EVALUATION AND OPTIMIZATION OF PVA REINFORCED CEMENTITIOUS COMPOSITE CONTAINING METAKAOLIN AND FLY ASH

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Due to high consumption of natural sources in the production of cement, scholars have been studying alternative materials that can replace cement in the concrete. Metakaolin and fly ash are used by scholars for their chemical properties and filler effect as pozzolanic material. In this study effect of PVA fiber, metakaolin and fly ash in concrete samples are investigated. The number of experiments to be carried out was reduced to 25 with the Taguchi optimization method, in this research. In addition, the recommended mixing ratios were obtained by this preliminary optimization study. Taguchi optimization were applied according to the experimental test results. F-value comparison of each ingredients were analyzed via ANOVA to list the most effective factors on the strengths based on the Taguchi optimization results. Later Response Surface Design was applied to the output parameters of the ANOVA and Taguchi methods to obtain better and detailed clarifications of relations between the input parameters and the strengths.

Keywords: *PVA fiber, fiber reinforced concrete, optimization*

1. Introduction and literature review

Concrete is an engineered cementitious composite (ECC), and an essential material of civilization, in terms of its total quantity yearly used throughout the world [1]. Concrete is affected by environmental factors and external loads and loses its strength, toughness and endurance due to these effect as time passes. Another characteristic feature of concrete is that the tensile strength is much lower than the compressive strength. Therefore, it needs to be strengthened to withstand tensile stresses. Consequently the researchers have focused on improvement of the weak properties of concrete such as energy absorption, brittle failure, low tensile strength, poor cracking resistance [2].There are many methods of reinforcing concrete in this regard, but the most widely used today is fiber reinforcement. In terms of tensile strength, toughness and ductility, fiber reinforced concrete has excellent mechanical performance[3]. Fiber presences in concrete also prevent or reduce local damages. Fiber reinforced concrete show better impact resistance test results compared to the conventional concrete[4]. Moreover, fiber reinforcement inclusion enhances the bearing capacity of the concrete, and reduce a considerable amount of traditional steel bars especially in prefabricated concrete segments[3,5,6]. A wide variety of fiber types are utilized to improve the mechanical properties of concrete, and the effect of fibers on concrete properties mainly depends on the nature, length, shape and volume fraction of their structures[7]. Macro fiber types such as steel fibers can enhance concrete 's tensile behavior in terms of crack resistance and toughness properties[8]. Microfibers such as polyvinyl alcohol (PVA) fibers can not only prevent concrete 's early plastic cracks but also increase the durability and impact resistance capabilities [9]. Furthermore, PVA fibers can also enhance the flexural creep behavior of concrete[10].

PVA which is a biodegradable polymer with multi-hydroxyl groups, has been widely used in various areas namely packing [11], biomedicine [12], adhesives [13] and reinforcing material [14] . PVA fibers have high modulus of elasticity that helps ECC in restricting crack width [15]. In order to obtain better toughness, and strain hardening properties commonly %2 volume of PVA is utilized in ECC. PVA fibers can form considerably strong chemical bonding with cement as it contains hydroxyl group in its molecular chain, which enhances the isotropic behavior of the material. As a result strong bond forms between PVA fibers and cement matrix [16]. However, such strong bond is undesirable as it can cause pre-mature fibre rupture. With the purpose of preventing such unsolicited situation, surface of PVA fibers are coated with oil which leads decrease in the bond between the fiber and cement matrix [17]. The difference between oiled and unoiled PVA fiber added ECC was also examined by the scholars. Oiled PVA fiber added mixtures exhibits higher compressive strength, tensile strength (7th day and 28th day) than unoiled PVA containing concrete mixtures. However, the both specimens tensile and compressive strength results are higher than mortar [18].

In the recent study PVA added cementitious material was investigated. It is shown that 2% PVA added concrete has shown 4% higher tensile strength performance [17]. PVA inclusion improves bonding strength [19]. PVA fiber along with high fly ash are commonly used to

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improve ductility of ECC. However, high fly ash content results decrease in compressive strength and tensile strength [20]. It is a common practice to hybridize steel fibers, PVA, basalt and polyethylene (PE). In the recent study hybridization 1.0 % steel fiber as the main fiber and 0.5% the second fiber (PVA, BF and PE) was investigated. The mixture which has 1.0% steel fiber and 0.5% PVE fiber of PVA fibers added mixture show higher compressive strength, lower density , lower first and ultimate tensile strength [21]. PVA fiber inclusion also improved durability performance of concrete. It is reported that 2% of PVA fiber inclusion decreases abrasion weight loss [22].

Due to high consumption of natural sources in the production of cement, scholars have been studying alternative materials that can replace cement in the concrete. Cement replacement with metakaolin (MK) results increase in compressive strength. However, the metakaolin replacement decreases the strength of concrete at elevated temperatures. Reason of this decrease are mainly dense micro-structure and lower porosity [23]. In addition to improvement in mechanical properties MK inclusion in concrete develops sulfate resistance [24].

Taguchi method is a statistical method which Genichi Taguchi introduced in the 1950s. In traditional experimental design, all factors are kept constant while changing one factor. The number of experiments will increase as the factors and levels in the design of the experiment increase. Therefore, it will not be possible to make such a design in practice [25]. Also, in the case of internal interaction between factors, the optimum conditions found in comparison to the traditional experimental design may not be the actual optimum conditions. Taguchi Method, one of the experimental design techniques, is successfully applied for optimization in systematic designs [26]. Performance statistics; Signal to Noise (S/N) are used as optimization criteria in Taguchi design. There are three types of performance statistics, the largest - best, nominal - best, and smallest - best. Using the data from the experiments, S/N values are calculated based on the largest best performance statistics S/N [27]. Taguchi methodology has been used in many studies. Especially in concrete experimental design usage of this method has attract more attention in last decade. In the literature, Taguchi method has been used to design optimum mix proportions for geopolymer concrete with ground granulated blast furnace slag as aluminosilicate source at ambient curing condition [28].

Taguchi optimization method is improved to model to optimum mix design of the high strength self-consolidating concrete [29]. TOPSIS based Taguchi method has been used to determine optimal mixture proportions of concrete contains polymers. In this study, researchers conducted an experimental study to statistically investigate the prominence of porosity, and compressive and flexural strength on dry-pressed concrete curbs and the statistical properties of the factors were assessed using Taguchi method [30]. The strengthening with polymer the polypropylene fiber reinforced concrete exposed to high temperature was examined and Taguchi L_9 was used for the design of experiments [31]. General design optimization of glass fibre reinforced concrete was conducted to help professionals in producing industrial concrete facades and walls and the optimum ingredient contents and their relations with concrete performance were determined through combined utilization of Taguchi, ANOVA and extreme vertices design methods [32]. The effect of viscosity and shear regime on air-void system of different self-consolidating concrete mixtures was evaluated using Taguchi method [33]. Taguchi system is utilized, which fuses the acoustic emission feature space to provide comprehensive and reliable degradation indicator with a feature selection method to determine useful features [34].

The Response Surface Design (RSD) was proposed and defined by Box and Wilson in 1951 under the name "Achievement of Trials to Optimum Conditions". It was first applied to the chemical industry. RSD method defined as a method in which statistical and mathematical techniques required for the development and optimization of the processes are used together Myers ve Montgomery [35] The RSD includes empirical modeling techniques to investigate the experimental space of process variables, empirical modeling techniques used to determine the response between the system's response and the independent variables that influence it, and optimization techniques used to find the levels at which the process variables exert the desired effect in the system's response [36] In general, the RSD consists of 3 stages (screening trials, region research and optimization of the process or product). Screening trials allow fewer and more efficient core trials. The aim of the second stage, the regional research, is to determine whether the values generated by the independent variables determined by the screening trials give results close to the optimum point. The third stage of the RSD begins when the process is approached to the optimum point. The actual response function shows a significant curvature around the optimum point. Nonlinear models, quadratic polynomial models or exponential models are generally used in estimating this curvature. Once a suitable model has been obtained, this model is used to investigate the optimum point [37]

In this research, an optimization and design tool was proposed with the combined utilization of Taguchi and response surface design methods in order to analyze the effects of Cement,

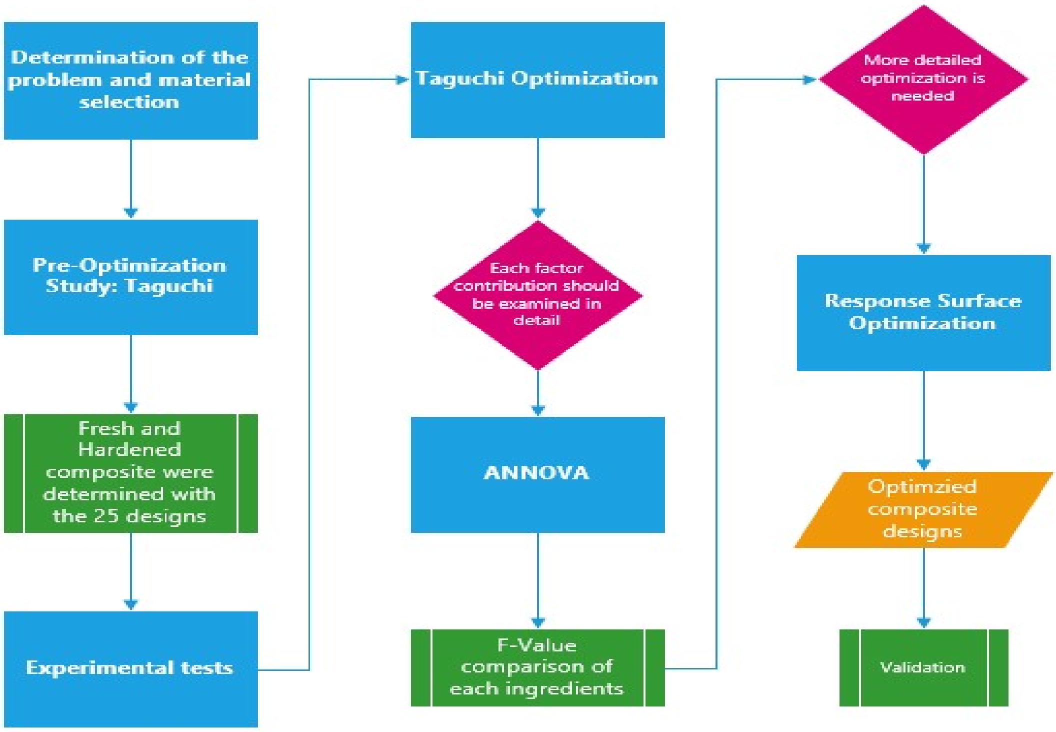

Fig. 1 - Flow chart of the proposed system

metakaolin, PVA fibers and fly ash on the some material properties of the PVA added composites. Subsequently, the optimal content of ingredients was obtained and evaluated using the proposed optimization system. Thus, this study is also aimed to contribute the related composite ingredients optimization studies by limiting the cement utilization.

2.Experimental and Optimization Program

The number of experiments to be carried out was reduced to 25 with the Taguchi optimization method, in this research. In addition, the recommended mixing ratios were obtained by this preliminary optimization study. Fresh and Hardened composite properties were determined with the 25 designs. Taguchi optimization were applied according to the experimental test results. F-value comparison of each ingredients were analyzed via ANOVA to list the most effective factors on the strengths based on the Taguchi optimization results. Response Surface Design was applied to the output parameters of the ANOVA to obtain better and detailed clarifications of relations between the input parameters and the strengths. Decision making chart of the proposed system is presented in Figure 1.

2.1. Materials, mixing proportions and test methods

Portland Cement utilized was grade 52.5 R in Turkish standard TS EN 197 with specific surface of 4500 cm^2/g and specific gravity of 3.08. Low Ca containing fly ash conforming to TS EN 450-1 standard was used with specific gravity of 2.11 and specific surface of 2412 cm^2/g. The chemical composition of the cement and fly ash are given in Table 1.

8 mm PVA fiber were employed in this study. Fiber properties are presented in Table 2. Silica sand with the AFS values of 30 to 35 was used, and its material properties were listed in Table 3 and Figure 2, respectively.

A commercially available Metakaolin (MK) was also utilized in this research. The chemical and physical characteristics of MK are given in Table 4. Potable water used, and polycarboxylate based hyper plasticizer was also applied to adjust the workability of the fresh cementitious composites.

In this present research, a low fraction of PVA fiber (< 1.5 %) was utilized in order provide the uniform distribution and good workability. Five PVA fiber contents (2.0 %, 2.5%, 3.0 %, 3.5 % and 4.0 % by weight) were added to the composites with water to binder ratio of 0.40. A total of 25 composite samples were prepared according to the Taguchi analysis recommendations. All mixture proportions are given in Table 5.

The composites were prepared using a concrete mixer. Cement, fly ash, silica sand and metakaolin were initially dry mixed for 3 minutes. Then a third of hyper plasticizer, PVA fibers, and water were added into the mixture and mixed for 2 minutes. This process repeated three times, and total mixing time was provided as 9 minutes in order to provide a uniform distribution of the ingredients. All specimens were demolded after 24

Table 1

Chemical composition of cement and fly ash

Chemical content (%)	Cement	Fly ash
SiO_2	21.5	51.12
Al_2O_3	4.03	17.74
Fe_2O_3	0.26	6.51
CaO	65.7	9.02
MgO	1.30	3.19
Na_2O	0.29	2.24
K_2O	0.34	2.04
SO_3	3.31	0.21

Table 2

Properties of PVA fiber

Diameter (μm)	Length (mm)	Density (g/cm^3)	Tensile strength (MPa)	Elastic Modulus (GPa)	Melting point (C°)	Water absorption (%)
25	8	1.35	1500	38	215	<1

Table 3

Properties of silica sand

Physical properties	
Clay content (%)	0.6-0.75
Specific weight	2.69
AFS value	34.2
Chemical composition (%)	
SiO_2	98.55
Fe_2O_3	0.13
MgO	0.03
CaO	0.01
K_2O	0.09
Na_2O	0.02
Al_2O_3	1.11

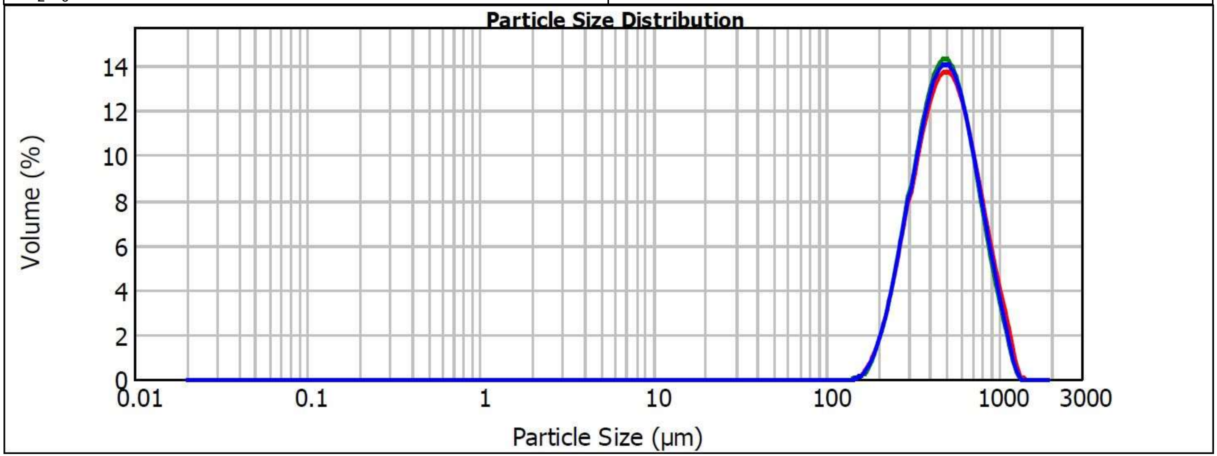

Fig. 2 - Particle size distribution of Silica Sand

Table 4

Chemical and physical characteristics of MK

Chemical and Physical properties	
Density (ISO 787-10) (g/cm3)	2.6
Hardness (Mohs)	2.2
Refractive Index	1.56
pH (ISO-787-9)	7
Oil absorption (ISO-787-5)	29g/100g
Density (Bulk, ISO 787-11) (g/ml)	0.62
Brightness CIE L (ASTM E-313)	93
Brightness Ry (DIN 53163)(%)	84
SiO_2 (%)	<76
Al_2O_3 (%)	>15
Fe_2O_3 (%)	<0.5
Insoluble HCl (%)	85
Loss on ignition (%)	8

Table 5

Mixture proportions of the composites

Mix	Cement (kg/m^3)	Fly ash (kg/m^3)	Silica sand (kg/m^3)	MK (kg/m^3)	Water/binder	PVA fiber (% by wt.)	Hyper plasticizer (kg/m^3)
C_0	750	0	550	0	0.40	2	3
C_1	750	41.66	550	41.66	0.40	2.5	3
C_2	750	83.33	550	83.33	0.40	3	3
C_3	750	125	550	125	0.40	3.5	3
C_4	750	166.66	550	166.66	0.40	4	3
C_5	770	83.33	550	41.66	0.40	2	3
C_6	770	125	550	83.33	0.40	2.5	3
C_7	770	166.66	550	125	0.40	3	3
C_8	770	0	550	166.66	0.40	3.5	3
C_9	770	41.66	550	0	0.40	4	3
C_{10}	790	166.66	550	83.33	0.40	2	3
C_{11}	790	0	550	125	0.40	2.5	3
C_{12}	790	41.66	550	166.66	0.40	3	3
C_{13}	790	83.33	550	0	0.40	3.5	3
C_{14}	790	125	550	41.66	0.40	4	3
C_{15}	810	41.66	550	125	0.40	2	3
C_{16}	810	83.33	550	166.66	0.40	2.5	3
C_{17}	810	125	550	0	0.40	3	3
C_{18}	810	166.66	550	41.66	0.40	3.5	3
C_{19}	810	0	550	83.33	0.40	4	3
C_{20}	833.33	125	550	166.66	0.40	2	3
C_{21}	833.33	166.66	550	0	0.40	2.5	3
C_{22}	833.33	0	550	41.66	0.40	3	3
C_{23}	833.33	41.66	550	83.33	0.40	3.5	3
C_{24}	833.33	83.33	550	125	0.40	4	3

hour and cured in 20 ° C and 95 % humidity until mechanical tests.

Slump test was conducted according to the requirements of EN 12350-1 [38] due to the fact that PVA fiber and MK inclusions reduced the spread diameter. Slump test were repeated two times, and the reported results are the averages for the all mixes. Water absorption of the samples were also recorded as per the ASTM C642[39] requirements. Compressive strength tests were performed on the Ø150/300 mm cylindrical specimens according to ASTM C 469 standard[40]. Flexural strength tests. were conducted with 100 x 100 x 500 mm prismatic samples according to the EN 14651[41]. Compressive and flexural strength test results were recorded at 7,14 and 28 days. Freezing and thawing resistance of the specimens were determined by the requirements of ASTM C 666[42]. The place of the specimens was frequently changed during the F&T experiments in order to obtain extreme temperature temperatures at different locations within the test cabinet. Based on the strength differences between the no F&T process applied samples and the specimens subjected to the F&T cycles, strength losses were measured as percentages.

2.2. Optimization studies

The Taguchi design of experiment method was used to determine optimal mixtures of the ingredients of prepared composites to maximize compressive and flexural strengths. Extensive results were obtained with a small number of trials with the application of this technique. Four factors related to the strength properties such as cement, metakaolin, PVA fiber, and fly ash contents were evaluated via Taguchi method. Ingredients content recommendations. Experimental factors and their levels in Taguchi design are given in Table 6. Signal-to-noise (S/N) ratios were applied as ‘Larger is better’ for the strength evaluations. The contribution of each factor on the strengths was also determined through ANOVA.

Many test methods relating to concrete can be implemented. Apart from the existing methods, many methods are developed and extensive research are being performed for their progress. Factors affecting concrete performance and properties are the components used in the concrete mixture. Response Surface Design was applied to the output parameters of the Taguchi and ANOVA in order to obtain better clarifications of relations between the factors and the strengths. Since there are more than one factor, the way to evaluate the effects more accurately is to consider them volumetrically. And the application of RSD in concrete studies were successfully preformed in many researches[43–45]; therefore, an analysis was developed in this study with RSD methodology.

3.Results and Discussion

3.1. Experimental Results

Water to cement ratio was selected to be 0.4 in all specimens. Compressive strength test results for 7, 14 and 28 days are shown in Fig. 3. Metakaolin has positive impact on compressive strength of concrete mixtures. This can be attributed to its particle size that fills the voids of

Table 6

Experimental factors and their levels (per 0.06 m^3 mixtures)

Factors	Level 1	Level 2	Level 3	Level 4	Level 5
Cement	45	46.2	47.4	48.6	50
PVA	2	2.5	3	3.5	4
Metakaolin	0	2.5	5	7.5	10
Fly ash	0	2.5	5	7.5	10

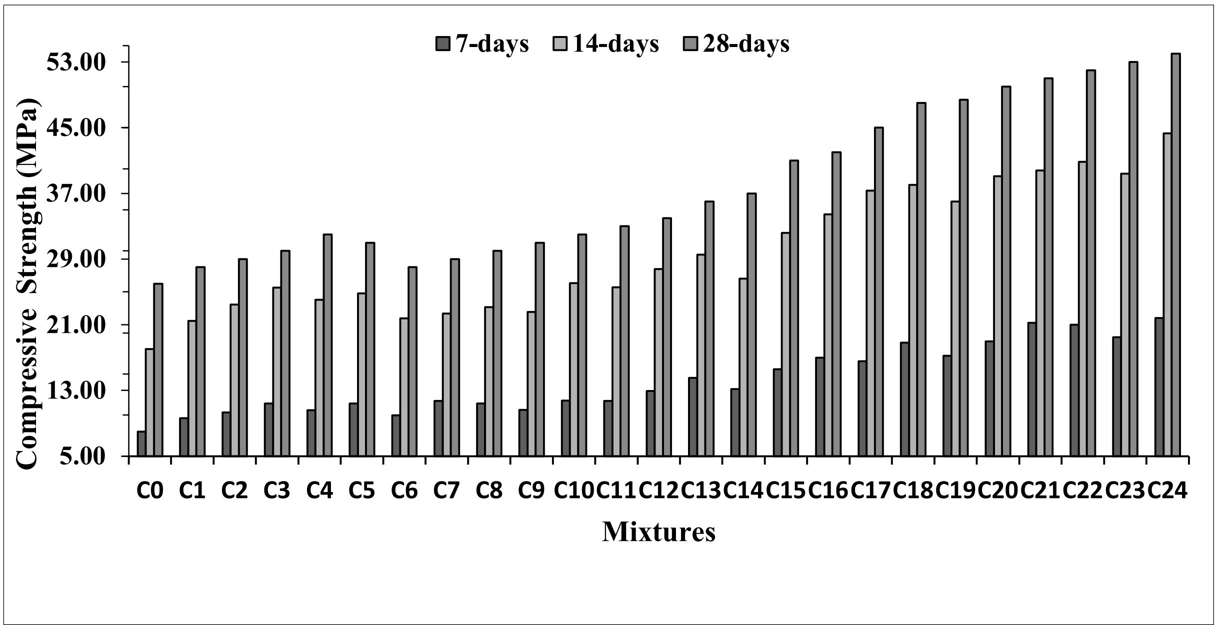

Fig. 3 - Compressive Strength Test Results

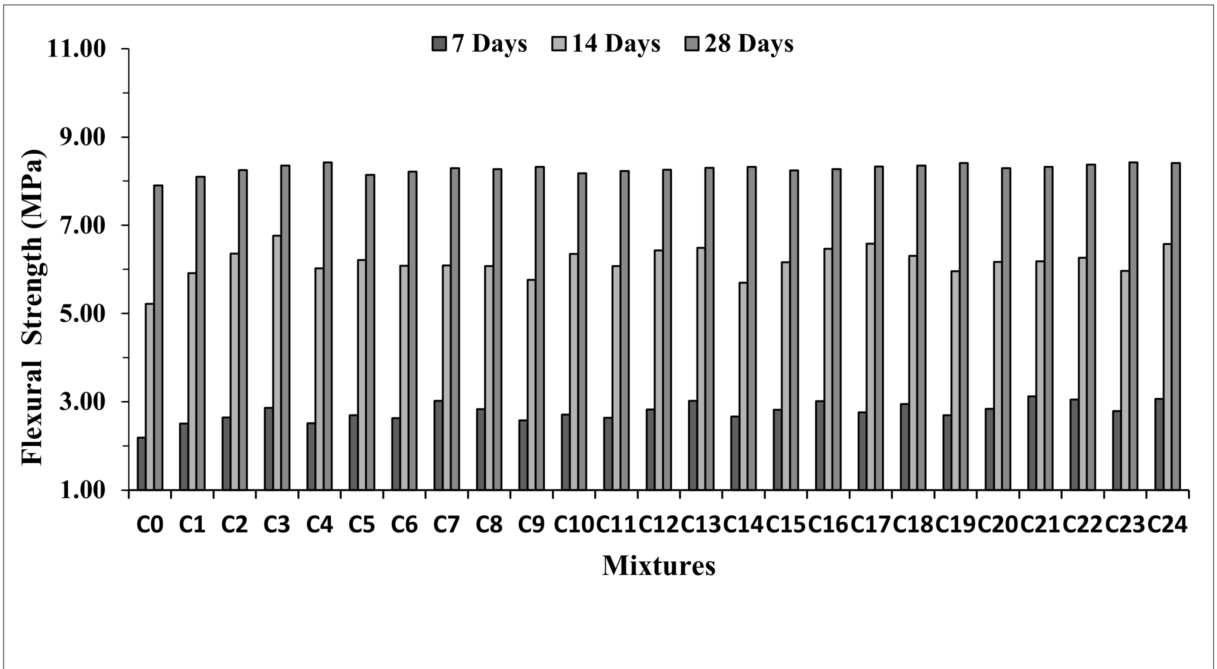

Fig. 4 - Flexural Strength Test Results

concrete and also increase pozzolanic reactions. Furthermore, PVA fiber improves compressive strength of concrete mixtures. The highest compressive strength was determined to be 54 MPa in the mixture that has 5% fly ash, 7.5% metakaolin and 4% fiber.

Flexural strength can be improved by using PVA fiber[46]. During the laboratory tests it was observed that with the increase of fiber and metakaolin the flexural strength was improved. This can be due to flexibility property of fiber and pozzolanic property of metakaolin which leads increase in early strength of concrete. The flexural strength test results are presented in Fig.4.

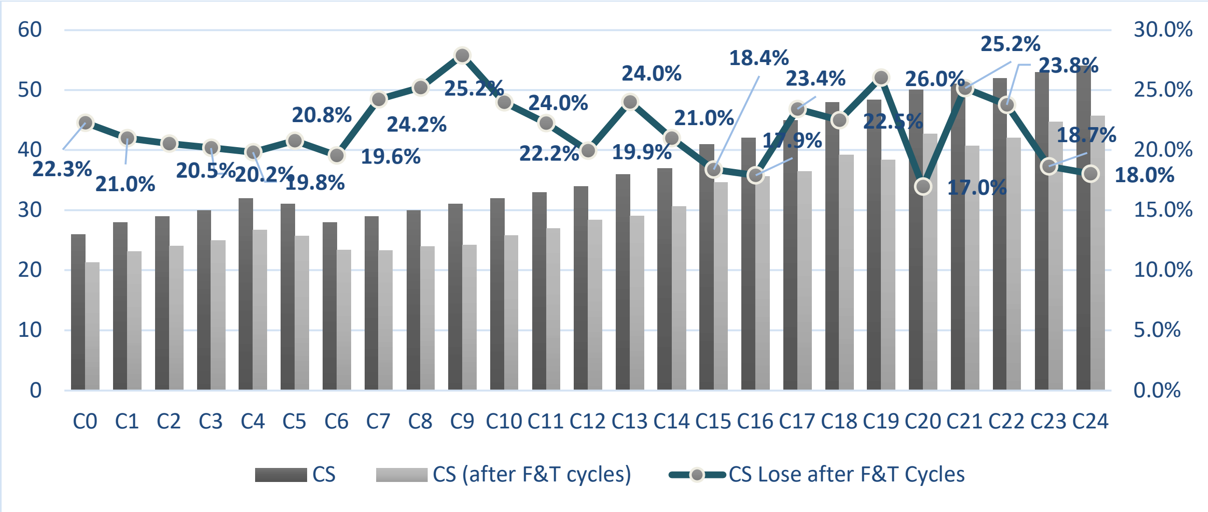

Fig. 5 - Compressive Strength Test Results after Freeze and Thaw Cycles

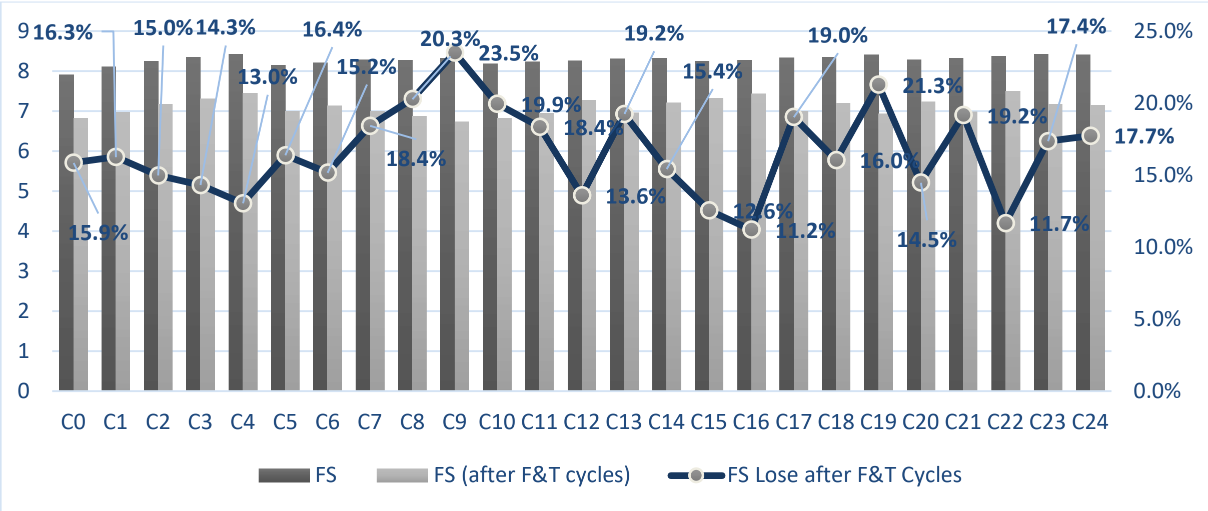

Fig. 6 - Flexural Strength Test Results after Freeze and Thaw Cycles

Freeze and thaw resistance property is one of the most common issues that encountered during the lifecycle of concrete. The concrete mixtures that exposed to freeze and thaw cycles were performed both compressive and flexural strength tests. These test results and lose of strength after freeze and thaw cycles are presented in Fig.5. and Fig 6.

Metakaolin with the filler effect and reducing air voids, the CS lose after freeze and thaw cycles are prone to reduce. This proves that less water absorption results decrease in strength lose after freeze and thaw cycles. In terms of air voids metakaolin and fly ash show good performance in improving durability properties of cementitious composites.

3.2. Optimization Results and Validation

Compressive strength optimization results based on the Taguchi method are given in Figure 7. According to the design results optimum results were obtained as 50 kg cement, 4 % PVA fiber, 2.5 % MK and 10 % fly ash contents. The compressive strength test results increased from 26 MPa to 54 MPa with the increase cement, fly ash and fiber contents, which is consistent with the results from literatures[47–49]. However, utilization of MK more than 2.5 % decreased the compressive strength test results. This can be attribute to the combined usage of MK, silica sand and Fly ash[50]. In addition, composites containing MK showed better performances compared to the MK-free mixtures.

Flexural strength optimization results are plotted in Figure 8. Optimum conditions for flexural strength are 50 kg cement, 4 % PVA fiber, 10 % MK and 10 % fly ash contents. All flexural strength results increased with the increase in all ingredients. These results are also consistent with the previous researches[51,52]. The enhancement in mechanical properties are due to filling effect of MK and fly ash among voids and unhydrated cement particles, therefore decreasing porosity[53,54].

Table 7 and 8 show the ANOVA results for compressive and flexural strengths at 28 days, respectively. As shown in Table 7 cement is the most effective parameter on compressive strength development, since F-value is greater compared to the other parameters. For flexural strength

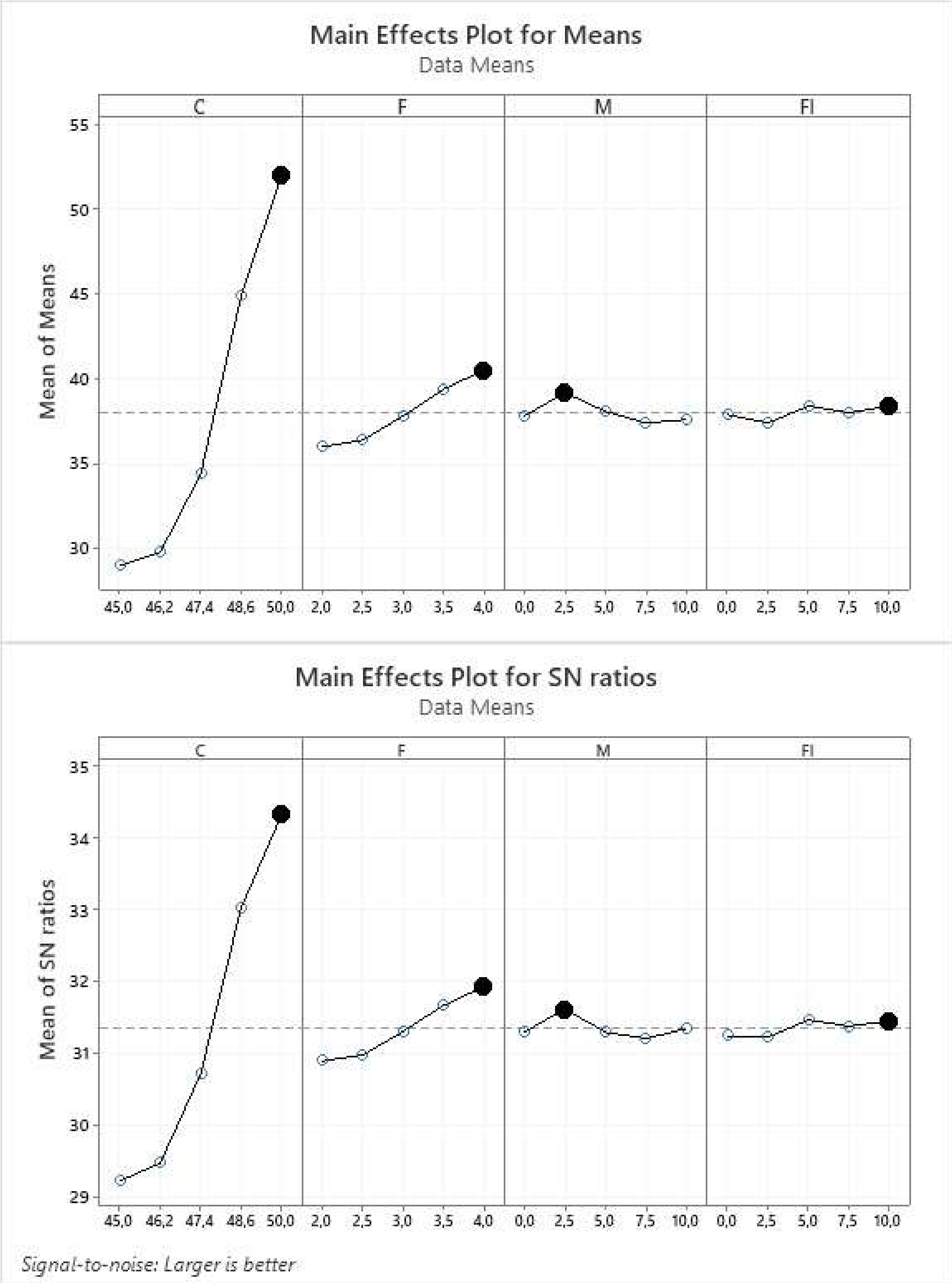

Fig. 7 - Compressive Strength - Taguchi Optimization Results

Table 7

ANOVA results for compressive strength at 28 days

Factor	d_f	Sum of Square (SS)	Adj mean of square (MS)	F-Value	P-Value
Cement	4	2022.67	505.666	324.89	0.00
PVA fiber	4	73.55	18.386	11.81	0.002
MK	4	10.03	2.506	1.61	0.262
Fly ash	4	3.47	0.866	0.56	0.701
Error	8	12.45	1.556		
Total	24				

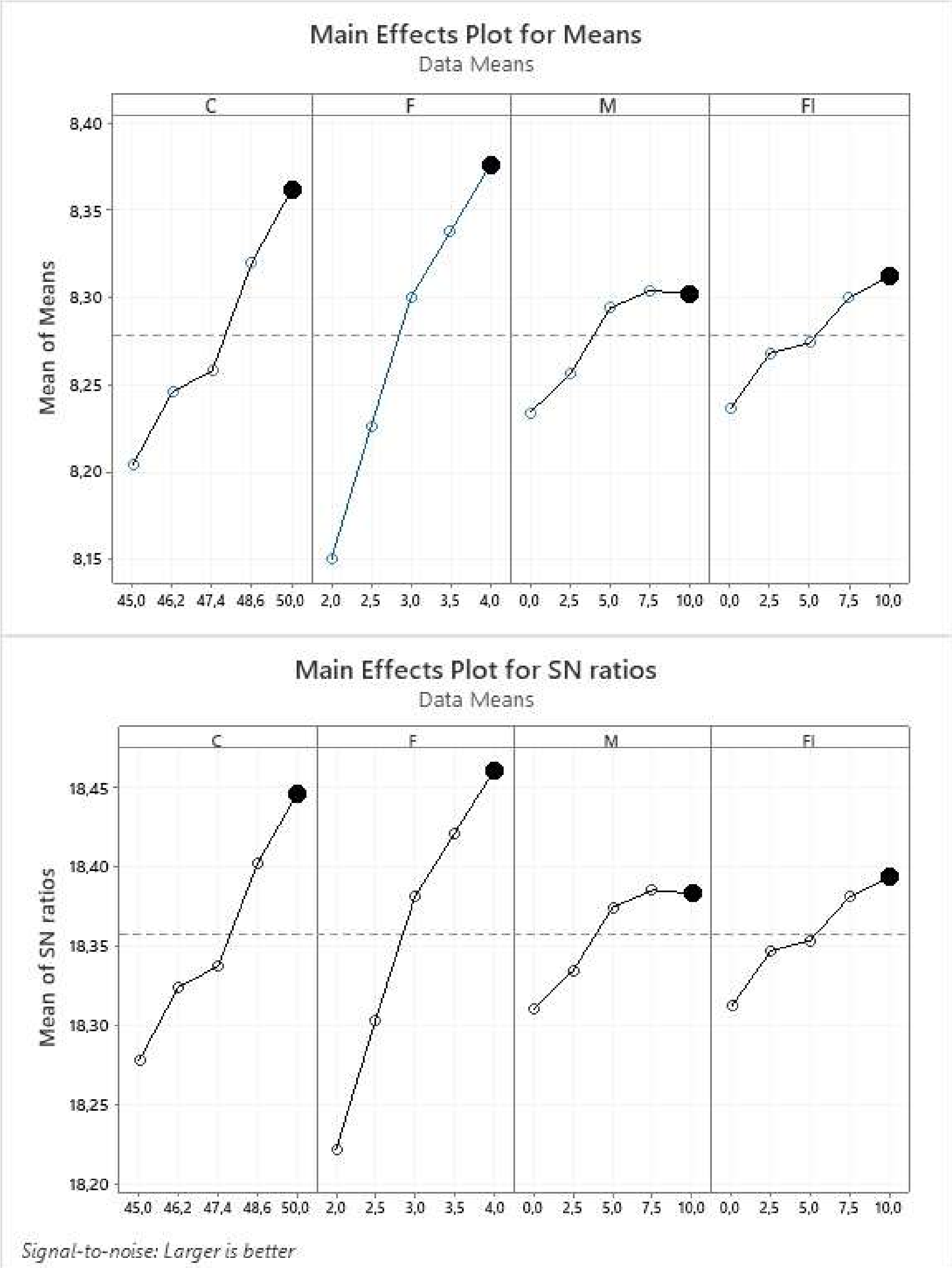

Fig. 8 - Flexural Strength -Taguchi Optimization Results

Table 8

ANOVA results for flexural strength at 28 days

Factor	d_f	Sum of Square (SS)	Adj mean of square (MS)	F-Value	P-Value
Cement	4	0.07860	0.019650	4.38	0.036
PVA fiber	4	0.16388	0.040970	9.13	0.004
MK	4	0.01964	0.004910	1.09	0.421
Fly ash	4	0.01760	0.004400		
Error	8	0.03588	0.004485		
Total	24				

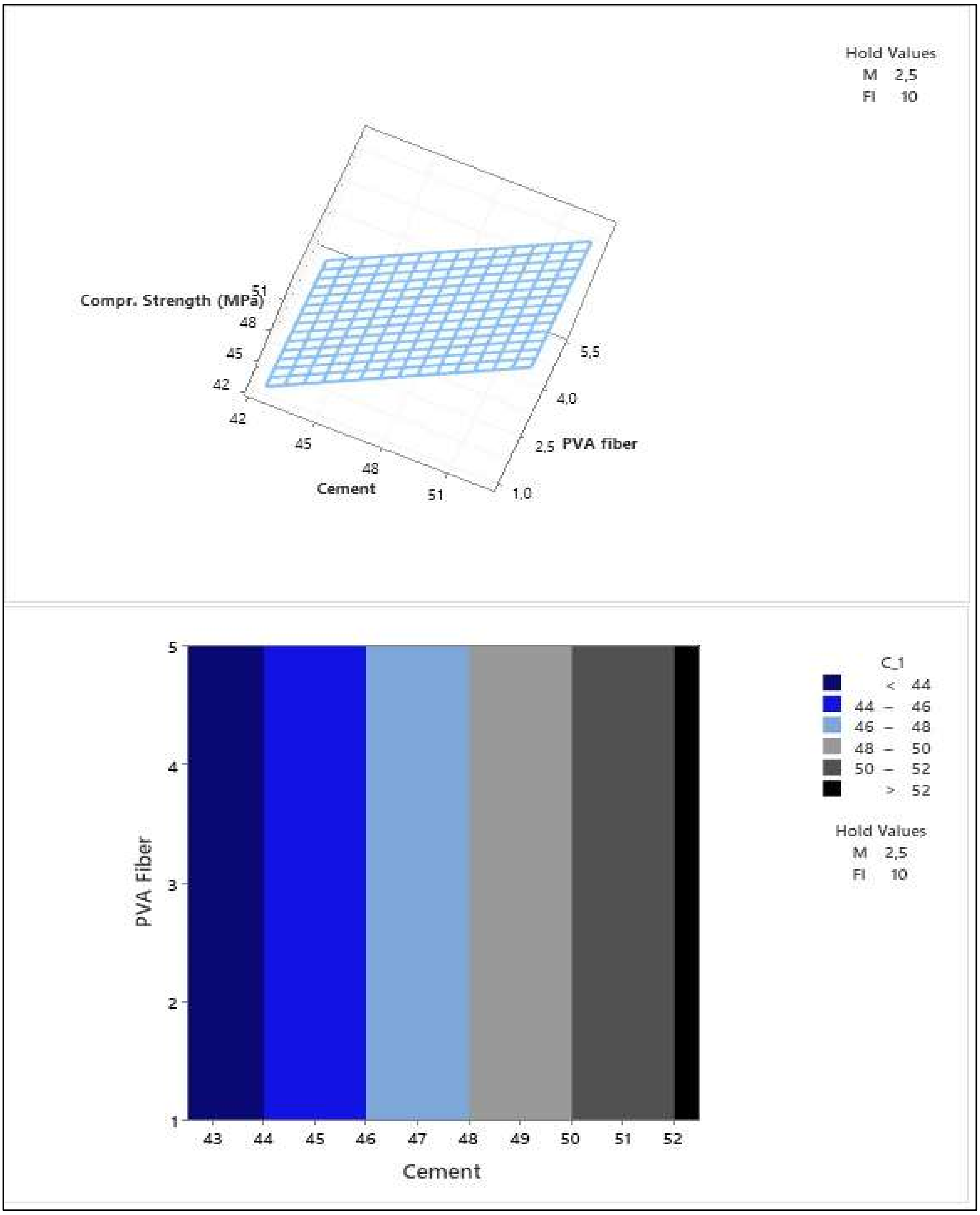

Fig.9 - Compressive Strength -Response Surface Optimization Results

development, PVA fiber contribution is higher compared with other parameters. The contribution of any factor cannot be ignored by examining the results in detail. Therefore, all factors were used during RSD process.

Response surface design results of compressive and flexural strength are plotted in Figure 9 and 10, respectively. Optimum ingredients content for compressive strength was obtained as 50 kg cement, 3 % PVA fiber, 2.5 % MK and 10 % fly ash. RSD results also show that optimum parameters for flexural strength were 50 kg cement, 4 % PVA fiber, 10 % MK and 10 % fly ash. The analysis results were validated through experimental tests to obtain the final mixture designs. It was also clearly seen that all variables utilized in the proposed models have an impact on the strengths within the scope of this research. As expected, an increase is seen from the Figure 10 with the increasing variables cement and PVA fiber. This situation is also supported by other studies in the literature[55,56]. In terms of compressive strength, cement is the main effecting responsible for the increasing trend. PVA fiber

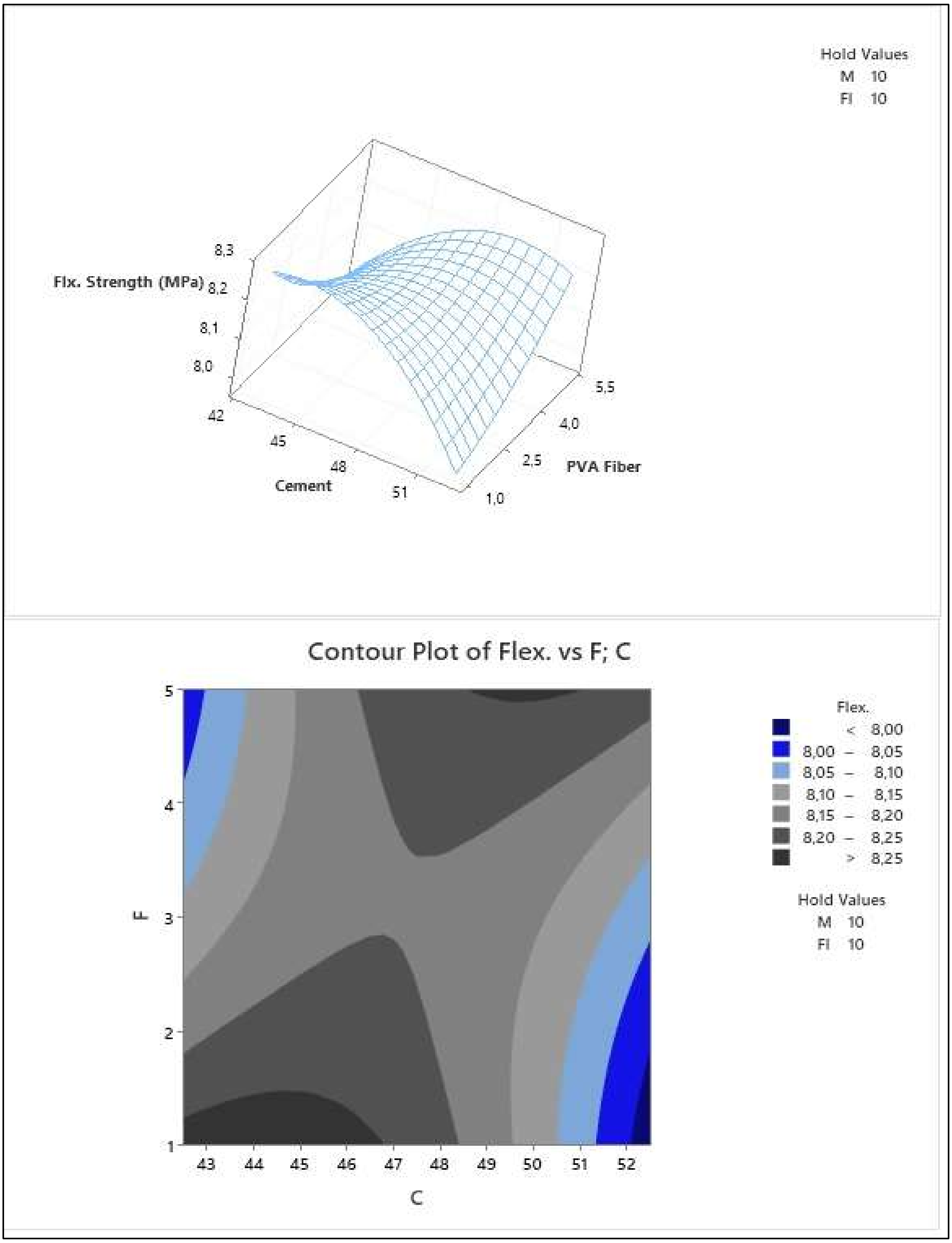

Fig. 10 - Flexural Strength -Response Surface Optimization Results

addition had very limited effect on the compressive strength. It has been experimentally confirmed that PVA inclusion increased the porosity, thus reduced the compressive strength[57]. However, according to the developed model, an increase was obtained with the increasing PVA fiber content. This can be contributed to the combined utilization of PVA, MK and Fly ash. Furthermore, the main factor behind this situation is the porosity filling effect of MK and Fly ash. This results are also consistent with the similar researches[58–61].

The mixture designs of the validation samples are presented in Table 9. Three samples for each dataset were prepared during validation. The validation test results at 28 days are presented in Table 9. As shown in Table 9, the proposed optimization model can be successfully applied to PVA fiber reinforced concrete containing MK and Fly ash.

Table 9

Validation Test Results for 0.06 m3(one bucket)

Sample	Cement(kg)	Fly Ash (%)	MK (%)	PVA fiber (%)	Test Result
C_{comp}	50	10%	2.50%	3%	49.58 Mpa
$C_{flexural}$	50	10%	10%	4%	7.8 Mpa

PVA fiber has bigger impact on flexural strength than it has over compressive strength of concrete samples. However, PVA fiber should be up to 5% in the samples in order to obtain the desired results.

$$RSD = \frac{Experimental - Model}{Experimental}\ x\ 100 \quad (1)$$

With the purpose of testing the validity of the developed model the eq. 1 is used [62]. If the result of the equation is between 0.5 to 7.5 this proves the reliability of the model. The result of this equation for compressive strength 4 % and 5 % is flexural strength. These results were determined to prove model's high accuracy.

4. Conclusion

In this research, a hybrid model was proposed by combined utilization of Taguchi optimization, ANOVA and Response Surface Design to optimize PVA fiber reinforced cementitious composites. The analysis results were also applied in the laboratory, and validation tests were conducted. The main conclusions can be drawn as follows:

- The water absorption results reduced with the inclusion of MK and fly ash. This decreased the air voids in the concrete samples due to the chemical hydration reactions are led by MK, and particle size of fly as it fills voids. Consequently, the effect of freeze and thaw cycles decreased, as well. According to the results MK and fly ash inclusion enhances freeze and thaw resistance properties of concrete samples
- Compressive strength increases with MK addition as MK is an active pozzolanic material and generates hydration products. This also increases early age strength. Also, according to the optimization results, MK proportions are to be used limited ratios.
- PVA fiber, MK and Fly ash addition had a combined effect on the compressive strength in increasing manner; however, only PVA fiber inclusion decreases the strength values in terms of compressive testing.
- The proposed model also enables to estimate the strength test results and decrease the required test numbers. In addition, less time and low laboratory cost can be achieved with the developed system.

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