	12.87	9.12	
AH_3	200–350	2.57	2.17	1.99	2.76	2.54	2.56	2.58	2.49	2.17	3.01	2.13	
CH	450–580	1.14	1.23	0.85	1.32	1.18	1.151	1.04	1.12	0.88	3.12	4.32	
$CaCO_3$	580–800	3.11	3.34	3.21	3.15	2.98	3.125	2.42	2.59	2.98	5.75	4.75	
		C3-0-0.28-2d			**C3-0-0.28–28d**			**C3-4EMC-0.28–28d**			**C3-11EMP-0.28–28d**		
CSH + AFm	50–200	8.55	7.27	–	11.2	9.56	9.43	10.72	10.26			9	9.14
AH_3	200–350	1.66	1.45		1.84	1.96	1.74	2.04	2.11			2.46	2.48
CH	450–580	0.7	0.63		0.61	0.71	0.47	0.58	0.38			1.15	1.4
$CaCO_3$	580–800	1.16	1.14		0.83	1.04	1.14	1.35	1.17			2.99	3.06
		CA-11EM1-0.4-2d			**CA-0-0.4-2d**			**CA-0-0.4–28d**			**CA-11EM1-0.4–28d**		
CSH + AFm	50–200	1.07	_		10.91			14.05	12.76	14.67		14.36	14.57
AH_3	200–350	1.68			8.79			9.31	9.5	9.37		9.35	8.3
CH	450–580	0.07			0.7			0.76	0.67	0.78		0.71	0.27
$CaCO_3$	580–800	1.01			0.67			0.47	0.3	0.23		0.41	0.02

[a]CSH = Calcium-silicate-hydrate. AFm = aluminate ferrite monosulfate AH3 = Aluminum hydroxide CH = portlandite.
CaCO3=Calcium Carbonate (Collier, 2016).

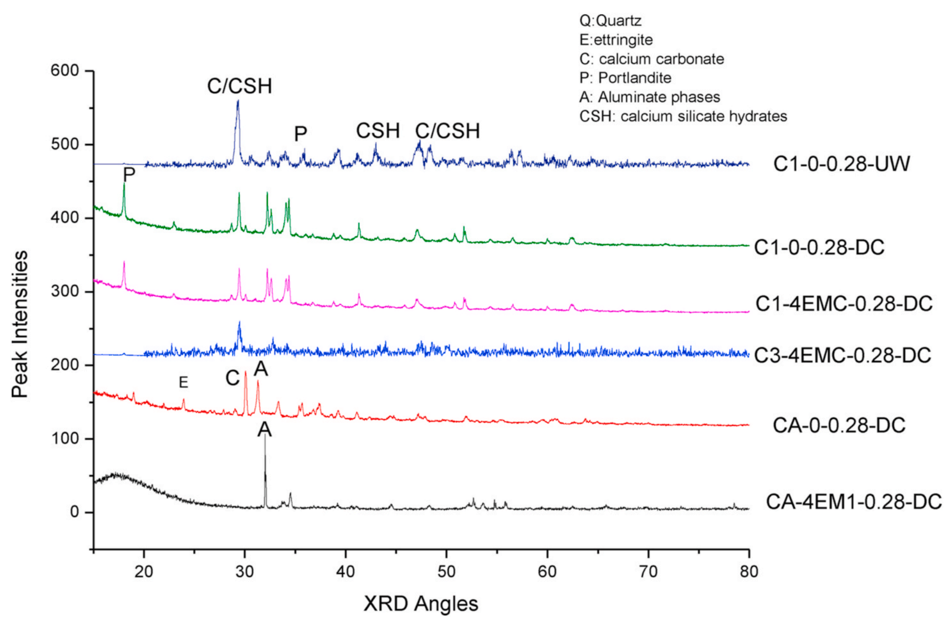


Fig. 13. XRD patterns of EMs composites showing peaks of hydrates.

Further, MIP results endorse the microstructural improvements at lower w/c and EMs replacement ratios.

6. The gravimetric thermal analysis confirms more weight loss in EM modified formulations owing to more hydrate's formation. Additionally, higher portlandite formation in desiccator curing and more carbonate hydrate in air curing specimens are observed. XRD profiles complimented the TGA observations.
7. The energy conservation aptitude of EMs improves with increase in EM% dosage having a maximum of 27% HVAC load reduction with EM1.

Certainly, the information about designed experiments presented here will be useful for the designing and optimization of the EMs modified cementitious systems for academic as well industrial applications. Moreover, Taguchi design of optimization for the experiments proved to be effective in saving time, energy and resources. However, Taguchi approach has limited experimental arrays and lacks in factor interaction and randomization. It is recommended to perform detailed investigation for optimization of different EMs% replacements levels and w/c ratio as only 3 levels are explored in the present study.

CRediT authorship contribution statement

Nafeesa Shaheen: Formal analysis, performed the experiments and analyzed the research outcomes. **Syed Ali Rizwan:** initiated the idea of research work. **Rao Arsalan Khushnood:** Writing – original draft, helped in writing manuscript and contributed to the interpretation of the results and. **Thomas A. Bier:** Supervision, supervised the research throughout.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

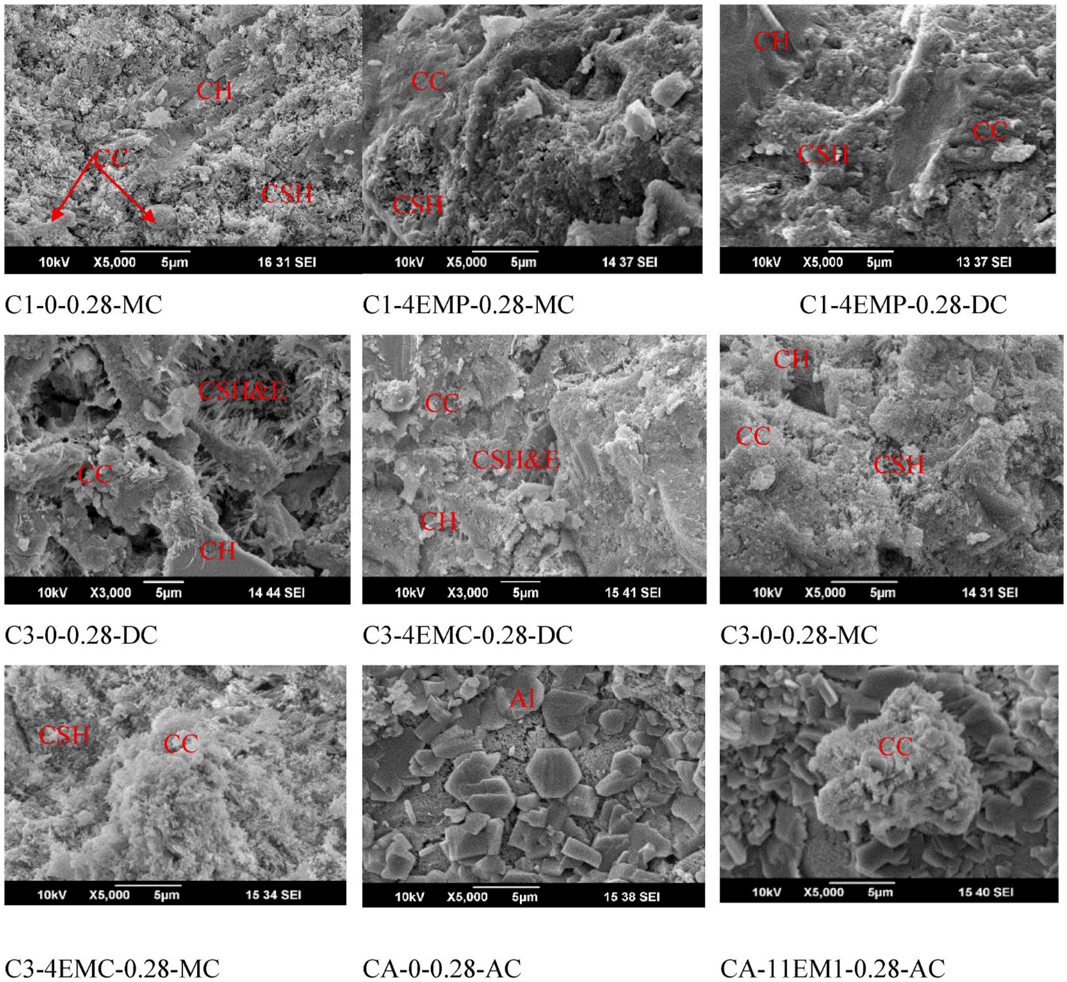


Fig. 14. SEM micrographs of EMs modified cementitious composites.

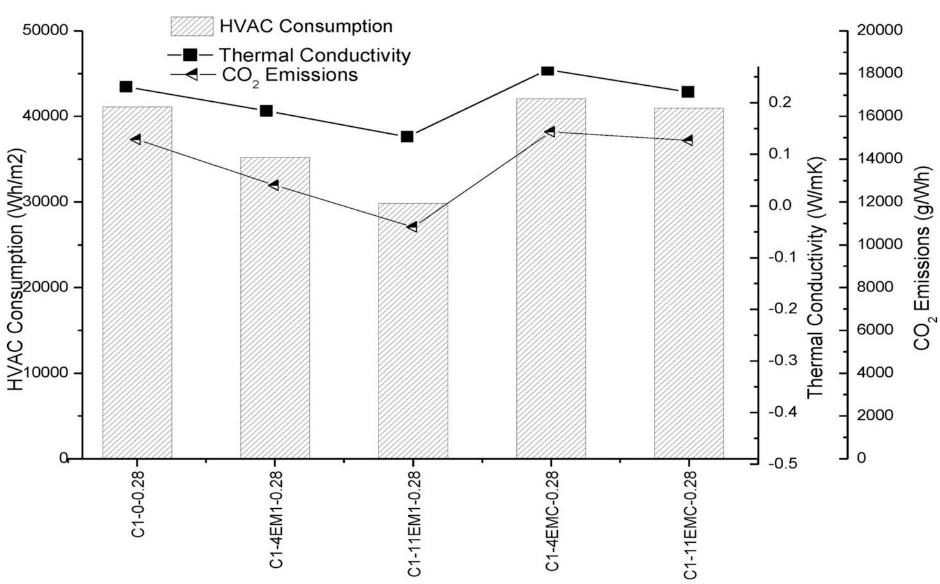


Fig. 15. Thermal conductivity and HVAC Consumption of selected formulations.

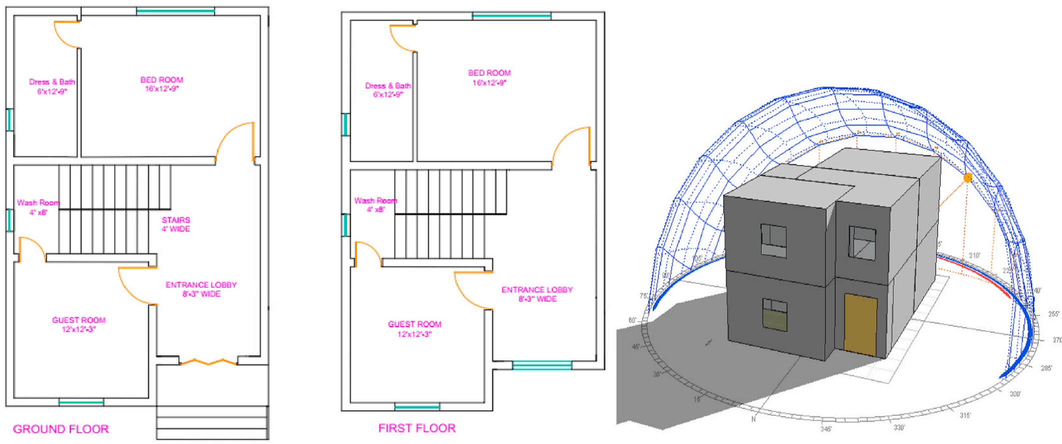


Fig. 16. Architectural plans and 3-D view of thermally analyzed house.

Acknowledgements

The research presented in this paper was sponsored by German Academic Exchange Service (Deutscher Akademischer Austauschdienst-DAAD) and was conducted at Technische Universität Bergakademie Freiberg, Germany. The Authors are also indebted to EMRO, Japan for provision of EMs.

References

Abd Rahman, N.A., Sam, A.R.M., Properties of Normal Cement Concrete Containing Effective Microorganism.
Administration, U.E.I., 2011. Annual Energy Outlook 2011: with Projections to 2035. Government Printing Office.
Andrew, T.C.S., Syahrizal, I.I., Jamaluddin, M.Y., 2012. Effective Microorganisms for Concrete (EMC) Admixture–Its Effects to the Mechanical Properties of Concrete, Awam International Conference on Civil Engineering (AICCE'12) Geohazard Information Zonation (GIZ'12).
Asiltürk, I., Akkuş, H., 2011. Determining the effect of cutting parameters on surface roughness in hard turning using the Taguchi method. Measurement 44 (9), 1697–1704.
C150, A., 2002. Standard Specification for Portland Cement. American Society for Testing and Materials:(ASTM) International West, Conshohocken, PA.
C157, A., 2006. Standard test method for length change of hardened hydraulic-cement mortar and concrete. Annu. Book ASTM Stand. 4, 96–101.
Chen, H.-J., Chang, S.-N., Tang, C.-W., 2017. Application of the Taguchi method for optimizing the process parameters of producing lightweight aggregates by incorporating tile grinding sludge with reservoir sediments. Materials 10 (11), 1294.
Clear, C., 2011. Cement type/early age properties. Concrete Today 12–14.
Collepardi, M., 2005. Admixtures: enhancing concrete performance, admixtures-enhancing concrete performance. In: Proceedings of the International Conference Held at the University of Dundee. Thomas Telford Publishing, Scotland, UK, pp. 217–230 on 6 July 2005.
Collier, N.C., 2016. Transition and decomposition temperatures of cement phases–a collection of thermal analysis data. Ceramics 60 (4).
Concrete, A.C.C.-o., Aggregates, C., 2008. Standard test method for time of setting of concrete mixtures by penetration resistance. ASTM International 4(2), 1–7. STD-8024773.
Cordeiro, G.C., Toledo Filho, R.D., Fairbairn, E.d.M.R., 2008. Use of ultra-fine sugar cane bagasse ash as mineral admixture for concrete. ACI Mater. J. 105 (5), 487.
De Schutter, G., Luo, L., 2004. Effect of corrosion inhibiting admixtures on concrete properties. Construct. Build. Mater. 18 (7), 483–489.
Din, E., 2005. Part 196-1: 2005-05. Methods of testing cement 1, 196-191.
EN, B.S., 2000. Cement-Part 1: Composition, Specifications and Conformity Criteria for Common Cements, vols. 197–1. British Standards Institution.
EN, T., 2004. 12504-4. Testing Concrete–Part 4: Determination of Ultrasonic Pulse Velocity, vol. 18. British Standards Institution.
Eşme, U., 2009. Application OF taguchi method for the optimization OF resistance spot welding process. In: Arabian Journal for Science & Engineering, vol. 34. Springer Science & Business Media BV.
Ferdous, W., Manalo, A., Aravinthan, T., 2017. Bond behaviour of composite sandwich panel and epoxy polymer matrix: Taguchi design of experiments and theoretical predictions. Construct. Build. Mater. 145, 76–87.
Ghosh, S.K., Pal, S., Roy, S.K., Pal, S.K., Basu, D., 2010. Modelling of flame temperature of solution combustion synthesis of nanocrystalline calcium hydroxyapatite material and its parametric optimization. Bull. Mater. Sci. 33 (4), 339–350.
Hadi, M.N., Farhan, N.A., Sheikh, M.N., 2017. Design of geopolymer concrete with GGBFS at ambient curing condition using Taguchi method. Construct. Build. Mater. 140, 424–431.
Higa, T., 1991. Effective microorganisms: a biotechnology for mankind. In: Proceedings of the First International Conference on Kyusei Nature Farming. US Department of Agriculture, Washington, DC, USA, pp. 8–14.
Hochstein, D.J., Ahmad, A.B., Magowan, R.E., 1997. Teaching the Taguchi Method of Experimental Design: Design and Testing of Concrete Mixes, Annual Conference, Milwaukee, Wisconsin, pp. 1–15.
Idris, I.H.M., Yusof, N.Z., 2018. Development of low thermal mass cement-sand block utilizing peat soil and effective microorganism. Case studies in construction materials 8, 8–15.
Iriti, M., Scarafoni, A., Pierce, S., Castorina, G., Vitalini, S., 2019. Soil application of effective microorganisms (EM) Maintains leaf photosynthetic efficiency, increases seed yield and quality traits of bean (Phaseolus vulgaris L.) plants grown on different substrates. Int. J. Mol. Sci. 20 (9), 2327.
Ishrat, S.I., Khan, Z.A., Siddiquee, A.N., Badruddin, I.A., Algahtani, A., Javaid, S., Gupta, R., 2019. Optimising parameters for expanded polystyrene based pod production using Taguchi method. Mathematics 7 (9), 847.
Ismail, N., Mohd Saman, H., 2014. Microstructure Examination and Strength Characteristics of Effective Microbed Cement.
Ismail, N., Saman, H.M., Jaafar, M.F.M., Abdullah, S.R., 2017. The Effect of Effective Microorganism (EM) Inclusion to the Setting Time of Microbed Cement Paste. MATEC Web of Conferences. EDP Sciences, 01022.
ISO, I., 2010. 9277: 2010 Determination of the Specific Surface Area of Solids by Gas Adsorption–BET Method. International Organization for Standardization, Geneva.
Jafari, K., Tabatabaeian, M., Joshaghani, A., Ozbakkaloglu, T., 2018. Optimizing the mixture design of polymer concrete: an experimental investigation. Construct. Build. Mater. 167, 185–196.
Kavas, T., Olgun, A., Erdogan, Y., 2005. Setting and hardening of borogypsum–Portland cement clinker–fly ash blends. Studies on effects of molasses on properties of mortar containing borogypsum. Cement Concr. Res. 35 (4), 711–718.
Kong, F.-r., Pan, L.-s., Wang, C.-m., Xu, N., 2016. Effects of polycarboxylate superplasticizers with different molecular structure on the hydration behavior of cement paste. Construct. Build. Mater. 105, 545–553.
Kowalczyk, M., 2014. Application of Taguchi and Anova methods in selection of process parameters for surface roughness in precision turning of titanium. Advances in Manufacturing Science and Technology 38 (2), 21–35. https://doi.org/10.2478/amst-2014-0008.
Kumar, B.L., Gopal, D.S., 2015. Effective role of indigenous microorganisms for sustainable environment. 3 Biotech 5 (6), 867–876.
Kumar, N., Simha, T., 2012. Experimental investigation on seismic resistance of recycled concrete in filled steel columns-taguchi's approach.
Maghsoodloo, S., Ozdemir, G., Jordan, V., Huang, C.-H., 2004. Strengths and limitations of Taguchi's contributions to quality, manufacturing, and process engineering. J. Manuf. Syst. 23 (2), 73–126.
Manivel, D., Gandhinathan, R., 2016. Optimization of surface roughness and tool wear in hard turning of austempered ductile iron (grade 3) using Taguchi method. Measurement 93, 108–116.
Matalkah, F., Soroushian, P., 2018. Synthesis and characterization of alkali aluminosilicate hydraulic cement that meets standard requirements for general use. Construct. Build. Mater. 158, 42–49.
Mindess, S., 1981. Concrete. Prentice Hall, New Jersey.
Mohammed, M.S., Mohamed, S.A., Johari, M.A.M., 2016. Influence of superplasticizer compatibility on the setting time, strength and stiffening characteristics of concrete. Advances in Applied Sciences 1 (2), 30–36.
Mohebi, R., Behfarnia, K., Shojaei, M., 2015. Abrasion resistance of alkali-activated slag concrete designed by Taguchi method. Construct. Build. Mater. 98, 792–798.
Myrdal, R., 2007. Accelerating Admixtures for Concrete. State of the Art.

Nathaniel, O., Sam, A.R.M., Lim, N.H.A.S., Adebisi, O., Abdulkareem, M., 2020. Biogenic approach for concrete durability and sustainability using effective microorganisms: a review. Construct. Build. Mater. 261, 119664.
Ozbay, E., Oztas, A., Baykasoglu, A., Ozbebek, H., 2009. Investigating mix proportions of high strength self compacting concrete by using Taguchi method. Construct. Build. Mater. 23 (2), 694–702.
Panagiotopoulou, C., Tsivilis, S., Kakali, G., 2015. Application of the Taguchi approach for the composition optimization of alkali activated fly ash binders. Construct. Build. Mater. 91, 17–22.
Parr, C., Simonin, F., Touzo, B., Wohrmeyer, C., Valdelièvre, B., Namba, A., 2005. The impact of calcium aluminate cement hydration upon the properties of refractory castables. Journal of Technical Association of Refractories Japan 25 (2), 78–88.
Rashed, E.M., Massoud, M., 2015. The effect of effective microorganisms (EM) on EBPR in modified contact stabilization system. HBRC Journal 11 (3), 384–392.
Rizwan, S.A., Bier, T.A., 2012. Blends of limestone powder and fly-ash enhance the response of self-compacting mortars. Construct. Build. Mater. 27 (1), 398–403.
Rizwan, S.A., Khan, H., Bier, T.A., Adnan, F., 2017. Use of Effective Micro-organisms (EM) technology and self-compacting concrete (SCC) technology improved the response of cementitious systems. Construct. Build. Mater. 152, 642–650.
Rizwan, S.A., Mahmood, N., Mahmood, U., Bier, T.A., 2018. Response of self-compacting paste (SCP) systems containing Acacia Modesta gum. Construct. Build. Mater. 161, 398–406.
Roy, R., 1990. A Primer on Taguchi Method. Van Noshtrand Reinhold Int. Co. Ltd., New York.
Sam, A.M., Khanom, F., Kamaruzzaman, M., Tahir, M.M., Lim, N.A.S., Awang, A., Ismail, A., 2019. Fresh and hardened properties of concrete containing effective microorganisms. In: IOP Conference Series: Earth and Environmental Science. IOP Publishing, 012050.
Sato, N., Higa, T., Shoya, M., Sugita, S., 2003. Some properties of concrete mixed with effective microorganisms and the on-site investigation of the completed structures. In: Proceedings of the 28th International conference, Our World in Concrete and Structures, pp. 28–29. August, 2003, Singapore.
Schenck zu Schweinsberg-Mickan, M., Müller, T., 2009. Impact of effective microorganisms and other biofertilizers on soil microbial characteristics, organic-matter decomposition, and plant growth. J. Plant Nutr. Soil Sci. 172 (5), 704–712.
Shaheen, N., Khushnood, R.A., 2018. Bioimmobilized limestone powder for autonomous healing of cementitious systems: a feasibility study. Advances in Materials Science and Engineering 2018.
Shaheen, N., Arif, S., Khan, A., 2016. Thermal performance of typical residence in karachi with different materials for construction. Mehran Univ. Res. J. Eng. Technol. 35 (2), 189–198.
Shaheen, N., Khushnood, R.A., Khaliq, W., Murtaza, H., Iqbal, R., Khan, M.H., 2019. Synthesis and characterization of bio-immobilized nano/micro inert and reactive additives for feasibility investigation in self-healing concrete. Construct. Build. Mater. 226, 492–506.
SI, S.N., 2016. Application of effective microorganism (EM) in food waste composting: a review. Asia Pacific Environmental and Occupational Health Journal 2 (1).
Siddique, R., Chahal, N.K., 2011. Effect of ureolytic bacteria on concrete properties. Construct. Build. Mater. 25 (10), 3791–3801.
Şimşek, B., Uygunoğlu, T., 2016. Multi-response optimization of polymer blended concrete: a TOPSIS based Taguchi application. Construct. Build. Mater. 117, 251–262.
Authority, P.E.M.R. (Ed.), 2020. State of Industry Report 2020.
Szymanski, N., Patterson, R.A., 2003. Effective microorganisms (EM) and wastewater systems. Future Directions for On-site Systems: Best Management Practice 347–355.
Tanyildizi, H., Şahin, M., 2015. Application of Taguchi method for optimization of concrete strengthened with polymer after high temperature. Construct. Build. Mater. 79, 97–103.
Tanyıldızı, H., 2014. Post-fire behavior of structural lightweight concrete designed by Taguchi method. Construct. Build. Mater. 68, 565–571.
Ukrainczyk, N., Matusinović, T., 2010. Thermal properties of hydrating calcium aluminate cement pastes. Cement Concr. Res. 40 (1), 128–136.
Ukrainczyk, N., Rogina, A., 2010. Setting time regulation of polymer modified calcium aluminate cement based materials, International conference on materials, tribology, recycling.
Varghese, B., Philip, N., 2016. A review: Taguchi experiment design for investigation of properties of concrete. Int. J. Civ. Eng. 5 (6), 11–16.
Venkovic, N., Sorelli, L., Martirena, F., 2014. Nanoindentation study of calcium silicate hydrates in concrete produced with effective microorganisms-based bioplasticizer. Cement Concr. Compos. 49, 127–139.
Walenta, G., Füllmann, T., 2004. Advances in quantitative XRD analysis for clinker, cements, and cementitious additions. Powder Diffr. 19 (ARTICLE), 40–44.
Yang, K., Teo, E.-C., Fuss, F.K., 2007. Application of Taguchi method in optimization of cervical ring cage. J. Biomech. 40 (14), 3251–3256.
Yatim, J.M., Ismail, M., Rahman, A.W.A., Yaw V., W., 2011a. Performance of concrete containing effective microorganisms (EM) under various environments. EACEF - International Conference of Civil Engineering 1, 782–786. https://doi.org/10.13140/RG.2.1.3467.3761.