Effect of Complex Organo-Mineral Modifier on the Properties of Corrosion-Resistant Concrete

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Abstract. This paper reports the results of a research study aimed at identifying the compositional features of fly ash (FA) TPP "Pha Lai" (Vietnam), silica fume SF-90 (SF90) and superplasticizer SilkRoad SR 5000F (SR5000) contents that influence the sulfate resistance of corrosion-resistant concrete. The effect of FA, SF90 and SR5000 on the strength of corrosion-resistant concrete is obtained quickly, high early-strength concrete. In details, the compressive strength at the age of 1 and 3 days are respectively 29.6% and 61.13% in comparison to 28 days period. The deformation of corrosion-resistant concrete prisms in 5% sodium sulfate solution after 28 days of testing were determined by Russian standard GOST P 56687-2015. Using the mathematical planning method for four factors and the Matlab computer programs was obtained the mathematical model, which was adequately described the influence ofthe water-binding ratio, FA, SF90 and SR5000 contents on the concrete deformation. It was also noted that the effect of FA content is more pronounced than the water-binding ratio and content SF90. In addition, the effect of SR5000 content on the deformation of concrete is negligible, so it was discarded.

1 Introduction

In the construction world the 21st century will be known as the century of concrete in the oceans. Early cracking of concrete and corrosion are a major concern for all offshore projects. The causes and mitigation alternatives are key aspects of creating and maintaining a sustainable offshore structure and hydroelectric project [1].

Vietnam, located in tropical climate area, has strong differentiation between the rainy season and the dry season. This country stretches lengthen from the North to the South, with a coastline of 3260 km long. Recently, the number of offshore and hydraulic projects in the coastal marine environment and the delta has increased several times. Based on some research analysis results, more than 50% of the nodes and structures of the coastal berthing and mooring facilities, breakwaters and tidal barriers, dry docks and jetties, container

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terminals, and offshore floating docks and drilling platforms in Vietnam were completely damaged or destroyed after 5 ÷ 20 years operation [2-4].

Accoding to [5-7] the performance of concrete against sulfate attack is associated with the permeability performance and the density of concrete. At the beginning, cement hydration dominantly happened to form a large amount of C-S-H gel, which provided the high strength for concrete mixture at an early ages. After one - two weeks, the pozzolanic reaction significantly happened. In the marine environment, it has happened the ion sulfate attack reaction (Equations 1 - 4) and become more dominant after several weeks. The deformation of gypsum and ettringite formed during the reaction process led to a reduction in strength of the concrete and maybe completely damaged structures.

In sulfate and marine environments, sulfate attack in concrete:

$$Na_2SO_4.2H_2O + Ca(OH)_2 \rightarrow CaSO_4.2H_2O + 2NaOH \quad (1)$$

$$Na_2SO_4.2H_2O + C\text{-}S\text{-}H \rightarrow CaSO_4.2H_2O + N\text{-}S\text{-}H \quad (2)$$

$$C_4AH_{13} + 3CaSO_4.2H_2O + 14H_2O \rightarrow C_3A.3CaSO_4.32H_2O + Ca(OH)_2 \quad (3)$$

$$C_3A.3CaSO_4.12H_2O + 2CaSO_4.2H_2O + 16H_2O \rightarrow C_3A.3CaSO_4.32H_2O \quad (4)$$

In fact, the deformation depend mainly on the concrete mixture design and to some extent on the type of cement used. In recent years organo-mineral modifiers such as superplasticizer an active mineral additive (natural pozzolan, rice husk ash, bottom ash and fly ash) obtained a wide circulation. The impact of various additives on the high performance concrete quality is studied [8, 9]. This is associated with the pozzolanic reaction to form the secondary C-S-H gel of mineral additives in concrete mix design [10, 11]. As such it is important to research effect of the complex organo-mineral modifier on the corrosion resistance of marine structures. In fact, the corrosion resistance of this concrete structures depend mainly on the concrete mixture design and to some extent on the type of cement used.

In this paper, the preliminary composition of corrosion-resistant concrete mixture is calculated according to the standard ACI 211.4R-08. The deformation of corrosion-resistant concrete prisms in 5% Na_2SO_4 solution after 28 days of testing were determined by Russian standard GOST P 56687-2015. The mathematical model is adequately described the influence of the complex organo-mineral modifiers on the concrete deformation.

2 Experimental work

2.1 Materials

a). Sulphate resisting portland cement (SPC) $PC_{SR}40$, manufactured at "Tam Diep" factory (Vietnam), specific weight of 3.15 g/cm^3. The results of physical properties, mechanical properties and cement's mineralogical composition are presented in Table 1 and 2 respectively.

Table 1. Chemical compositions of sulphate resisting Portland cement $PC_{SR}40$ (%)

CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	R_2O	SO_3	LOI [(a)]
63.48	21.37	4.14	5.15	2.53	1.05	0.63	1.65

Note: [(a)]*LOI - loss on ignition.*

Table 2. Physical and mechanical properties of sulphate resisting Portland cement $PC_{SR}40$

Specific weight (g/cm^3)	Surface area (cm^2/g)	Time of setting (min)		Compressive strength (MPa)			Standard consistency (%)
		Initial	Final	3 days	7 days	28 days	
3.15	3650	115	245	18.4	40.5	50.2	29.5

b). Quartz sand (QS): originally from the golden sand of "Lo River" (Vietnam), M_K = 3.1, specific weight of 2.65 g/cm^3. The volume of compacted state is 1540 kg/m^3.

c). Crushed limestone (CL) (Vietnam) with the size of 5 ÷ 10 mm, specific weight of 2.67 g/cm^3. The volume of the compacted state is 1580 kg/m^3.

d). Fly Ash (FA) TPP "Pha Lai" (Vietnam) class F and Silica Fume SF-90 (SF90) (Vina Pacific). The chemical composition and physical properties of the FA TPP "Pha Lai" and Silica Fume SF-90 are presented in Table 3 and 4.

Table 3. Chemical compositions of FA TPP "Pha Lai" and Silica Fume SF-90

Mineral additives	Average chemical composition (%)									
	SiO_2	Al_2O_3	Fe_2O_3	SO_3	K_2O	Na_2O	MgO	CaO	P_2O_5	LOI
FA TPP "Pha Lai"	54.62	24.17	6.15	2.81	1.28	1.25	1.57	1.48	1.63	5.04
Silica Fume SF-90	91.65	2.25	2.47	-	-	0.58	-	0.51	-	2.54

Table 4. Physical properties of FA TPP "Pha Lai" and Silica Fume SF-90

Свойства	FA TPP "Pha Lai"	Silica Fume SF-90
Specific weight (g/cm^3)	2.23	2.15
The volume of natural porous state (kg/m^3)	770	450
Specific surface area (m^2/g)	14.455	22.52
Humidity (%)	11	0
Retained content on sieve with a hole size of 45 μm (%)	18.6	0.25

e). Superplasticizer SilkRoad SR 5000F (SR5000) (South Korea). It is a new generation chemical additives based on polycarboxylate ethers with specific weight of 1.1 g/cm^3 at 20 ± 5 °C.

f). Ordinary clean tap water (W) was used for both mixing concrete and curing of test specimens.

2.2 Methods

Calculation method of concrete mixture composition is applied in accordance with ACI 211.4R-08 and the volume method. According to ASTM C143, the concrete mixture workability is determined by the standard slump cone with dimensions of 100x200x300 mm.

The compressive strength and tensile strength of corrosion-resistant concrete are by a 100x100x100 mm cube pattern by Russian standard GOST 10180-2012. These test samples are demolded after 24 hours later casting and placed in a 25 ± 5 ^{0}C water curing tank until the experiments. The corrosion-resistant concrete compressive strengths are measured at the ages of 1, 3, 7, 14 and 28 days. Deformation of corrosion-resistant concrete exposed to a sulfate solution is determined by a prism pattern of size 25x25x254 mm by Russian standard GOST P 56687-2015. Furthermore, the mathematical modeling, depicting the dependence of the water-binding ratio $(\frac{W}{BID})$, FA TPP "Pha Lai", SF90 and SR5000 on the concrete deformation, uses the mathematical experiment planning method with first-order for four factors [12-14].

3 Results and discussion

3.1 Mixture proportions and mixing procedures

The standard ACI 211.4R-08 (American method), which is used to determine the preliminary composition of the concrete mixture, requires the standard slump cone of 10 ÷ 18 cm and allows to obtain 70 MPa compressive strength corrosion-resistant concrete at the age of 28 days with ordinary hardening. In this research used fly ash TPP "Pha Lai" to replace 30% of the mass cement in concrete mixture. The amount of SR 5000F is 1% by the mass of Portland cement and the decrease of water content of the concrete mixture is about 30%.

All mixtures were mixed in a laboratory "Controls" mixer with a capacity of 150 liters. The mix ingredients placed in the mixer was in the following order; dry aggregates, cement, fly ash and Silica Fume were mixed in the mixer for 5 minutes then chemical additives and water were added gradually in 30 seconds and the mixing continued for 10 minutes. Therefore, the total mixing time was 15 ÷ 16 minutes for each concrete mixture. After mixing, a series of 100-mm cubes and prisms of size 25x25x254 mm concrete specimens were cast in pre-oiled moulds and fully compacted using vibration table and the top surface was leveled and finished by trowel. The fresh concrete properties as well as the concrete properties are represented in Table 5 and 6.

Table 5. Mixture composition and properties of concrete mixture

Concrete mixture compositions (kg/m^3)							Properties of fresh concrete mixture		
SPC	FA	SF90	QS	CL	SR5000	W	$\frac{W}{BID}$ (b)	Unit weight (kg/m^3)	Slump (cm)
406	122	40.6	595	1027	4.1	159	0.3	2353.7	16.5

<u>Note</u>: [b]BID = SPC + FA + SF90 and $\frac{W}{BID}$ *- ratio by weight.*

Table 6. Properties of corrosion-resistant concrete

Average density of concrete (kg/m^3)	Average compressive strength at different ages (MPa)					Average tensile strength at the age of 28 days (MPa)
	1 days	3 days	7 days	14 days	28 days	
2326.5	21.4	44.2	55.4	66.4	72.3	6.15

It can be seen from Table 4 that the corrosion-resistant concrete mixture composition calculation with 30% mass FA, 10% mass SF90 and 1% mass SR5000 the specified workability allow to obtain the required strength concrete. In addition, the test results showed that the effect of organic additives on the strength of corrosion-resistant concrete is obtained quickly, high early-strength concrete. In details, the compressive strength at the age of 1 and 3 days are respectively 29.6% and 61.13% in comparison to 28 days period.

3.2 Effect of organic additives on the deformation of corrosion-resistant concrete

In this paper, the achieved mathematical regression equation of the objective function is the deformation of corrosion-resistant concrete prisms by using the mathematical experiment planning method with first-order for four influencing factors.

3.2.1 Definition of objective functions for the description of empirical models

Y - deformation of corrosion-resistant concrete prisms in 5% Na_2SO_4 solution after 28 days of testing is considered as an objective function of the experimental model and is determined by formula (5):

$$\begin{aligned} У = {} & \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_1 x_2 + \beta_6 x_1 x_3 + \beta_7 x_1 x_4 + \beta_8 x_2 x_3 \\ & + \beta_9 x_2 x_4 + \beta_{10} x_3 x_4 + \beta_{11} x_1 x_2 x_3 + \beta_{12} x_1 x_3 x_4 + \beta_{13} x_2 x_3 x_4 + \beta_{14} x_1 x_2 x_3 x_4 \end{aligned} \quad (5)$$

where, Y is the response variable - (D, %) deformation of corrosion-resistant concrete prisms after 28 days of testing; β_0 to β_4 are the coefficients of linear model terms; β_5 to β_{10} are the coefficients of quadratic model terms, β_{11} to β_{14} are the coefficients of cubic model terms with added interaction terms; while x_1, x_2, x_3 and x_4 represent the factors employed during the current optimization studies.

3.2.2 Input influencing factors and their limitation

Input influencing factors and their limitation are sorted as below:

+ x_1 - the water-binding ratio $\frac{W}{BID}$, is in the range from 0.24 to 0.32;

+ x_2 - the content of FA, is in the range from 20% to 40% by mass SPC;

+ x_3 - the content of SF90, is in the range from 5% to 15% by mass SPC;

+ x_4 - the content of SR5000, is in the range from 0.5% to 1.5% by mass SPC.

In the concrete mixtures, the QS and CL contents are constants and equal, respectively, 595 kg and 1027 kg.

The input factors and intervals of their variation are shown in Table 7.

Table 7. Levels and intervals of influencing factors

Factors		Levels varying factors			Intervals varying factors
Parameter	Description	-1	0	+1	
$\frac{W}{BID}$	x_1	0.24	0.28	0.32	0.04
The content of FA (%)	x_2	20	30	40	10%
The content of SF90 (%)	x_3	5	10	15	5%
The content of SR5000 (%)	x_4	0.5	0.1	1.5	0.5%

The number of necessary experiments - N in first-order planning was determined by formula (6):

$$N = 2^k;\ \text{where k – the number of factors, k = 4.} \tag{6}$$

Consequently: $N = 2^4 = 16$.

Compositions of corrosion-resistant concrete, calculated by the method of the mathematical experiment planning for four influencing factors in first-order planning, are displayed in Table 8. The values of deformations concrete in 5% Na_2SO_4 solution after 28 days of testing are presented in Tables 9.

Table 8. Matrix of first-order planning for four factors and compositions of concrete mixtures

Trial No.	Description of code				Parameter				Compositions of concrete mixtures (kg/m^3)						
	x_1	x_2	x_3	x_4	$\frac{W}{BID}$	FA, %	SF90, %	SR5000, %	SPC	QS	CL	FA	SF90	SR5000	W
1	+	+	+	+	0.32	40	15	1.5	340	595	1027	136	51	5.1	168
2	-	+	+	+	0.24	40	15	1.5	384	595	1027	154	57.6	5.8	143
3	+	-	+	+	0.32	20	15	1.5	396	595	1027	79	59.5	5.9	171
4	-	-	+	+	0.24	20	15	1.5	449	595	1027	90	67.3	6.7	145
5	+	+	-	+	0.32	40	5	1.5	366	595	1027	146	18.3	5.5	170
6	-	+	-	+	0.24	40	5	1.5	414	595	1027	165	20.7	6.2	144
7	+	-	-	+	0.32	20	5	1.5	432	595	1027	86	21.6	6.5	173

8	-	-	-	+	0.24	20	5	1.5	490	595	1027	98	24.5	7.4	147
9	+	+	+	-	0.32	40	15	0.5	343	595	1027	137	51.4	1.7	170
10	-	+	+	-	0.24	40	15	0.5	388	595	1027	155	58.1	1.9	144
11	+	-	+	-	0.32	20	15	0.5	400	595	1027	80	60.1	2	173
12	-	-	+	-	0.24	20	15	0.5	454	595	1027	91	68.1	2.3	147
13	+	+	-	-	0.32	40	5	0.5	369	595	1027	148	18.4	1.8	171
14	-	+	-	-	0.24	40	5	0.5	418	595	1027	167	20.9	2.1	145
15	+	-	-	-	0.32	20	5	0.5	437	595	1027	87	21.8	2.2	175
16	-	-	-	-	0.24	20	5	0.5	496	595	1027	99	24.8	2.5	149

Table 9. Deformations of corrosion-resistant concrete in 5% Na_2SO_4 solution after 28 days of testing

Trial No.	Parameter				Deformations of corrosion-resistant concrete (%)						Ошибки дисперсии S_i^2, (10^{-7})
	$\frac{W}{BID}$	FA, %	SF90, %	SR5000, %	D_1	D_2	D_3	$Y_i^{cp}=D_i^{cp}$	$\overline{Y}_j$	$(Y_j-\hat{Y}_j)^2$ (10^{-5})	
1	0.32	40	15	1.5	0.02	0.021	0.02	0.02	0.027	4.976	3.33
2	0.24	40	15	1.5	0.034	0.035	0.035	0.035	0.035	0.006	3.43
3	0.32	20	15	1.5	0.01	0.011	0.012	0.011	0.008	0.789	3.73
4	0.24	20	15	1.5	0.014	0.015	0.014	0.014	0.016	0.366	0.93
5	0.32	40	5	1.5	0.032	0.034	0.033	0.033	0.033	0.071	10
6	0.24	40	5	1.5	0.043	0.044	0.042	0.043	0.041	0.197	6.03
7	0.32	20	5	1.5	0.013	0.013	0.014	0.013	0.015	0.181	0.23
8	0.24	20	5	1.5	0.015	0.016	0.016	0.015	0.023	5.476	0.43
9	0.32	40	15	0.5	0.029	0.03	0.03	0.029	0.027	0.648	7.43
10	0.24	40	15	0.5	0.035	0.036	0.036	0.036	0.035	0.048	4.3
11	0.32	20	15	0.5	0.013	0.013	0.013	0.013	0.008	2.063	0.9
12	0.24	20	15	0.5	0.016	0.014	0.015	0.015	0.016	0.19	4.3
13	0.32	40	5	0.5	0.033	0.035	0.034	0.034	0.033	0.021	13.3
14	0.24	40	5	0.5	0.047	0.047	0.041	0.045	0.041	0.923	120
15	0.32	20	5	0.5	0.014	0.014	0.014	0.014	0.015	0.124	0.3
16	0.24	20	5	0.5	0.027	0.027	0.032	0.029	0.023	3.364	87.03
Max $S_i^2 = 1.2.10^{-5}$						$\Sigma\left(Y_i^{cp}-\overline{Y_i}\right)^2 = 19.4.10^{-5}$				$S_{ll}^2 = \sum S_i^2 = 2.66.10^{-5}$	

3.2.3 The mathematical regression equation for the description of empirical models

The first-order regression equation and the values of the coefficients shown in formula (7), according to the calculated results obtained by Matlab program.

$$\begin{aligned} У_1 = {} & 0.02486 - 0.00406x_1 + 0.009315x_2 - 0.003227x_3 - 0.001823x_4 - 0.001223x_1x_2 \\ & + 0.0007101x_1x_3 + 0.000215x_1x_4 - 0.001015x_2x_3 + 0.0001396x_2x_4 + 0.000223x_3x_4 \\ & - 0.000727x_1x_2x_3 - 0.001448x_1x_3x_4 - 0.001173x_2x_3x_4 + 0.0003229x_1x_2x_3x_4 \end{aligned} \quad (7)$$

3.2.4 Checking the significance of the coefficients of the regression equation

According to [8, 9, 13, 14], checking the significance of the regression coefficients of the equation is performed by Student's criterion (t_α (f_2)). The coefficient b_j is considered significant if:

$$t_{bj} \geq t_\alpha (f_2), \quad (8)$$

where: t_α (f_2) - the value Student's criterion, obtained from table 3.2 [15]. At a significant level of $\alpha = 0.01$ and number of degrees of freedom $f_2 = N\times(k-1) = 16\times(3-1) = 48$ obtained $t_{0.001}$ $(48) = 2.4066$.

The values of the Student's criterion t_{bj} for the coefficients b_j are determined by the formula (9):

$$t_{bj} = \frac{|b_j|}{S_{bj}} \quad (9)$$

where: S_{bj} - dispersion estimates for the coefficients b_j of the regression equation of the experiment, determined by formula (10):

$$S_{b\delta} = \sqrt{\frac{S_{ll}^2}{N}} = \sqrt{\frac{2.66.10^{-5}}{16}} = 0.00129 \quad (10)$$

Table 10 are given the values of the Student's criterion for the coefficients of regression equation (6), according to the calculated results obtained by by the formula (9).

Table 10. The values of the Student's criterion of the coefficients of the regression equation (6)

j	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	b_0	b_1	b_2	b_3	b_4	b_{12}	b_{13}	b_{14}	b_{23}	b_{24}	b_{34}	b_{123}	b_{134}	b_{234}	b_{1234}
$b_j.10^{-5}$	**248.6**	**-40.6**	**93.15**	**-322.7**	-182.3	-122.3	71.01	21.5	-101.5	13.96	22.3	-72.7	-144.8	-117.3	32.29
$\|b_j\|.10^{-5}$	**248.6**	**40.6**	**93.15**	**322.7**	182.3	122.3	71.01	21.5	101.5	13.96	22.3	72.7	144.8	117.3	32.29
t_{bj}	19.29	3.15	7.23	2.504	1.414	0.949	0.551	0.166	0.787	0.108	0.173	0.564	1.124	0.91	0.251

After checking the significance of the coefficients of regression equation (6), insignificant coefficients were discarded, in the results of which the following equation was obtained:

$$У_1 = 0.02486 - 0.00406x_1 + 0.009315x_2 - 0.003227x_3 \quad (11)$$

3.2.5 Checking the adequacy of the experimental model

According to [4, 8, 9, 13, 14], the adequacy of the regression equations is checked by means of the Fisher criterion. The calculated value of which is the following ratio (12):

$$F_0 = \frac{S_d^2}{S_{ll}^2} \quad (12)$$

where: S^2_{ll}- variance estimates the reproducibility of the experiment $S_{ll}^2 = \sum S_i^2 = 2.66.10^{-5}$

S^2_d - estimation of the dispersion of inadequacy, determined by the formula (8):

$$S_d^2 = \frac{\sum_{j=1}^{N}(Y_j - \overline{Y}_j)^2}{N - m} \tag{13}$$

where: m - the number of coefficients of the regression equation (6), which are significant, m = 5 and N = 16;

Y_j - the observed value of the i-th experiment (in Table 9);

$\overline{Y}_j$ - the obtained value of the experimental function in accordance with the i-th experiment (in Table 5);

$$S_d^2 = \frac{19.4.10^{-5}}{16-4} = 1.62.10^{-5} \rightarrow F_{pacc} = \frac{S_d^2}{S_{ll}^2} = \frac{1.62.10^{-5}}{2.66.10^{-5}} = 0.609$$

F_α, (f_1, f_2) - the value Fisher criterion, obtained from table 3.5 [15], with a significant level of α = 0,01; f_1- number of degrees of freedom for residual variance f_1 =N =16 and f_2 - number of degrees of freedom for estimating the variance of observations f_2 = N - m = 16 - 4 = 12.

Consequently: $F_{0.01}$ (16, 12) = 4.2509.

As F_0 = 0.609 < $F_{0.01}$ (16, 12) = 4.2509, the experimental model described by mathematical regression equation (11) is adequate.

The regression equation (11) of experimental design showed that in the case of water-binding ratio and the content of SF90 decrease, but the content of FA increase, the deformation of corrosion-resistant concrete prisms in 5% Na_2SO_4 solution after 28 days of testing grow up. However, the effect of FA content is more pronounced than the water-binding ratio and the content of SF90.

4 Conclusion

Using the American standard ACI 211.4R-08, the preliminary composition of the concrete mixture with standard slump cone of 16 cm was calculated. The corrosion-resistant concrete was obtained with 72.3 MPa compressive strength and 6.15 MPa tensile strength at 28 day age of normal hardening.

1. The effect of fly ash, silica fume SF-90 and superplasticizer SilkRoad SR 5000F on the strength of corrosion-resistant concrete is obtained quickly, high early-strength concrete. In details, the compressive strength at the age of 1 and 3 days are respectively 29.6% and 61.13% in comparison to 28 days period.

2. The mathematical regression equation (11) of this experimental model is adequate, which was obtained by mathematical planning method for four influencing factors and the Matlab computer programs.

3. The deformation of corrosion-resistant concrete prisms were determined depending on the water-binding ratio, FA, SF90 and SR5000 contents in accordance with Russian standard GOST P 56687-2015. It was also noted that the effect of FA content is more pronounced than the water-binding ratio and SF90 content. In addition, the effect of SR5000 content on the deformation of concrete is negligible, so it was discarded.

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