Influence of Mix Design Variables on Engineering Properties of Carbon Fiber-Modified Electrically Conductive Concrete

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ABSTRACT

This research was inspired by the need to optimize the mix design of electrically conductive concrete (ECON) for field implementation. Carbon fiber was used for producing ECON with different mixing proportions and constituents. Calcium nitrite-based corrosion inhibitor admixture and methylcellulose were used as conductivity-enhancing agent (CEA) and fiber-dispersive agent (FDA) respectively. Five easy-to-change mix design variables were evaluated for their effects on electrical conductivity and strength of ECON: carbon fiber dosage, fiber length, coarse-to-fine aggregate volume ratio (C/F), CEA dosage, and FDA dosage. The results approved the effectiveness of the applied CEA in improving electrical conductivity while positively influencing strength. Conductivity was significantly influenced by: fiber content, C/F, fiber length, and CEA dosage. The dosages of Fiber, CEA, and FDA exerted significant influence on compressive strength. C/F and FDA dosage were significant variables influencing flexural strength.

Keywords: Electrically conductive concrete (ECON), Carbon fiber, Mix design, Fiber content, Corrosion inhibitor admixture, Methylcellulose.

Abbreviations: PC – Portland cement, PCC - Portland cement concrete, ECON – Electrically conductive concrete, ECA - electrically conductive additive, CEA – conductivity-enhancing agent, FDA – fiber-dispersive agent, DOE – design of experiments, C/F- coarse-to-fine aggregate volume ratio.

1 Introduction

Electrically conductive concrete (ECON) is a versatile type of concrete with potential benefits in different applications such as: self-sensing construction material for structural health monitoring practices [1–3], electromagnetic radiation reflector for electromagnetic interference (EMI) shielding [4–6], and resistance heating material in self-heating pavement systems [7–10]. Recent attention to ECON is mainly related to self-heating pavement systems [8,11,12] because of the inadequacy of common ice and snow removal methods [12–15].

The basic mixture components of ECON are cementitious materials, coarse and fine aggregates, water, electrically conductive additive (ECA), and possibly chemical admixtures [11]. Air-dried normal Portland cement concrete (PCC) has an electrical resistivity in a range between 6.54×10^5 and 11.4×10^5 Ω-cm [16], while, electrical resistivity of ECON is orders of magnitude lower (30 Ω-cm to 1.00×10^4 Ω-cm) [4,12,17–20]. The primary source of electrical conductivity that renders ECON considerably more conductive than normal concrete is the ECA component that creates a continuous path for electricity conduction. ECA portion of ECON may consist of a single material or a mixture of two or more different materials all possessing high electrical conductivity. Forming a continuous network in concrete matrix by the ECA materials is referred to as percolation phenomenon and the volume content of ECA enabling the percolation is called the percolation threshold [21–23].

Since 1965, when the first patent related to ECON was issued [18], numerous mix designs have been proposed for production of electrically conductive cement paste, mortar, or concrete [4,22,24]. Carbon fiber is a material that has been used and tested as an ECA in production of electrically conductive cementitious composites for different purposes [7,25,26]. Moreover, previous research have postulated [27–29] that carbon fiber-reinforced concrete provides better characteristics in terms of freeze-thaw durability, tensile strength, fatigue cracking, shrinkage cracking potential, and expansion cracking susceptibility. Wu et al. [12] produced carbon fiber-modified ECON with 4,000 Ω-cm resistivity using 0.8% (Vol.) carbon fiber dosage. While, Kraus and Naik [30] achieved 127 Ω-cm electrical resistivity with only 0.5% (Vol.) carbon fiber and Galao et al. [31] produced ECON with 40 Ω-cm resistivity using <0.2 % (Vol.) carbon fiber in the concrete mix. This shows that the electrical resistivity of carbon fiber-modified ECON is dependent on multiple factors and not only the carbon fiber dosage. Speaking of carbon fiber-related factors, in addition to carbon fiber dosage [12], the properties of the fibers such as fiber length [21,22], material origin (e.g. pitch-based or polyacrylonitrile-based) [22,32] , and the surface chemistry of carbon fiber strands [32,33] influence the effectiveness of fibers in modifying the properties of concrete. Percolation of fibers in concrete depends on dispersion level, that is, controlled by fiber properties, mixture constituents, mix proportions, and mixing procedure [21,28,34,35]. Improved fiber dispersion leads to improved fiber-cement paste bond, higher ductility, and reduced electrical resistivity [33]. A variety of chemicals can be used for facilitating the dispersion of fibers in concrete mixture; methylcellulose is a fiber dispersive material that is effective in minor dosages [28,36].

In both normal concrete and ECON, the mix design variables such as cement content, aggregate-to-cement volume ratio, and coarse-to-fine aggregate volume ratio (C/F) exert a significant influence on the electrical conductivity of the concrete [37–39]. In addition to the conventional applications of chemical admixtures - such as improvement of workability, air entrainment, etc.-, they can be used for engineering the internal environment of concrete to boost electricity conduction; for instance, calcium nitrite-based corrosion inhibitor admixtures can change the electrical conductivity of concrete [40,41]. While, sodium-based corrosion inhibitors tend to decrease the compressive strength of concrete, calcium compounds do not exert any reducing effect on concrete strength properties; in fact, calcium nitrite, which is an anodic corrosion inhibitor, has been found to increase the 28-day compressive strength of concrete [42]. Due to the presence of different ionic entities within the pore solution, it acts as the primary medium for ion/charge transfer within the concrete. The principal ions in the pore solution that enable the flow of electricity are Ca^{2+}, K^{+}, Na^{+}, SO_4^{2-}, and HO^{-} [16]. Hence, the ionic composition of pore solution and the ion concentration in the pore solution play an important role in electricity conduction by concrete [43]. It was reported, that when calcium nitrite is added to the mix water of concrete, the concentration of nitrite in the pore solution is comparable to the mix water, i.e. the majority of the nitrite is diffused in the pore solution. Furthermore, at high calcium nitrite contents, hydroxyl ions concentration in the pore solution is increased due to competitive adsorption of nitrite ions on the surface of cement hydration products [44,45]. Therefore, calcium nitrite admixture tends to enhance electricity conduction by increasing the ion concentration in the pore solution of concrete.

Different materials have been proposed in the literature to improve the dispersion of synthetic and/or natural fibers. Examples are Acrylic with or without Silica fume, Styrene acrylic [28], Latex with or without Silica fume [46,47], and Methyl cellulose [20]. Effective fiber dispersion by using Acrylic, Styrene Acrylic, and Latex is associated with high dosages of fiber dispersive agent (FDA) – in the range of 10 to 20% by weight of cementitious - [21,28,34]. On the contrary, the dosage of Methyl Cellulose as a FDA in carbon fiber-modified concrete/mortar can be as low as 0.4% by weight of cementitious materials [20,36].

Developing an ECON mix design with desirable multifunctional behavior calls for adjusting all variables/constituents to achieve required electrical resistivity [39]. Furthermore, the final product should possess desirable mechanical properties. The majority of studies on electrically conductive cementitious composites have followed a trial–error approach using trial mix batches [12]. Regarding the heterogeneity of concrete and the uncertainty it adds to the evaluation of different variables' effects on concrete properties [48], generalizing the results of such trial-and-error studies is not an efficient and reliable way for development of a mix design; on the other hand, the heterogeneity of concrete continuum is more significant in fiber-containing concrete [49]. There are limited studies investigating the electrical conductivity of ECON in light of the ratio of mixture constituents. Wen and Chung [38] studied the double percolation of carbon fibers and cement paste in mortar. Baeza et al. [39] investigated the double and triple percolation of carbon fibers, cement paste, and mortar within a concrete mixture. Another example is the study by Shi [37] that investigated the effect of cement composition, aggregate content, and mineral admixtures on electrical resistivity of plain concrete.

Application of ECON in a real project calls for developing a project-specific mix design or using a previously proposed mix design. However, most ECON products presented in the existing literature suffer from disadvantages such as inadequate strength, high cost, low workability, and poor functionality [50]. On the other hand, developing a mix design in a timely manner requires knowledge about the effect of each component on the final product's characteristics. The dynamic of modern markets being profit-driven [51], necessitates thorough investigation of any new technology –such as ECON- before it gains wide acceptance by the industries. Therefore, the needs have arisen to investigate the role of each component of ECON in order to enable the producers to tailor the final product to their needs.

The use of statistical design of experiments (DOE) and regression analysis of the results is a powerful means of evaluating the influence of different variables on concrete characteristics [52]. By this method, the main effects of single variables and the interactions can be quantitatively evaluated with a certain confidence level [53]. ECON mix designs based on such pre-defined, structured DOE will be more rational and universal than those based on trial–error approach.

The primary objective of this study is to identify the effects of easy-to-change mix design variables on electrical conductivity and mechanical properties of ECON. The findings of this paper can provide a basis for developing an optimized ECON mix design that satisfies both electrical conductivity and strength requirements for a given project with efficient amount of ECA materials and admixtures. Therefore, it is worth mentioning that producing high-conductivity ECON samples did not lie within the scope of this research; rather, it was attempted to change different variables to evaluate their respective effects on the product characteristics. Furthermore, this paper -for the first time in the literature- investigates the application of calcium nitrite-based corrosion inhibitor admixtures as a conductivity-enhancing agent in ECON production. A statistical DOE was performed to develop an experimental plan for investigating the effects of five mix design variables on three different responses; then, the measurement results were analyzed by regression analysis.

2 Methodology and materials

2.1 Mix design variables

Selected variables were carbon fiber content, carbon fiber length, Coarse-to-fine aggregate volume ratio (C/F), fiber-dispersive agent (FDA) dosage, and conductivity-enhancing agent (CEA) dosage (Table 1). Since electrical conductivity of concrete decreases with increase of aggregate volume [37], C/F ratio was used as a mix design variable instead of total aggregate volume. Methylcellulose and corrosion inhibitor admixture were used as FDA and CEA respectively.

A screening experiment design (a.k.a. 2^k design, k being the number of variables) was used for evaluating the effect of each variable at two levels. Screening DOE enables the number of experiments for each variable to be minimized [53]. By applying a fractional factorial design, it was made possible to evaluate variable effects with reduced number of experiments. Nineteen ECON types, including three replicate center points, were produced. Table 2 gives the variable combinations for each mix design. The responses of the statistical model were electrical resistivity, compressive strength, and flexural strength. The DOE and analysis of results were performed using a commercial software (JMP®).

Table 1. Variable description in the screening DOE.

Variable	Unit	Levels		Variable type
Carbon fiber content	% of total mix volume (% Vol.)	0.1	1.0	Continuous
Carbon fiber length	mm	6.0	12.0	Categorical
C/F	N.A.	0.7	1.2	Continuous
FDA dosage	% of the cement weight	0.0	0.4	Continuous
CEA dosage	kg/m^3	0.0	15.0	Continuous

Note: N.A - Not Applicable.

Table 2. Combination of variables for ECON mix designs.

Mix design No.	Variable				
	Fiber length (mm)	Fiber content (% Vol.)	C/F	FDA dosage (% wt. cem.)	CEA dosage (kg/m^3)
1	6	0.10	1.20	0.4	15.0
2	12	0.10	0.70	0.4	15.0
3	12	0.10	1.20	0.4	0.0
4	6	0.10	1.20	0.0	0.0
5*	6	0.55	0.95	0.2	7.5
6	12	0.10	1.20	0.0	15.0
7*	6	0.55	0.95	0.2	7.5
8	12	1.00	0.70	0.4	0.0
9	6	1.00	0.70	0.0	0.0
10	6	0.10	0.70	0.4	0.0
11	6	1.00	0.70	0.4	15.0
12	12	0.10	0.70	0.0	0.0
13	6	1.00	1.20	0.4	0.0
14	12	1.00	1.20	0.0	0.0
15	6	0.10	0.70	0.0	15.0
16*	6	0.55	0.95	0.2	7.5
17	6	1.00	1.20	0.0	15.0
18	12	1.00	1.20	0.4	15.0
19	12	1.00	0.70	0.0	15.0

Note: * sign marks the center points

The percolation transition zone of carbon fiber in mortar and concrete have been reported in the range of 0.4-1 % (Vol.) [12,22]. Therefore, the fiber dosages were selected in the proximity of percolation threshold. Carbon fiber was supplied in three nominal lengths of 3, 6, and 12 mm. Because the fiber length added a categorical variable to the experiments and the higher fiber length is desirable for achieving electrical conductivity in concrete [12], the two longer fibers (6- and 12-mm) were used in order to minimize the number of categorical variables and evaluate the length effect in the higher length range. In addition, a previous study have suggested that carbon fibers with 6-12.7 mm length were more desirable with respect to compressive and flexural strength than shorter fibers [54]. Fiber-dispersive agent was used in the optimum dosage range recommended in the literature [36]. Coarse-to-fine aggregate volume ratio was selected in the ranges recommended by Iowa DOT [55] for Portland cement concretes that can be used for both paving and construction purposes. Conductivity-enhancing agent was used in the manufacturer-recommended dosage range.

2.2 Mixture components

Variation of C/F in a concrete mix requires adjustments to the entire mix proportions. In this study, C/F of the concrete mix design was a variable with two experimental levels and one additional level for center points. Therefore, to maintain the consistency among specimens, three basic normal Portland cement concrete (PCC) mixtures were designed as the basis upon which the ECON mix designs were developed by applying required changes to proportions and/or mixture components. According to the variable combination corresponding to each ECON type, the mix designs were made by replacing given volume fraction of fine aggregate with carbon fiber. In addition to carbon fiber, each ECON mix design had specific admixture requirements. After incorporation of carbon fiber and admixtures into the mix design, the required adjustments to the mix proportions were made in accordance to specific gravity and water absorption capacity of the materials to maintain fixed values of water-to-cement and C/F ratios. The amount of mix water was not changed during mixing; instead, water-reducing agent was used for achieving target slump of 75-100 mm.

Table 3 shows the mix proportions of the three PCC mix design types. Materials used in preparation of samples were as follows:

- ASTM C 33 [56] D-57 Coarse aggregate- nominal maximum size 25 mm.
- Fine aggregate conforming to ASTM C 33.
- ASTM C150 [57] type I/II cement manufactured by Holcim.

- ASTM C 494 [58] high-range water reducing – type F- admixture (MasterGlenium 7500 obtained from BASF).
- Methylcellulose in fine powder form as FDA. The FDA was dissolved in the mix water before being added to the batch. The FDA powder was gradually added to the water and hand-mixed in during 30 minutes; blender mixing was not used in order to prevent foaming.
- WR Grace & Co. Derex Corrosion Inhibitor (DCI) admixture used as CEA. Mix designs accounted for the extra water added to the mix by DCI.
- Chopped carbon fiber was polyacrylonitrile(PAN)-based with 7.2 μm diameter, 95% carbon content, and electrical resistivity of 1.55×10^{-3} Ω-cm. PAN-based carbon fiber gives better electrical conductivity than pitch-based types [12]. Two different length size classes of the same type carbon fiber were used, namely PX35-0.25 and PX35-0.50 with respectively 6 mm and 12 mm nominal length. Specific gravity and water absorption capacity of the carbon fiber were 1.81 and 7.35 (% wt.) respectively.

Table 3. Mix proportions of the basic PCC mixtures.

Component	Properties			Mix design C/F ratio		
				0.7	0.95	1.2
	Type	Specific gravity	Absorption (% wt.)	Volume fraction		
Cement	Type I/II	3.15	-	0.135	0.135	0.135
Coarse aggregate	Lime stone	2.67	1.4	0.290	0.334	0.378
Fine aggregate	River sand	2.49	1.7	0.395	0.351	0.307
Water	Tap water	1.00	-	0.180	0.180	0.180

2.3 Sample preparation and electrical resistivity measurement

The ECON mix designs, as explained in section 2.1, were used for making concrete samples. The batches were mixed using a 0.5 m^3-capacity rotating pan mixer. Three batches were prepared with each mix design. From each batch three 100 × 200 mm cylinders, three 75×75×300 mm beams, and three 100×100×100 mm cubic specimens were prepared for compressive strength, flexural strength, and electrical resistivity measurements respectively. All specimens were cured at 100% relative humidity and 23° C temperature during the entire study. Compressive and flexural strength tests were respectively performed according to ASTM C 39 [59] and ASTM C 78 [60]. Electrical resistivity was measured at three ages (3, 7, and 28 days), while, compressive strength, and flexural strength were measured at 28-days.

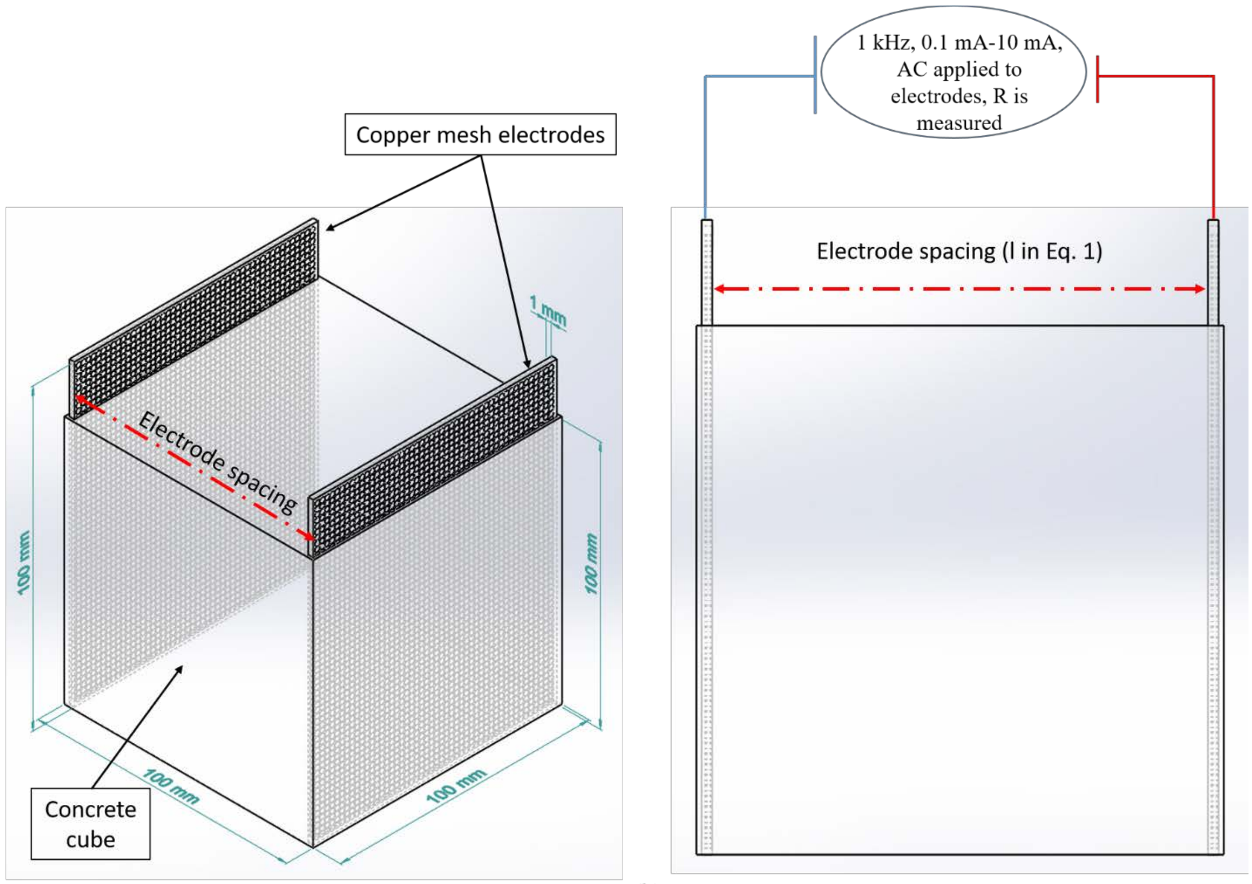
The electrical resistance of a concrete specimen can be measured using direct current (DC) or alternating current (AC). The use of AC measurement is preferred due to difficulties associated with polarization effect and permanent microstructure changes induced by DC methods [61]. Commonly used techniques for measuring electrical resistivity of concrete are electrode probe method, Wenner probe and rapid chloride permeability test (RCPT). RCPT uses high voltage and DC current that limit each sample to only one measurement at a certain age because of permanent microstructure changes made to the concrete [61,62]. Wenner probe technique uses low frequency AC that is associated with polarization; on the other hand, Wenner probe is designed to measure surface resistivity and has high dependency on surface texture, surface moisture, and specimen geometry[62]. Using electrode probes with AC measurements can determine the bulk resistivity of concrete specimens in a repeatable manner [62]. Electrode probe method has the advantages of rapid testing, repeatability, and simple geometry factor [62,63]. Using AC with a frequency of 1,000 Hz eliminates the problems caused by electrode polarization and reactance [61].

The experimental setup for measuring electrical resistivity consisted of two copper mesh electrodes of compatible cross section embedded inside the concrete cubes as shown in Figure 1. Ohmic resistance across the concrete was measured on hardened specimens and electrical resistivity was calculated using equation 1:

$$\rho = R\ (A/l) \qquad \text{(Eq. 1)}$$

Where, ρ represents the electrical resistivity in ohm-centimeters (Ω-cm), R is the electrical resistance in Ohms (Ω), A (cm^2) is the cross sectional area between the electrodes normal to the current direction, and l (cm) is the electrode spacing measured for each specimen.

Factors affecting the measurement in electrode probe method are: (1) voltage, current, and frequency, (2) specimen geometry, (3) electrode contact, (4) degree of saturation of specimens, and (5) measurement temperature [61–64]. In this research, using low-current and low-voltage AC measurement with 1,000 Hz frequency and applying the required geometry factor (A/l in Eq. 1) maintained measurement consistency and helped avoid the problems associated with polarization, reactance, and microstructure change. Copper mesh electrodes were embedded in fresh concrete and vibration was applied in order to ensure good bond between electrode and cementitious matrix; presence of water in fully saturated specimens ensured full electrical contact between electrodes and concrete matrix. For a concrete specimen, higher degree of saturation and higher temperature lead to lower resistivity [63,64]. On the other hand, electrolytic conduction (that is an important electricity conduction mechanism in concrete) occurs through capillary water that is present in >40% degree of saturation [63,64]. Hence, in order to guarantee the consistency of measurements, involvement of electrolytic conduction, and elimination of surface moisture effect all specimens were tested in saturated surface-dry condition and 23°C ambient temperature. Electrical resistance was measured by an LCR meter (BK Precision® 875B).



(a) (b)

Figure 1. Electrical resistivity measurement specimen: (a) schematic of cubic ECON specimens with embedded electrodes, (b) measurement set-up.

2.4 Data analysis

Variable estimates were derived by standard least square regression analysis of the measured responses. This method applies a separate two-way analysis of variance (2-way ANOVA) on each response to generate a model for a particular response. The significance of each variable's effect on each response is then indicated by the p-value (i.e. probability) parameter. The confidence interval (1-α) was selected as 0.95. A variable was considered to be significant if its p-value was smaller than α; therefore, the effects corresponding to a p-value smaller than 0.05

would be significant. The smaller the p-value the higher the significance level. The estimates for a variable refer to the coefficients of the model built up by least square analysis [52]. The model feature of the software was used to simulate the effect of individual variables on the responses as well as the coupled variable interactions. For a 2^k experiment design, a regression model is more natural and intuitive [53], therefore, in this research a regression model was used for analyzing the results as given in equation 2 [53]:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum\sum_{i<j} \beta_{ij} x_i x_j + \epsilon \qquad \text{(Eq. 2)}$$

Where, y is the response, x_i and x_j are the coded variables, k is number of variables, β's are regression coefficients, and ϵ is random error. The third term on the right side of the equation shows the interaction between variables x_i and x_j; significant interactions were included in the model.

3 Results and discussion

The test results used to derive the response prediction models are presented in Table 4. Considering the number of batches and prepared specimens explained in section 2.3, each value in the table is the average of nine measurements. Table 5 shows the regression coefficients for each response and the predicted values are given in Table 6.

297 **Table 4. Averaged measurement results for each mix design.**

Age (days)		3		7		28		28		28	
Mix design No.	**HRWR (% wt. cem.)**	**Resistivity (Ω-cm)**	**SD**	**Resistivity (Ω-cm)**	**SD**	**Resistivity (Ω-cm)**	**SD**	**Compressive strength (MPa)**	**SD**	**Flexural strength (MPa)**	**SD**
1	1.0	2,543	125	3,370	130	4,313	326	55	1.6	6.5	2.2
2	1.0	2,263	265	2,953	318	3,730	361	59	2.6	5.5	0.1
3	1.0	3,193	181	4,023	248	5,293	320	44	1.4	7.0	0.4
4	1.0	4,197	536	4,873	560	5,890	541	46	0.7	6.0	0.6
5	1.0	2,583	258	2,837	104	3,177	281	50	1.6	8.0	1.5
6	1.0	2,963	187	3,560	192	3,943	264	65	3.0	8.5	0.5
7	1.0	2,457	35	2,670	44	2,910	167	52	1.4	6.0	0.8
8	3.0	897	38	1,087	55	1,163	58	37	0.4	6.5	0.5
9	3.0	988	170	1,107	237	1,563	313	22	0.1	5.5	0.9
10	3.0	2,673	58	3,567	104	4,093	129	45	3.1	7.0	0.1
11	2.0	1,350	30	1,520	30	1,753	29	53	1.6	7.5	0.2
12	1.0	3,953	59	4,593	60	5,373	32	52	2.3	6.0	0.4
13	2.5	1,997	95	2,273	127	2,643	129	39	4.0	7.0	0.7
14	3.0	600	66	647	68	800	87	20	2.7	6.5	0.4
15	1.0	2,697	35	2,727	21	3,297	42	66	0.5	6.0	0.5
16	1.0	2,080	80	2,323	32	2,750	55	53	2.9	7.0	1.1
17	2.5	1,950	141	2,337	180	2,653	176	31	5.2	6.5	0.3
18	2.5	1,463	45	1,737	67	1,983	32	60	1.2	9.0	0.8
19	3.0	780	56	870	62	1,110	89	24	4.5	5.5	0.5

298 Note: HRWR=high range water reducing admixture, SD=standard deviation

299 **Table 5. Regression coefficients of the model for the three responses.**

Parameters	Responses		
	Electrical resistivity	Compressive strength	Modulus of rupture
	Regression coefficients		
Intercept (β_0)	3055.2	45.8	6.74
Fiber content	-1391.5	-9.1	0.07
Fiber length	130.6	0.7	-0.06
C/F	339.8	0.3	0.49
CEA dosage	-252.3	6.8	0.19
FDA dosage	21.5	4.1	0.32
Fiber content × CEA dosage	418.5	-0.5	0.14
Fiber content × Fiber length	269.0	0.7	0.06
C/F × Fiber length	259.4	-2.0	-0.45
Fiber content × FDA dosage	155.6	7.4	0.47
C/F × FDA dosage	96.8	0.3	-0.10
CEA dosage × FDA dosage	75.6	1.1	-0.04
Fiber length × FDA dosage	-96.5	-0.9	0.17

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Table 6. Comparison of measured and predicted responses.

Mix design No.	Electrical resistivity (Ω-cm)			Compressive strength (MPa)			Modulus of rupture (MPa)		
	Measured	Predicted	Residual	Measured	Predicted	Residual	Measured	Predicted	Residual
1	4,313	4,269	45	55.3	55.9	-0.5	6.6	6.4	0.2
2	3,730	3,775	-45	58.8	58.2	0.5	5.4	5.6	-0.2
3	5,293	5,320	-26	43.9	46.7	-2.7	6.9	7.3	-0.4
4	5,890	5,878	12	46.5	48.9	-2.5	6.2	6.5	-0.2
5	2,970	2,946	24	50.7	51.5	-0.8	6.6	7.0	-0.4
6	3,943	3,860	84	65.1	65.4	-0.3	8.6	8.2	0.4
7	3,060	2,946	114	50.9	51.5	-0.6	7.2	7.0	0.2
8	1,163	1,119	45	37.2	37.7	-0.5	6.5	6.3	0.2
9	1,563	1,480	84	22.2	22.4	-0.3	5.6	5.2	0.4
10	4,093	4,067	26	44.5	41.8	2.7	6.9	6.5	0.4
11	1,753	1,780	-26	52.8	55.6	-2.7	7.6	8.0	-0.4
12	5,373	5,386	-12	51.7	49.2	2.5	6.2	6.0	0.2
13	2,643	2,688	-45	38.6	38.1	0.5	7.1	7.3	-0.2
14	800	884	-84	20.0	19.7	0.3	6.5	6.9	-0.4
15	3,297	3,380	-84	65.6	65.4	0.3	6.0	6.4	-0.4
16	2,807	2,946	-139	53.0	51.5	1.4	7.1	7.0	0.1
17	2,653	2,666	-12	31.0	28.5	2.5	6.5	6.2	0.2
18	1,983	1,957	26	60.4	57.7	2.7	8.9	8.6	0.4
19	1,110	1,098	12	23.5	26.0	-2.5	5.3	5.5	-0.2

3.1 Effect of variables on ECON electrical resistivity

Table 7 presents p-values and the respective values of standard error (SE) for the main and coupled effects of variables on electrical resistivity of ECON. Concerning only main effects, for the selected confidence interval ($1-\alpha = 0.95$) four variables (fiber content, fiber length, C/F, and CEA dosage) were found to be significant at all ages with the p-values smaller than 0.05. FDA dosage was significant only at the 3-day age. This indicated that variable are likely age-dependent. Variation of FDA effectiveness with cement hydration time can be attributed to the effect of porosity at early ages when fibers are clustered/flocculated in absence of FDA. Nevertheless, as the results showed, the influence of FDA on conductivity was dwarfed by evolution of cement hydration. This is also in agreement with the findings of previous studies of the effect of porosity and hydration on electrical resistivity of normal concrete [37–39,61]. Therefore, 28-day measurement results would be more reliable than earlier ages because cement has undergone most of its hydration by this age. Considering 28-day results analysis, fiber content and C/F ratio having infinitesimally small p-values were the most significant variables followed by CEA dosage and fiber length.

Although the main effects of variables can reveal the importance of individual variables, the analyses results should also account for any existing interactions between variables. As shown in Table 7, some interactions between the studied variables were found to be significant at 28 days with p-values smaller than 0.05. These are the fiber content × CEA dosage coupled effect, the fiber content × fiber length coupled effect, the C/F × fiber length coupled effect, and the fiber content × FDA dosage coupled effect.

Table 7. Effect of the variables on electrical resistivity

Variables	Age (days)					
	3		7		28	
	P-value	SE	P-value	SE	P-value	SE
Fiber content	0.00E+00	2.77	0.00E+00	3.07	0.00E+00	3.27
Fiber length	5.35E-07	2.57	5.27E-05	2.85	3.04E-06	3.27
C/F	4.00E-09	2.77	0.00E+00	3.07	0.00E+00	2.70
CEA dosage	1.51E-06	2.50	1.58E-07	3.07	1.20E-09	3.27
FDA dosage	3.03E-04	2.77	7.11E-01	3.00	5.16E-01	2.60
Fiber content × CEA dosage	0.00E+00	2.77	0.00E+00	3.07	0.00E+00	3.27
Fiber content × Fiber length	1.42E-07	2.77	1.19E-08	3.07	2.00E-10	3.27
C/F × Fiber length	4.52E-07	2.77	1.32E-08	3.00	6.00E-10	3.20
Fiber content × FDA dosage	0.00E+00	2.70	1.11E-08	2.80	2.25E-05	3.27
C/F × FDA dosage	1.11E-01	2.77	7.51E-01	3.07	5.89E-02	3.27
CEA dosage × FDA dosage	6.41E-01	2.77	4.72E-01	3.07	1.66E-01	3.27
Fiber length × FDA dosage	8.25E-02	2.77	3.72E-01	3.07	5.52E-02	3.27

Note: × sign shows interaction between two variables.

3.2 ECON electrical resistivity response prediction

Figure 2 shows the predicted-versus-measured electrical resistivity values at 28-day age. The standard error of estimate (SEE) for prediction of 28-day electrical resistivity was 4.7 (Ω-cm) which is 0.15% of the mean measured resistivity. The SEE and coefficient of determination (R^2) showed a strong correlation between the predicted and measured resistivity values (Figure 2).

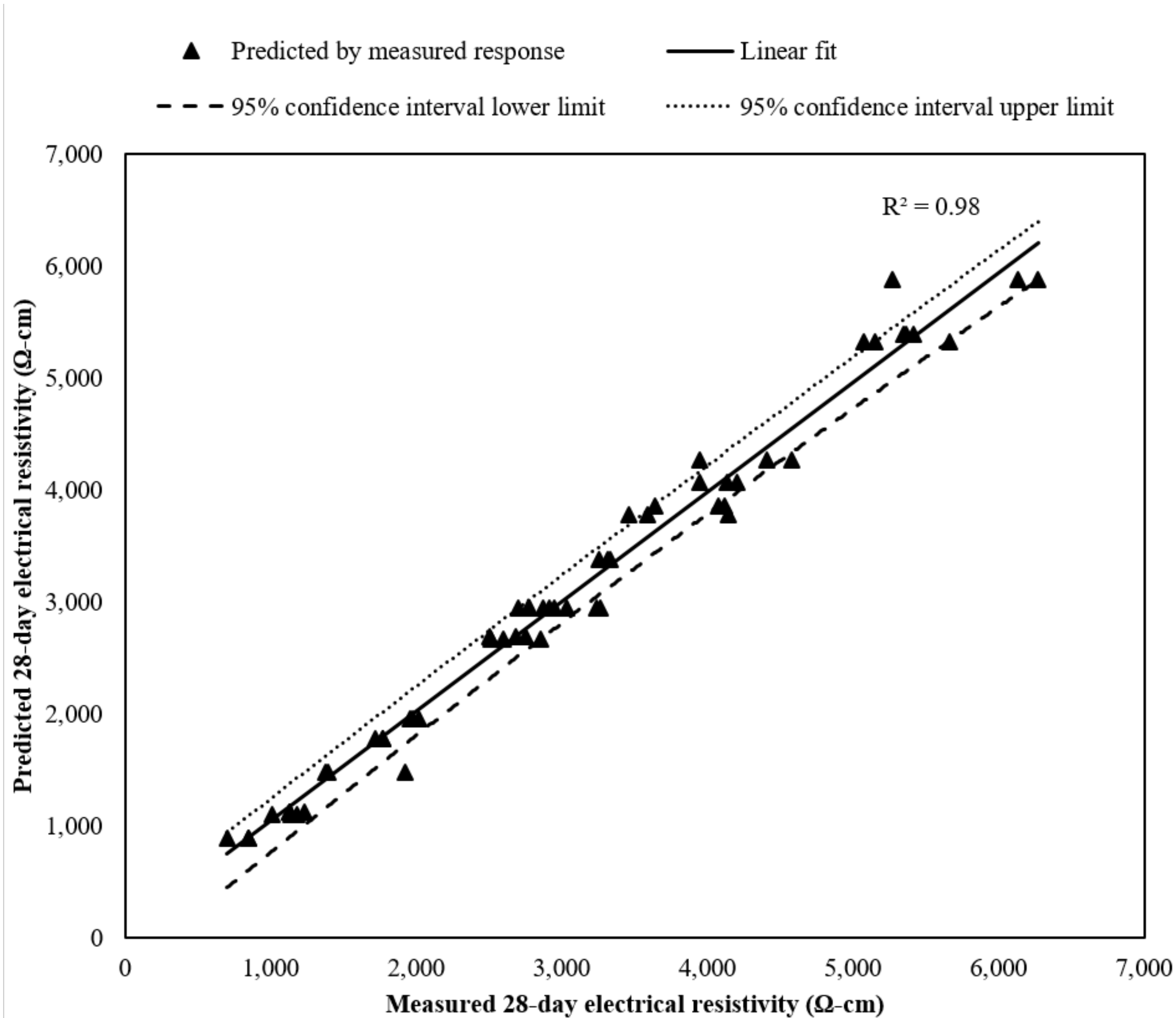

Figure 2. Predicted-versus-measured values of electrical resistivity at 28-day age.

3.3 Sensitivity assessment of variables for ECON electrical resistivity response

Figure 3 shows the prediction profiler for the variation of resistivity with each variable at 28 days. Resistivity decreased with increasing fiber content, fiber length, CEA dosage, and FDA dosage, whereas, higher C/F led to increased resistivity. Electrical resistivity exhibited a decreasing trend by the increase of fiber dosage; however, the rate of resistivity drop by fiber addition was reduced at higher fiber dosages. This is in agreement with the findings of Baeza et al. [39] who showed that the electrical resistivity decreasing rate significantly reduced beyond fiber dosage of 0.5% (Vol.). It was also suggested by Baeza et al. [39] that higher coarse-to-fine aggregate ratio gives higher electrical resistivity. This effect was also observed and verified in this research as shown in Figure 3, where, increasing the C/F resulted in the higher electrical resistivity. As Figure 3 reveals, the CEA showed a significant reducing effect on electrical resistivity, however, this effect was dependent on synergic influence of fiber content and CEA dosage as will be discussed later on.

Indicated by the slope of resistivity-FDA dosage line in Figure 3, the effect of FDA dosage was negligible that is in agreement with the calculated p-value (5.16E-01 in Table 7). Several studies have shown the effectiveness of methylcellulose in improving carbon fiber dispersion [28,47,65,66] and electrical conductivity [65,66] of cementitious composites using mortar or cement paste samples. Methyl cellulose was used for improving carbon fiber dispersion in concrete samples [12], however, the degree of effectiveness was not reported. Using cement paste and mortar test results, it was postulated that methyl cellulose provides desirable effectiveness particularly when used in combination with silica fume [28,36]. In this study, methylcellulose was used as the only fiber-dispersive material; hence, its low effect on improvement of electrical conductivity comes in agreement with the findings of previous studies. Nevertheless, the effectiveness level of the material in concrete still needs to be more deeply investigated.

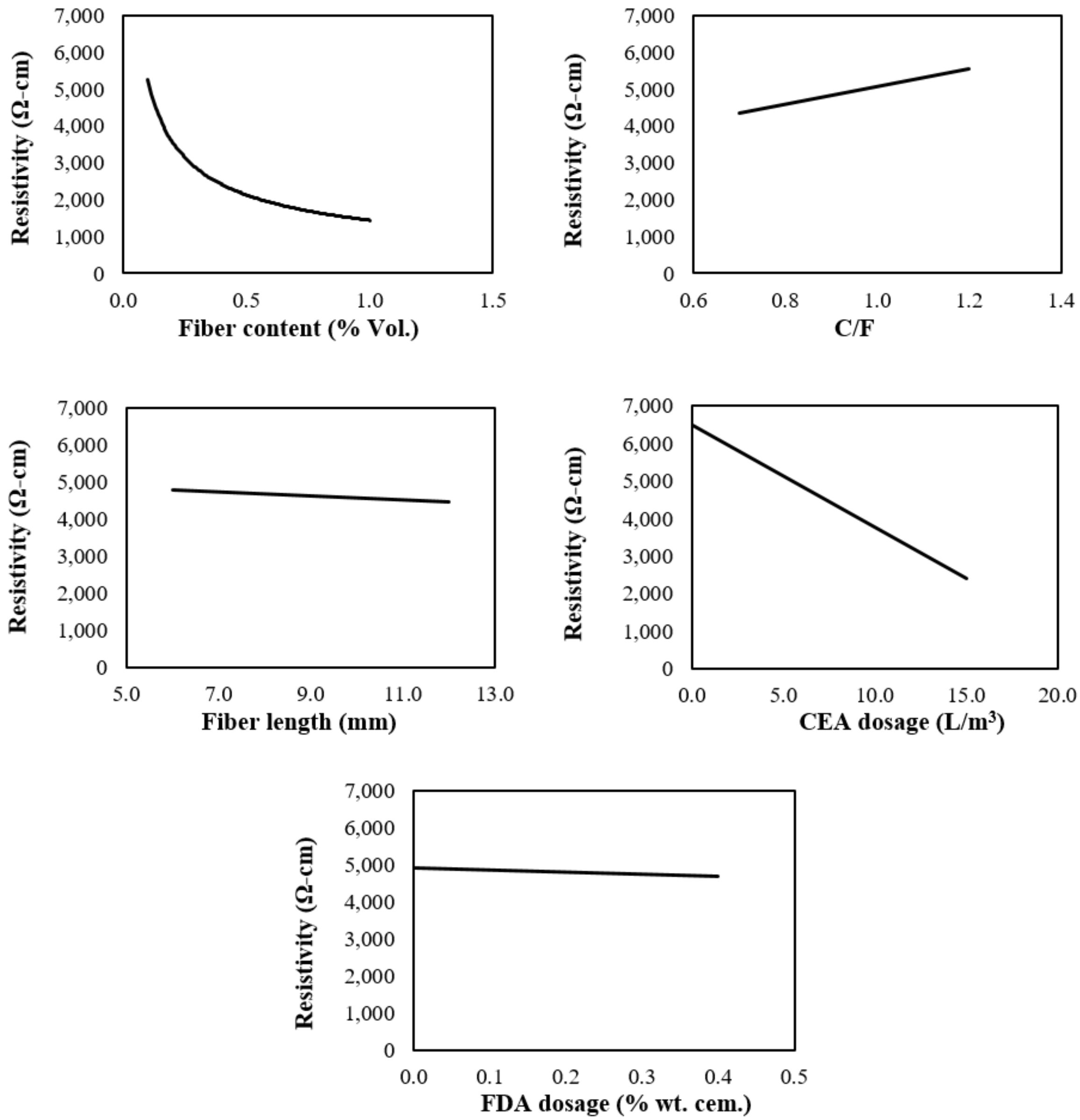

Figure 3. Sensitivity assessment of individual variables for electrical resistivity response.

Figure 4 shows all significant interactions for electrical resistivity response. According to Figure 4(a), electrical resistivity decreased with increasing fiber content regardless of whether or not FDA was applied. However, above a certain fiber content (0.55%) the FDA-modified mixes gave higher predictions for electrical resistivity. Figure 4(b) reveals that CEA was more effective in low fiber contents. Up to 0.87% fiber content the CEA-modified mixes showed lower electrical resistivity, however, as fiber content increased the difference between the CEA-modified and non-modified mixes became smaller. Above 0.87% fiber content the predicted resistivity values for CEA-modified mixes exceeded those of non-modified ECON. Figure 4(c) reveals that with both fiber size classes electrical resistivity decreased by fiber content while the resistivity of ECON was less sensitive to variation of fiber content in the case of 6-mm fiber. Regarding the coupled effect of fiber length and C/F (Figure 4(d)), with 6-mm fiber electrical resistivity dramatically increased by increasing C/F, whereas, it was negligibly increased in the case of 12-mm fiber.

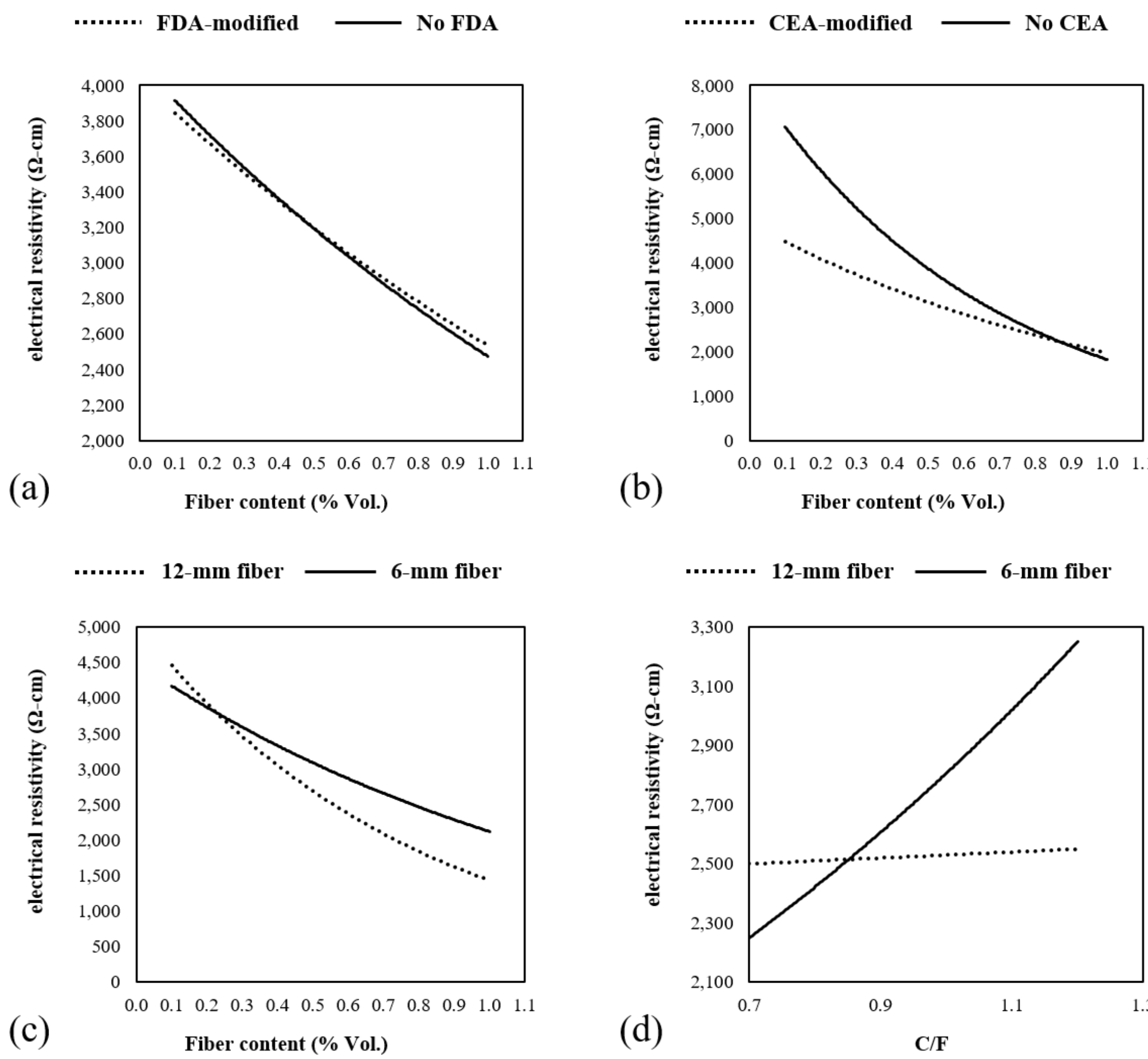

Figure 4. Sensitivity assessment of significant variable interactions on electrical resistivity response: (a) FDA dosage-fiber content coupled effect, (b) CEA dosage-fiber content coupled effect, (c) fiber length-fiber content coupled effect, and (d) fiber length-C/F coupled effect.

3.4 Effect of variables on ECON strength properties

Variable effects on strength properties are shown in Table 8. Regarding the p-values of main effects, three variables of fiber content, CEA dosage, and FDA dosage were significant with respect to compressive strength, while, only C/F ratio and FDA dosage were significant variables influencing flexural strength. The interactions between the studied variables identified to be significant are the C/F× fiber length coupled effect on compressive strength, the C/F × fiber length coupled effect on flexural strength, the fiber content × FDA dosage coupled effect on compressive strength, and the fiber content × FDA dosage on flexural strength.

Table 8. Effect of the variables on compressive and flexural strengths.

Variables	Response			
	Compressive strength		Flexural strength	
	P-value	SE	P-value	SE
Fiber content	0.00E+00	0.45	5.79E-01	0.12
Fiber length	5.82E-01	0.45	3.35E-01	0.12
C/F	5.41E-01	0.41	2.79E-04	0.13
CEA dosage	0.00E+00	0.45	1.36E-01	0.12
FDA dosage	0.00E+00	0.46	1.18E-02	0.18
Fiber content × CEA dosage	2.56E-01	0.45	2.65E-01	0.12
Fiber content × Fiber length	1.36E-01	0.41	6.43E-01	0.13
C/F × Fiber length	7.28E-05	0.45	6.04E-04	0.12
Fiber content × FDA dosage	0.00E+00	0.45	3.98E-04	0.12
C/F × FDA dosage	4.53E-01	0.43	4.01E-01	0.12
CEA dosage × FDA dosage	1.17E-01	0.45	7.48E-01	0.13
Fiber length × FDA dosage	6.28E-02	0.46	1.76E-01	0.11

3.5 ECON strength response prediction

The predicted versus measured response plots for compressive strength and flexural strength are respectively given in figures 5 and 6. Compressive strength predictions gave good convergence for predicted and measured responses with a SEE of 2.7 (6% of measured data mean) and coefficient of determination of 0.96. The accuracy of flexural strength predictions with a SEE of o.73 (i.e. 11% of measured data mean) and coefficient of determination of 0.58 was not as good as compressive strength predictions.

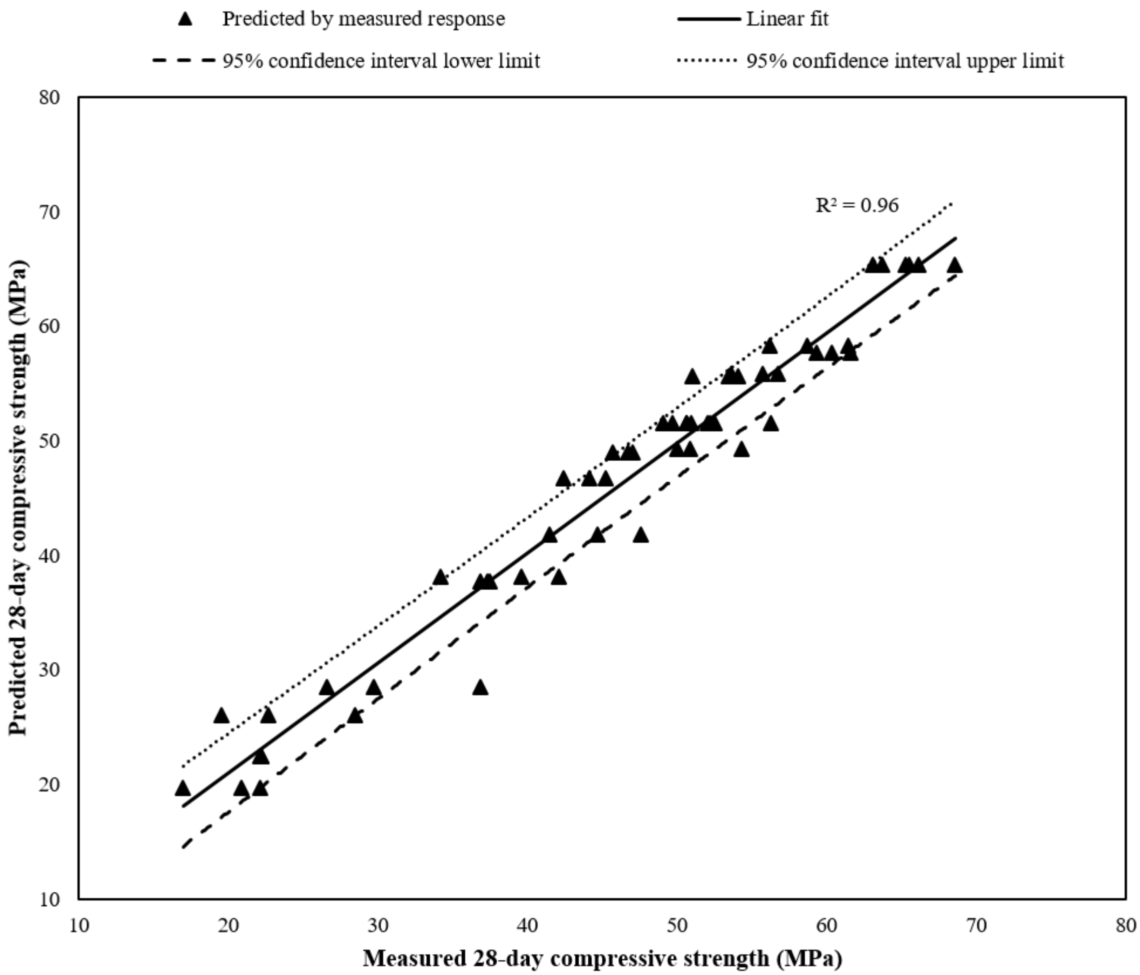

Figure 5. Predicted-versus-measured values of compressive strength.

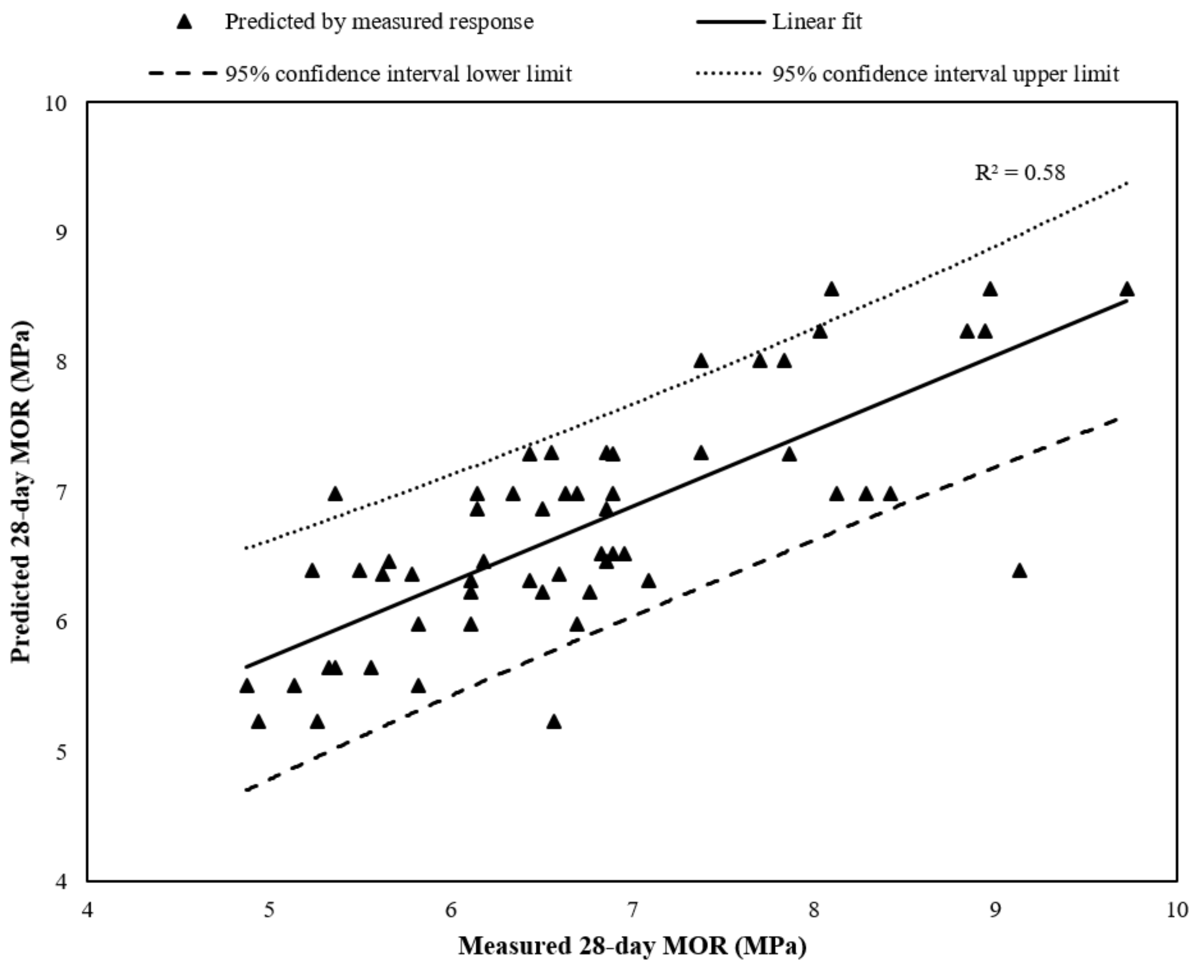

Figure 6. Predicted-versus-measured values of flexural strength.

3.6 Sensitivity assessment of variables for ECON strength property responses

Figures 7 and 8 respectively present the prediction profilers for compressive and flexural strengths with respect to each variable. As revealed in Figure 7, fibers positively affected compressive strength only to a certain fiber content (0.55%). Unlike compressive strength, flexural strength maintained a constantly increasing trend with increasing fiber content; however, this effect is tied to the coupled effect of variables and needs to be discussed with regard to interaction of fiber content and FDA dosage.

Both compressive and flexural strengths were increased with increasing CEA dosage or using longer fibers. Positive effect of fiber length on compressive strength is in contrast to the findings of Chen and Chung [54] who showed that in mortar samples, compressive strength decreases by fiber length while flexural strength is improved. However, the difference of the behavior of mortar and concrete samples makes it difficult to compare these two conclusions. Increasing fiber dosage up to 0.55% (Vol.) improved compressive strength, while, beyond 0.55% the compressive strength deceased by increasing the fiber dosage. This can be attributed to the higher void content of concrete when fiber dosage passes a certain threshold [67].

Higher C/F or higher FDA dosage resulted in lower compressive and flexural strengths. Xu and Chung [36] postulated that application of methylcellulose up to 0.8% (wt. cem.) improved tensile strength and decreased compressive strength of cement pastes. Also, carbon fiber in dosages up to 4% (Vol.) was used with methylcellulose as dispersive agent to improve flexural strength of carbon fiber-reinforced cement pastes [65]; however, the effect was not compared with fiber-reinforced samples without methylcellulose. The reduction of compressive strength by methylcellulose found in this research is in agreement with the findings of the previous studies, while, the reduction of flexural strength in carbon fiber-reinforced concrete samples has not been reported in the existing literature. On the contrary, Chen and Chung [54] reported slight improvement of flexural strength of carbon fiber-reinforced concrete by application of methyl cellulose; however, they used very low fiber dosage (0.2% Vol.). Note that the effects of fiber length and C/F variables on compressive strength as well as the effects of fiber content, fiber length, and CEA dosage on flexural strength were non-significant.

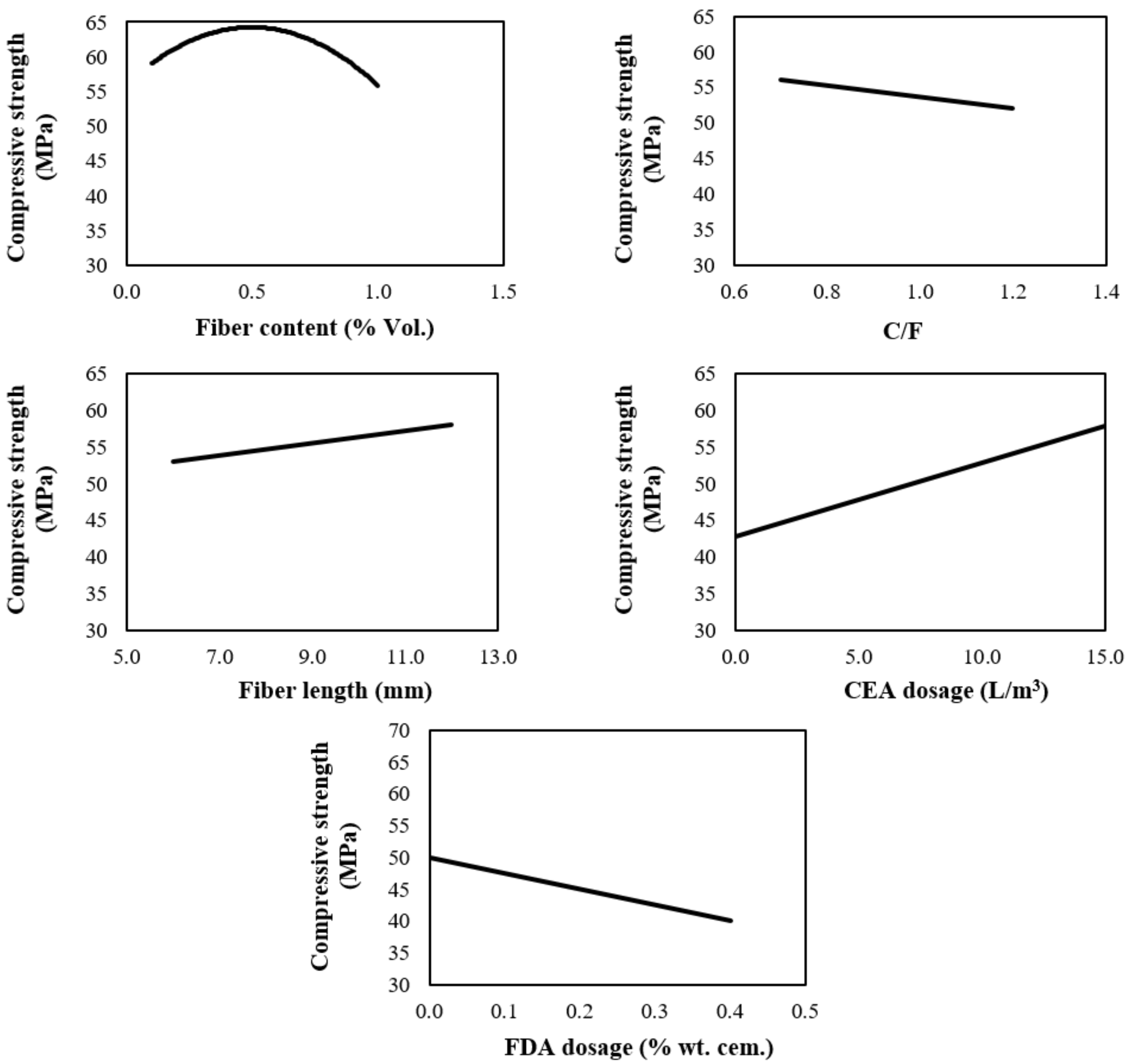

Figure 7. Sensitivity assessment of individual variables on compressive strength responses.

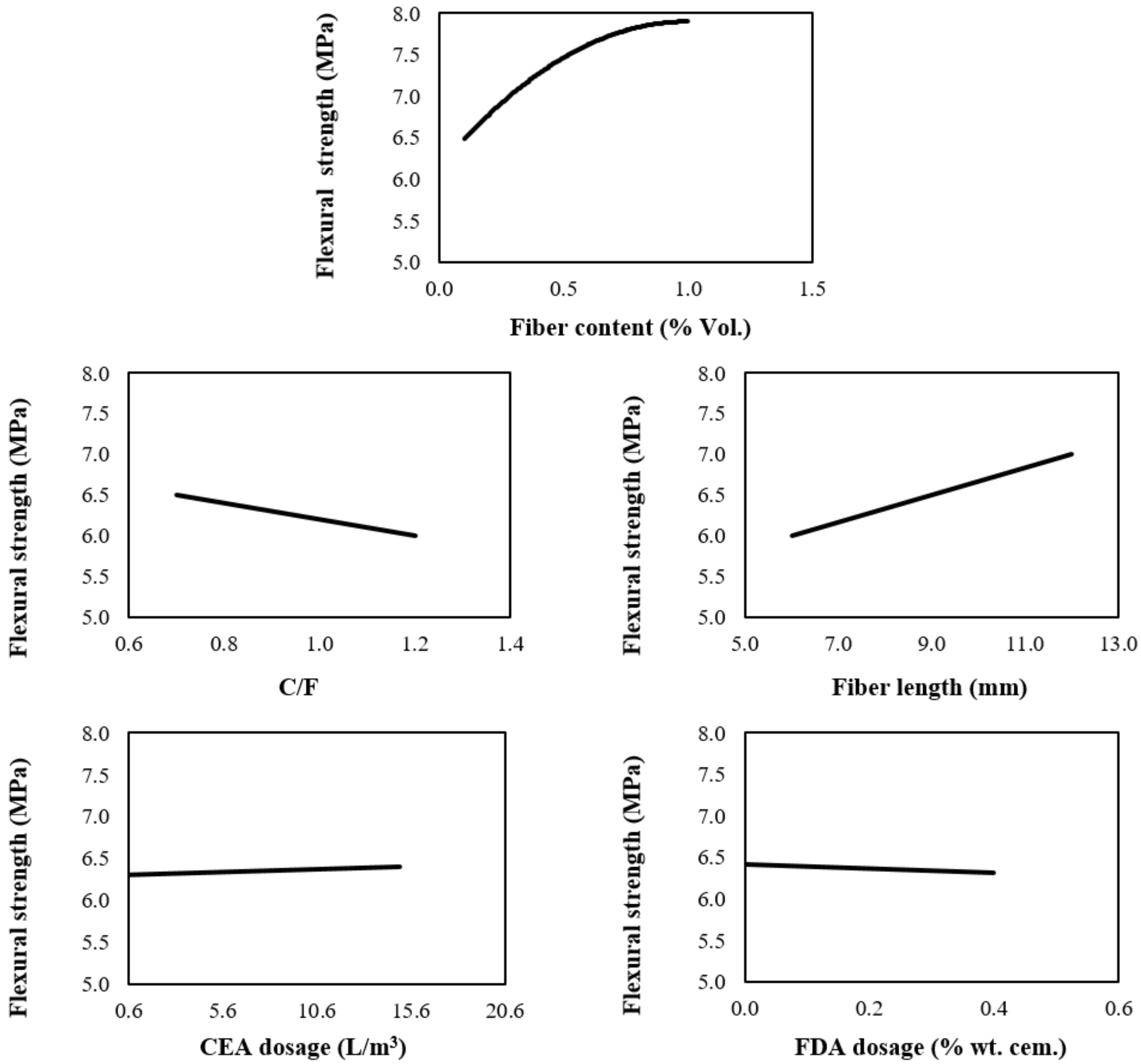

Figure 8. Sensitivity assessment of individual variables on flexural strength.

Figure 9 shows all significant interactions for strength property responses. Figure 9(a) and (b) show the interaction of FDA and fiber content in the case of compressive and flexural strengths respectively. In absence of FDA, compressive strength followed a decreasing trend with increasing fiber content. The variation of compressive strength with fiber content was not steady in presence of FDA. Up to 0.55% fiber content, FDA application led to increased compressive strength. Beyond this percentage, compressive strength experienced a falling trend with increase of fiber content. Without FDA addition, increasing fiber content resulted in lower flexural strength. However, when FDA was present, the flexural strength increased with increasing fiber content. Carbon fiber-reinforced concrete is expected to exhibit better flexural behavior than normal PCC [68]. The results of this research do not contradict this statement; rather, it shows that in carbon fiber-reinforced concrete in absence of FDA increasing the amount of fiber led to flexural strength reduction. On the contrary, when FDA was used to enhance the dispersion of fibers, a higher efficiency was observed in improving flexural behavior. Based on this observation, higher fiber content leads to higher flexural strength.

As seen in Figure 9(c), the effect of C/F on compressive strength was different between the ECONs containing 6-mm and 12-mm fibers. Simulations showed that when the ECON was made with 6-mm fibers, compressive strength decreased with increasing C/F, whereas, it followed an opposite trend with 12-mm fiber. When 6-mm fiber was used, flexural strength slightly decreased by increasing C/F; on the contrary, higher C/F resulted in considerably higher flexural strength upon application of 12-mm fiber (Figure 9(d)).

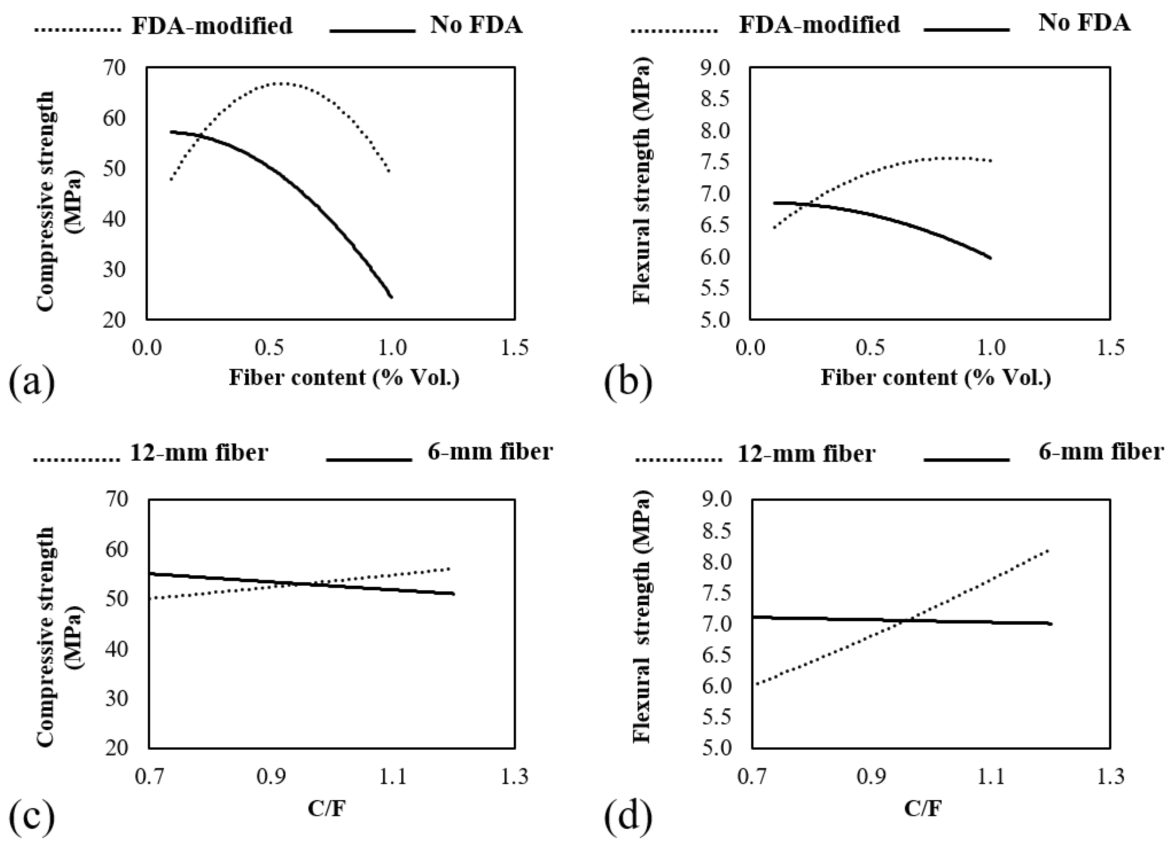

Figure 9. Sensitivity assessment of significant variable interactions on strength responses: (a) FDA dosage-fiber content coupled effect on compressive strength, (b) FDA dosage-fiber content coupled effect on flexural strength, (c) fiber length-C/F coupled effect on compressive strength, and (d) fiber length-C/F coupled effect on flexural strength.

3.7 Discussion

The foregoing results suggest that use of fiber in a volume fraction that lies within the 0.4 to 0.8% (Vol.) range was desirable with respect to the electrical resistivity and strength properties of ECON. This is in agreement with the results of previous studies that reported percolation threshold of carbon fibers with fiber dosages of 0.55-0.8 Vol. % in cement paste [38,69], 0.4-1 Vol% in mortar [22,67], and between 0.5 and 0.6 Vol.% in concrete[39]. It is not recommended to consider carbon fiber contents higher than the amounts required for achieving the intended performance. It was revealed that longer fiber resulted in lower resistivity and higher strength values; however, the effects of fiber length on the reponses were either moderate or non-significant.

Using 6-mm fiber rendered the mixture very sensitive to variation of C/F with electrical resistivity dramatically increased with increasing C/F. Furthermore, mixtures with low (0.7) or moderate (0.95) C/F values exhibited considerably better workability and cohesiveness. Hence, practical considerations and C/F effect on all responses suggest reducing C/F as much as allowed by the relevant specifications and/or application-induced requirements.

The effect of FDA on electrical resistivity was found to be non-significant. However, when fiber content is in the percolation threshold range (as reported in the literature), minor dosages of FDA can result in improvement of both compressive and flexural strengths. CEA exerted a significant reducing effect on electrical resistivity as long as fiber content did not exceed percolation threshold. Moreover, CEA helped improve compressive and flexural strengths.

4 Conclusions

The effects of five variables on the electrical resistivity and mechanical properties of electrically conductive concrete (ECON) were investigated. The responses were electrical resistivity, compressive strength, and flexural strength. Statistical analysis provided the significance levels of each variable and/or interaction of variables in terms of p-value and prediction profiler provided the trend of responses with change in each variable. The results can be summarized in the following statements:

- Calcium nitrite-based corrosion inhibitor admixture was successfully used as conductivity-enhancing agent (CEA) for improving the electrical conductivity of ECON samples. However, the prediction profiler showed that CEA was effective in conductivity improvement up to fiber dosage of 0.87% (Vol.). CEA improved electrical conductivity and compressive strength, especially at low fiber contents. Although CEA improved flexural strength, its effect was not significant.
- Significance of variables varied by hydration time (age) becoming almost steady after 7 days. It is recommended to use 28-day or later age measurement results for analyses.
- Four significant variables affecting electrical resistivity -arranged from most to least significant- were fiber content, coarse-to-fine aggregate volume ratio (C/F), fiber length, and conductivity-enhancing agent (CEA) dosage.
- Compressive strength was significantly influenced by fiber content, CEA dosage, and fiber-dispersive agent (FDA) dosage.
- Two variables showed significant effect on flexural strength, namely, C/F and FDA dosage.
- Interactions between different variables resulted in coupled effects on the responses.
- In addition to main effects and coupled effects of variables, the ECON mix design should account for practical and implementation considerations.
- Although using 12-mm fiber exerted positive effect on the responses, the effect was moderate in case of electrical resistivity and non-significant on strength properties. Therefore, regarding mixing and implementation considerations it is recommended to use 6-mm fiber in production of ECON.

Future studies can use the results of this research to develop an experimental plan for investigating the discussed effects with more levels of the significant variables. A full factorial DOE with the significant variables can be used to obtain predictions that are more accurate. A variety of admixtures and electrically conductive materials still need to be evaluated for improvement of electrical conductivity of ECON. Developing three-phase electrically conductive composites [12] is a viable method that can be integrated with the methods proposed in this study to expand the functionality of ECON beyond the levels so far achieved.

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