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Mechanical properties of mortars with EarthZyme additive

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Research Paper

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Mechanical properties of mortars with EarthZyme additive

Concrete is the most widely used building material in the world. However, owing to the high CO_2 emissions from the production of cement, its use has been questioned, and attempts have been made to improve it. Various chemical additives are being used to improve concrete properties. Enzymes are organic materials and have been especially favoured in recent years owing to their low costs when used in traditional soil stabilisation methods. This study used the 'EarthZyme' enzyme as a plaster mortar additive and investigated its effects on the mechanical properties of mortars. EarthZyme completely biodegrades in nature and is used for soil stabilisation. By producing mortar specimens with additive enzyme ratios of 0 %, 0.01 %, 0.02 %, 0.03 %, and 0.04 %, the effects of the enzyme additions on the mechanical properties of the mortars (ultrasonic pulse velocity UPV, flexural strength f_f and compressive strength f_c) were determined. The flow table values of the mortar specimens in the flow table test varied within the range of 15–17 cm. According to the results, the flow table values of the mortars increase with an increasing addition rate of EarthZyme. Although the additive ratio of the EarthZyme does not significantly affect the mechanical properties of mortars at early ages (3 days), the enzyme addition ratio reduces the UPV and fc while improving ff at late ages (28 days). The enzyme addition ratio has no significant effect on the UPV and f_f but has a significant effect on f_c.

Key words:

EarthZyme, mortar, ultrasonic pulse velocity, compressive strength, flexural strength, ANOVA

Prethodno priopćenje

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Mehanička svojstva morta s dodatkom aditiva EarthZyme

Beton je najzastupljeniji građevni materijal na svijetu. Međutim, zbog visoke razine emisije CO_2 iz proizvodnje cementa, njegova se upotreba dovodi u pitanje, te ga se pokušava poboljšati. Za poboljšanje svojstava betona primjenjuju se različiti kemijski aditivi. Enzimi su organski materijali i posebno su favorizirani posljednjih godina zbog svoje niske cijene pri upotrebi u tradicionalnim metodama stabilizacije tla. U ovom se istraživanju primijenio enzim 'EarthZyme' kao aditiv mortu za žbuku te su se ispitali njegovi učinci na mehanička svojstva mortova. EarthZyme je u potpunosti biorazgradiv u prirodi i upotrebljava se za stabilizaciju tla. Izradom uzoraka morta s udjelima enzima od 0 %, 0,01 %, 0,02 %, 0,03 % i 0,04 %, utvrđeni su učinci dodatka enzima na mehanička svojstva morta (brzina ultrazvučnog impulsa (UPV), čvrstoća na savijanje f_f i tlačna čvrstoća f_c). Vrijednosti uzoraka morta dobivene pomoću ispitivanja rasprostiranjem varirale su u rasponu od 15 do 17 cm, pri čemu vrijednosti rastu s povećanjem udjela EarthZymea. Iako udio EarthZymea ne utječe značajno na mehanička svojstva uzoraka morta u ranoj starosti (3 dana), udio enzima smanjuje UPV i f_c, a u isto vrijeme poboljšava ff kod starijih uzoraka (28 dana). Udio enzima nema značajan učinak na UPV i f_f, ali ima značajan učinak na f_c.

Ključne riječi:

EarthZyme, mort, brzina ultrazvučnog impulsa, tlačna čvrstoća, čvrstoća na savijanje, ANOVA

1. Introduction

Concrete is the most widely used building material in Türkiye and the world owing to the easy access to its components, generality of its manufacturing technologies, resistance to fire and environmental impacts, relatively low cost, ability to be produced in a desired form with easy technology and so on [1–3]. However, the large amounts of CO_2 gas released into the atmosphere during concrete production, the use of enormous amounts of energy in such production and creation of environmental pollution have led to perspectives questioning the use of concrete materials. The cement industry is estimated to be responsible for 6–7 % of the total CO_2 released into the atmosphere [4, 5]. The first step in achieving a sustainable, healthy environment for life and preventing adverse developments (such as increased global warming owing to increases in greenhouse gas) is the selection and development of building materials in line with the possibilities provided by construction technology [6]. The cement and binder types used in cement composites are being re-examined owing to environmental concerns. Research is being conducted on the materials and additives used as concrete components; in this context, efforts are being made to improve concrete properties and produce more environmentally friendly concrete by adding new materials to concrete compositions [4, 5]. Studies are continuously being conducted to produce sustainable and less environmentally damaging concrete types with improved durability, processability, etc., by using different additive materials [7–14].

Improving the mechanical properties of these increasingly used cement composites to meet emerging housing needs owing to population growth, reducing energy consumption and producing them in a manner less damaging to the environment are enormously important issues for both the present and future.

EarthZyme is an enzyme often used to increase soil stabilisation and reduce the dust on roads made of earth. The use of EarthZyme to improve the properties of mortars was the main subject of this study and more generally, the additives used in concrete and mortar (other than the basic components) and their effect levels on concrete and mortar properties. Accordingly, this study examined the effects of using the EarthZyme material in the production of mortars on the mechanical properties of such mortars.

2. EarthZyme

Enzymes are biological catalysts found in all living organisms. They are organic materials and are generally supplied as concentrated fluids. They are obtained through extraction from plants and animals, including microorganisms, using proper solvents [15]. Enzymes have been favoured in recent years due to their low costs when used in traditional soil stabilisation methods. They are used to improve the properties of various superstructure layers as well as in other ground applications such as sets [16, 17]. Enzymes are assumed to work as catalysts, i.e., increasing the speed of chemical reactions without being part of any final product. They attach themselves to larger organic molecules to form a reactant intermediary. In the soil, this intermediary exchanges ions with the clay structure, shattering the clay lattice and halting water absorption [16]. EarthZyme is a non-toxic soil stabiliser used in clay soils to reduce the cost of road maintenance; it improves compression and increases strength values. During the compression process of the mixture, EarthZyme reduces the optimal water content values and increases the dry density values. In general, surfactants facilitate ionic changes by increasing the diffusion of ion solutions into the soil capillary structure [18]. EarthZyme is biodegradable as it performs its enzymatic function. In particular, 82 % of EarthZyme biodegrades within 14 days and almost 100 % biodegrades within 28 days. As such, pathways and surfaces treated with EarthZyme do not suffer chemical, ultraviolet, or any other degradation in integrity as time passes [19].

Yardımlı et al. [20] used EarthZyme and polymer-based additives to improve the water and pressure resistance of a soil material used in adobe structures. They conducted water absorption and pressure experiments to compare enzyme- and polymer-doped samples and unadulterated samples at the end of 30 days. Considering that soil must contain 5 %–30 % clay and silt for enzymes to work as additives, it was concluded that the compressive strengths of the samples with enzyme and polymer additives increased relative to those without additives. In addition, their water resistance increased. Thus, the additives positively affected the investigated properties of the adobe material.

Abdulkareem et al. [18] evaluated using EarthZyme (a liquid-based nanomaterial) as an additive to cement kiln dust to improve the ground properties. To this end, they created sandy and fine-grained soil mixtures of EarthZyme with and without additives and cement kiln dust. The effectiveness of the additives in soil improvement was investigated by conducting experiments on the prepared mixtures. The improvement process from the enzyme was found to be more effective on soil floors with high clay contents.

The Shengli open-coal mining company tested a 500 m mine path built using EarthZyme and achieved good results, resulting in the 10 km-long road built with EarthZyme in 2014; this road was later studied by Shude et al. [21]. In general, it has been found that a mine road made using EarthZyme is more resistant than an undoped road. In addition, owing to being flat, there are large reductions in the road dust, fuel consumption and tire abrasion of vehicles using the road.

Khan et al. [16] conducted a California bearing ratio strength experiment to assess the pressure strengths of doped ground (sedimentary soil) samples with three different enzyme types. Different doses of doped and undoped soil samples were prepared and cured for four months, then submerged in water for four days before the experiment. The results of experiments on the submerged samples submerged showed that the doped samples had no significant increase in pressure strength compared to the undoped samples.

Khan & Taha [15] used enzymes produced under different commercial names in three different countries to improve the

Table 1. Chemical composition and physical properties of cement

Chemical composition [%]											
CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Cl	Na_2O/K_2O	Free lime	Insoluble residue	Loss on ignition	Unidentified
62.62	19.88	5.23	3.60	0.85	3.23	0.03	0.58/0.74	1.20	0.96	2.45	0.79

Physical properties				
Specific gravity [g/cm²]	Setting time (Vicat) [min]		Soundness (Le Chatelier) [mm]	Fineness
	Initial setting	Final setting		Blaine specific surface [cm²/g]
3.16	119	170	1	3550

Table 2. Amounts of mortar component materials (for six specimens)

Enzyme addition ratio (EAR) [%]	**Cement** [g]	**Standard sand** [g]	**Water** [g]	**Enzyme** [g]
0.00	900	2700	450	0.00
0.01	900	2700	450	0.09
0.02	900	2700	450	0.18
0.03	900	2700	450	0.27
0.04	900	2700	450	0.36

soil (ground) at the Kebangsaan University of Malaysia. According to the test results from three different enzymes applied in two different doses, they found that although there were slight improvements in the compression properties and compressive strengths of the doped soils compared to the undoped soils, these improvements were insignificant.

Zidan et al. [22] studied the use of enzymes within the scope of soil improvement in road construction and investigated the resilient modulus of the enzyme-doped soil. The resilient modulus values increased by 40 % in the enzyme-doped samples compared to those with undoped soil. This result demonstrated a highly significant quality improvement in terms of soil strength.

3. Material and method

In the study, the mechanical properties of enzyme-added mortars were investigated using experiments conducted in a laboratory. The effects of the addition of the enzyme on the properties of the mortars (ultrasonic pulse velocity (UPV), flexural strength (f_f) and compressive strength (f_c)) were determined statistically from the obtained numerical data.

3.1. Material

The mortar specimens were prepared using drinkable tap water, standard reference sand (in accordance with Turkish Standard - European Norm / TS EN 196-1 [23] and CEM I 42.5 R cement. The chemical and physical properties of the cement are shown in Table 1.

Figure 1. Sample production

The enzyme used in the study was EarthZyme, which is used as a plaster mortar additive. It is a commercial product of Cypher International Ltd. (EarthZyme HS code: 3824 9099). As reported by the manufacturer, EarthZyme can cause mild irritation to the skin and eyes upon contact. It exhibits ultimate biodegradability under anaerobic conditions as defined by US Environmental Protection Agency methods (40 CFR part 796.3180) and is non-toxic and pathogen-free. As mentioned above, EarthZyme is an additive used in earth road construction. EarthZyme's manufacturer strives to produce industry-leading eco-friendly solutions for dust control and soil stabilisation [19].

3.2. Method

Within the scope of the study, the enzyme addition ratio (EAR) (by binder weight) and specimen age were determined as the effect variables and the UPV, f_f and f_c were determined as the response variables. The variation intervals for the enzyme addition ratio were selected as 0 %, 0.01 %, 0.02 %, 0.03 % and 0.04 % and those for the specimen age were 3, 7 and 28 days.

To determine the effect levels of the effect variables on the response variables, a total of 135 rectangular prism samples with dimensions of 40 x 40 x 160 mm were produced in accordance with the TS EN 196-1 standard (Figure 1) [23]. The flow table values of the mortar specimens (TS EN 1015-3/A2) [24] from the flow table test (Figure 2) varied in the range of 15–17 cm and the flow table values of the mortars tended to increase as the enzyme addition ratio increased. The amounts of the materials used in the mortar production and flow table values are shown in Tables 2 and 3, respectively.

Figure 2. Flow table test

To determine the effect levels of the main and interaction terms of the effect variables on the response variables, 15 run points were selected in the experimental design. The values for each run point were obtained by the means from the test results of nine specimens. In the selected variation intervals, the effect levels of the effect variables on the response variables were determined based on an analysis of variance (ANOVA).

The UPV values of the response variables were obtained by dividing the sample length (160 mm) by the ultrasonic pulse time (Figure 3a) as determined according to the TS EN 12504-4 [25]. The flexural strengths of the mortar specimens were determined using the equation $f_f = 1.5 \cdot F_f \cdot l/b^3$ (in the equation; b is the side length of the square section of the prism in mm, F_f is the maximum load applied to the middle of the prism in Newtons and l is the distance between the roller support in mm) according to TS EN 196-1 [23] by applying three-point loading (Figure 3b). The compressive strength was calculated based on the equation $f_c = F_c/1600$ (F_c is the maximum load in Newtons and 1600 is the area of the plates in mm^2) according to TS EN 196-1 [23] while using prism halves divided into two parts during the flexural test (Figure 3c).

4. Experimental results

As noted above, in the experimental design, a total of 135 mortar specimens were produced for 15 run points. The run points and experimental results are shown in Table 3 and the experimental design summaries for the factors and responses are shown in Table 4.

The effect levels of the main and interaction terms of the effect variables on the response variables were determined according to the ANOVA. In addition, models were obtained for the prediction of the response variables depending on the effect variables. Moreover, interaction, contour and 3D graphics of terms with high effect levels were obtained using the Design Expert Version 13 trial program [26]. The variance analysis results for the UPV, f_f and f_c are given in Table 5.

According to the variance analysis results, the F-values of the models obtained for the UPV, f_f and f_c are 29.83 (p-value < 0.0001), 20.83 (p-value = 0.0001) and 45.66 (p-value < 0.0001), respectively. The p-values of the terms in the model are less than 0.05, indicating that the model terms are significant (significantly affecting the response variable). The p-values greater than 0.10 indicate that the model terms are insignificant (not significantly affecting the response variable) [26]. In this case, although the terms B, AB, A^2 and B^2 have significant effects on the UPV, the term A has no significant effect. The terms B and B^2 have significant effects on f_f, but the terms A, AB and A^2 do not. For f_c, the terms A, B and B^2 are the significant model terms, whereas the terms AB and A^2 are not

Equations (models) were used to obtain predicted values of response variables at the selected variation intervals of each effect variable. The models for the UPV, $f_{f \text{ and }} f_c$ are given in Equations 1, 2 and 3, respectively. The fit statistic results for the response variables are shown in Table 6.

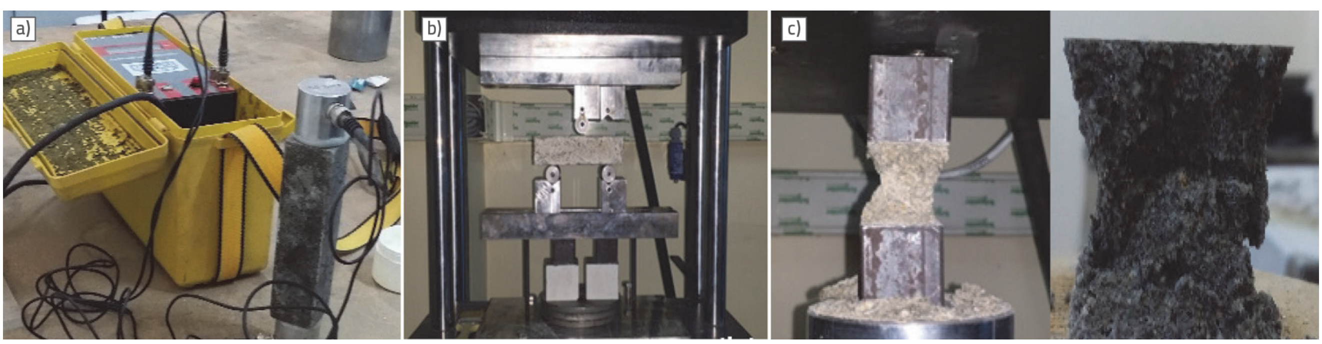

Figure 3. Experimental tests: a) UPV; b) f_f; c) f_c

Table 3. Run points

Run	Factor 1 A: EAR [%]	Factor 2 B: Specimen age [Day]	Response 1 Ultrasonic pulse velocity (UPV) [km/s]	Response 2 f_f [MPa]	Response 3 f_c [MPa]	Flow table value [cm]
1	0.00	3	4.15	5.57	29.61	15.5
2	0.00	7	4.28	6.30	34.62	15.5
3	0.00	28	4.42	7.24	42.06	15.5
4	0.01	3	4.19	5.65	31.02	15.5
5	0.01	7	4.33	7.05	37.64	15.5
6	0.01	28	4.45	7.39	40.26	15.5
7	0.02	3	4.22	5.55	30.19	16.0
8	0.02	7	4.39	6.51	35.87	16.0
9	0.02	28	4.53	6.82	39.32	16.0
10	0.03	3	4.20	5.70	29.53	16.4
11	0.03	7	4.32	6.53	34.38	16.4
12	0.03	28	4.39	7.40	39.94	16.4
13	0.04	3	4.20	5.41	30.40	16.6
14	0.04	7	4.33	6.43	33.95	16.6
15	0.04	28	4.33	7.56	38.56	16.6

Table 4. Summary of experimental design

Factor	Name	Units	Type	Min.*	Maks.*	Coded low	Coded high	Mean	SD*
A	EAR	%	Numeric	0.00	0.04	-1 ↔ 0.00	+1 ↔ 0.04	0.020	0.015
B	Specimen age	Day	Numeric	3.00	28.00	-1 ↔ 3.00	+1 ↔ 28.00	12.67	11.35

Response	Name	Units	Observations	Analysis	Min.*	Maks.*	Mean	SD*	Ratio	Transform	Model
R1	UPV	km/s	15	Polynomial	4.11	4.53	4.32	0.108	1.09	None	Quadratic
R2	f_f	MPa	15	Polynomial	5.41	7.56	6.47	0.758	1.40	None	Quadratic
R3	f_c	MPa	15	Polynomial	29.53	42.06	35.16	4.320	1.42	None	Quadratic

Min.* - minimum, Maks.* - maximum, SD* - standard deviation

Table 5. Analysis of variance (ANOVA) for the response variables

	Source	Sum of squares	df*	Mean square	F-value	p-value	Significance
Ultrasonic pulse velocity - UPV	**Model**	0.1546	5	0.0309	29.83	< 0.0001	significant
	A - EAR	0.0008	1	0.0008	0.7381	0.4126	no significant
	B - Specimen age	0.1335	1	0.1335	128.77	< 0.0001	significant
	AB	0.0081	1	0.0081	7.84	0.0207	significant
	A^2	0.0115	1	0.0115	11.11	0.0088	significant
	B^2	0.0287	1	0.0287	27.64	0.0005	significant
	Residual	0.0093	9	0.0010			
	Total	0.1640	14				

df* - decrement factor

Table 5. Analysis of variance (ANOVA) for the response variables - continuation

	Source	Sum of squares	df*	Mean square	F-value	p-value	Significance
Flexural strength - f_f	**Model**	7.39	5	1.48	20.83	0.0001	significant
	A - EAR	0.0065	1	0.0065	0.0918	0.7688	no significant
	B - Specimen age	7.27	1	7.27	102.38	< 0.0001	significant
	AB	0.0571	1	0.0571	0.8039	0.3933	no significant
	A^2	0.0051	1	0.0051	0.0724	0.7939	no significant
	B^2	1.48	1	1.48	20.91	0.0013	significant
	Residual	0.6389	9	0.0710			
	Total	8.03	14				
Compressive strength - f_c	**Model**	251.36	5	50.27	45.66	< 0.0001	significant
	A - EAR	6.00	1	6.00	5.45	0.0444	significant
	B - Specimen age	243.86	1	243.86	221.50	< 0.0001	significant
	AB	2.09	1	2.09	1.89	0.2020	no significant
	A^2	0.6339	1	0.6339	0.5758	0.4674	no significant
	B^2	36.59	1	36.59	33.24	0.0003	significant
	Residual	9.91	9	1.10			
	Total	261.27	14				

*df - decrement factor

Table 6. Fit statistic results

Response	Standard deviation	Mean	Coefficient of variation [%]	R^2	Adjusted R^2	Predicted R^2	Adequate precision
UPV [km/s]	0.0322	4.32	0.7560	0.9431	0.9115	0.8558	16.2958
f_f [MPa]	0.2664	6.47	4.12	0.9205	0.8763	0.7813	11.2985
f_c [MPa]	1.05	35.16	2.98	0.9621	0.9410	0.8880	17.6668

$$UPV = 3{,}9970 + 4{,}4297 \cdot A + 0{,}0490 \cdot B - 0{,}1501 \cdot AB - 165{,}6066 \cdot A^2 - 0{,}0012 \cdot B^2 \quad (1)$$

$$f_f = 4{,}7247 - 0{,}2174 \cdot A + 0{,}3248 \cdot B + 0{,}3978 \cdot AB - 110{,}6461 \cdot A^2 - 0{,}0085 \cdot B^2 \quad (2)$$

$$f_c = 25{,}3402 + 40{,}2266 \cdot A + 1{,}7567 \cdot B - 2{,}4048 \cdot AB - 1228{,}5094 \cdot A^2 - 0{,}0424 \cdot B^2 \quad (3)$$

The R^2 values for the response variables (UPV, f_f and f_c) are 0.94, 0.92 and 0.96, respectively. The adjusted R^2 value shows that both the conformity of the obtained model and terms added to the model have significant effects on the response variable [26]. Ideally, the adjusted R^2 value should be high and should not show large deviations from the R^2 value. The differences between the R^2 and adjusted R^2 values of the models obtained for the UPV, f_f and f_c are 0.03, 0.04 and 0.02, respectively. In addition, the reasonable levels of the adjusted R^2 and estimated R^2 values (adjusted R^2-estimated R^2 = 0.20) [26] show that the amount of variability (estimation error) in the new data obtained from the models is appropriate. The adjusted R^2 - estimated R^2 values of the models obtained for the UPV, f_f and f_c are 0.056, 0.095 and 0.053, respectively.

The adequate precision value is used to calculate the signal-to-noise ratio; this value is greater than 4 [26]. The fact that the adequate precision values for all response variables (UPV; 16.296, f_f; 11.299 and f_c; 17.667) exceed the desired value indicates that the models create appropriate (sufficient) signals within the design space (within the selected variation intervals). The interaction plots for the UPV, f_f and f_c depending on the enzyme addition ratio at the maximum and minimum specimen ages, are shown in Figure 4.

The interaction graphs show that the 3-day f_f and f_c values do not vary as the EAR increases, but the UPV value tends to increase. Thus, it can be said that an increase in the amount of enzyme at an early age reduces the number of voids in the

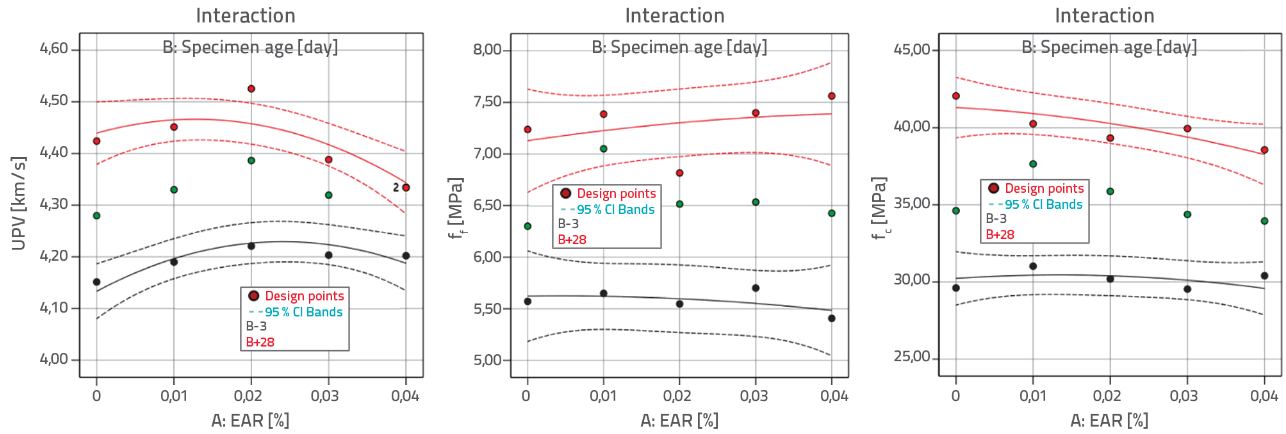

Figure 4. Interaction plots of UPV, f_f and f_c

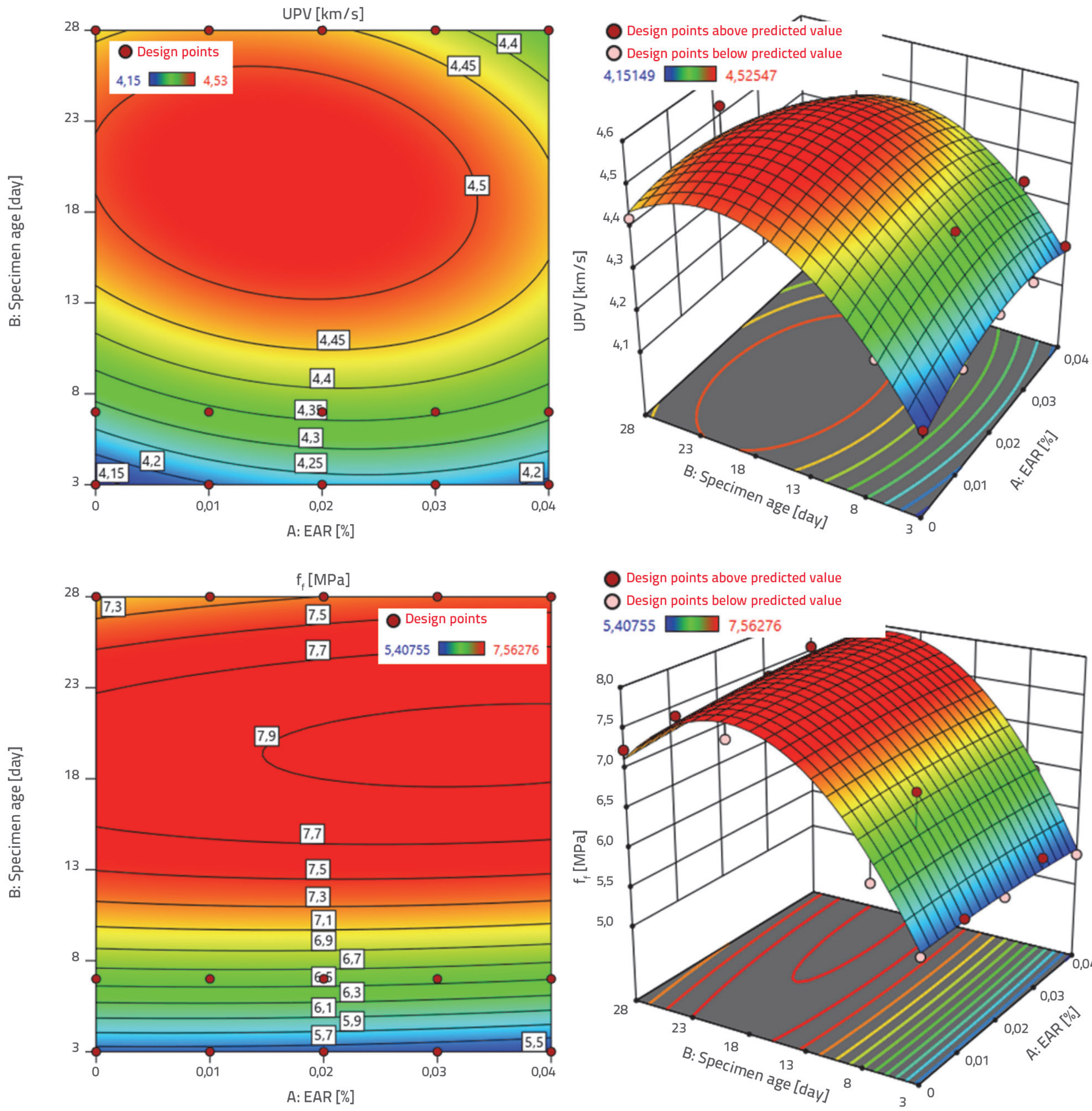

Figure 5. Contour and 3D plots of UPV, f_f and f_c

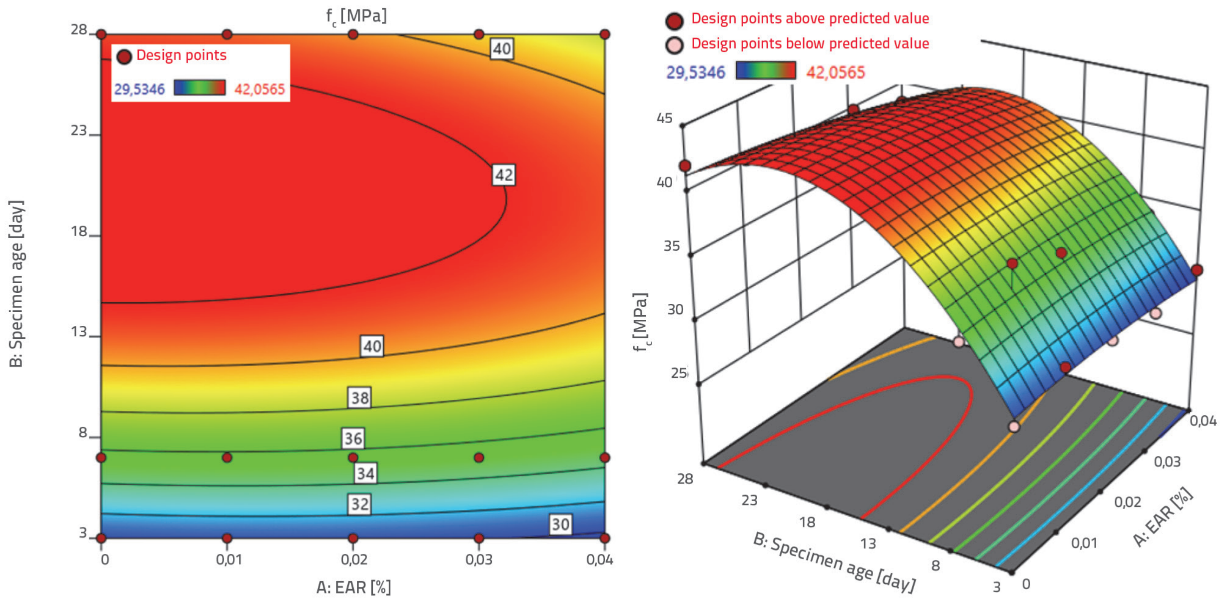

Figure 5. Contour and 3D plots of UPV, f_f and f_c - continuation

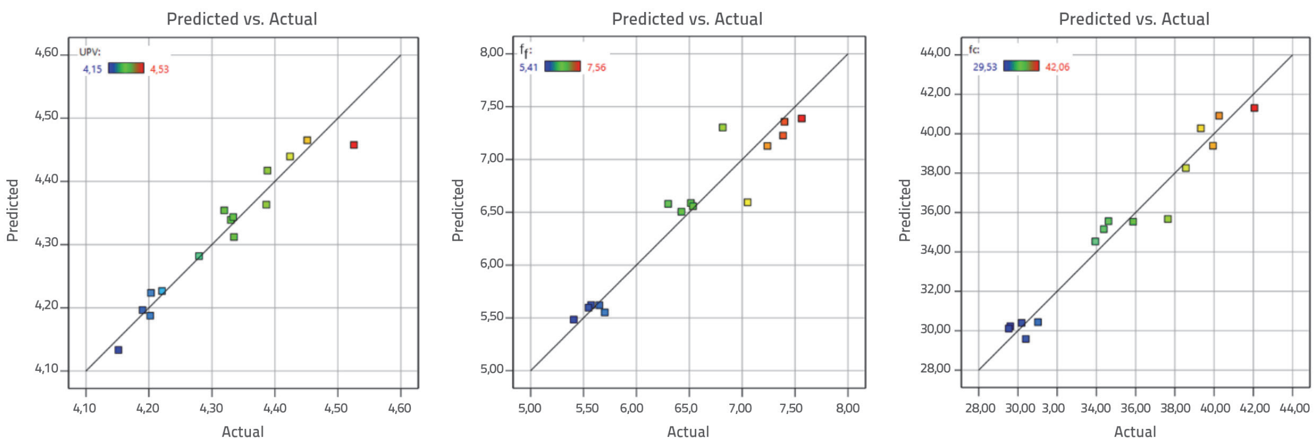

Figure 6. Predicted and actual values of response variables

mortars. In the 28-day samples, the UPV and fc values decrease as the EAR increases, whereas the f_f value slightly increases.

Contour and 3D plots for the UPV, f_f and f_c response variables are shown in Figure 5.

From Figure 5, it can be seen that the response surfaces and contour plots for the UPV show a simple maximum, but for f_f and f_c, they show rising bridges. The predicted and actual values of the response variables obtained as a result of the experimental design are shown in Figure 6.

The predicted values obtained from the models generated for the response variables overlap with the actual values (experimental results) at a very high level (Figure 5).

5. Conclusions

The usability of an enzyme admixture material (EarthZyme) used very effectively in soil compaction in mortars was determined by considering the effect levels of EAR and specimen age effect variables on the UPV, f_f and f_c response variables. The results are summarised below.

- As the EAR increases, the flow table values of the mortars increase.
- At early ages, (e.g., 3 days), the EAR does not significantly affect the properties of the mortar.
- At later ages (e.g., 28 days), the EAR decreases the UPV and f_c while increasing f_f.
- The main term of EAR has no significant effect on the UPV and f_f (p-value of UPV = 0.4126, p-value of f_f = 0.7688) but has a significant effect on f_c (p-value of f_c = 0.0444).
- The main term of specimen age significantly affects UPV, f_f and f_c (p-value < 0.0001) for all cement composites.
- The quadratic models obtained for the response variables, depending on the selected variation intervals of the effect variables, show very high estimation accuracies (R^2 >

0.92). According to the generated models, the actual and predicted values of the response variables are very close (Figure 6).

- The response surfaces and contour plots for UPV show a simple maximum, but show rising bridges for f_f and f_c.

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