Strength of copolymer grouting material based on orthogonal experiment

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Abstract: Using the orthogonal experimental design method involving three factors and three levels, the flexural strength and the compressive strength of copolymer grouting material were studied with different compositions of water-cement ratio (mass fraction of water to cement), epoxy resin content, and waterborne epoxy curing agent content. By orthogonal range and variance analysis, the orders of three factors to influence the strength, the significance levels of different factors, and the optimized compound ratio scheme of copolymer grouting material mixture at different curing ages were determined. An empirical relationship among the strength of copolymer grouting material, the water-cement ratio, the epoxy resin content, and the waterborne epoxy curing agent content was established by multivariate regression analysis. The results indicate that water-cement ratio is the most principal and significant influencing factor on the strength. Epoxy resin content and waterborne epoxy curing agent content also have a significant influence on the strength. But epoxy resin content has a greater influence on the 7-day and 28-day flexural strength, and waterborne epoxy curing agent content of 0.4, epoxy resin content of 8% (mass fraction) and waterborne epoxy curing agent content of 2% (mass fraction) is the best one for repairing of cement concrete pavement. The flexural strength and the compressive strength have good correlation, and the ratio of compressive strength to flexural strength is between 1.0 and 3.3.

Key words: strength; copolymer; chemical grouting; orthogonal method; regression model

1 Introduction

Nowadays, more and more cement concrete pavements are aging in China. But the pavement performance is affected by the properties of the concrete, such as the strong rigidity, insufficient flexibility, and low deformability [1-2]. Under the vehicle load, the pumping area will be triggered between the pavement and the subgrade, or the pavement may be cracked. Cracks and holes of the pavements threaten the safety. Maintenance, repair, and strengthening of concrete structure have become a very important part of the activities of the concrete industry. It is a more convenient and cheaper way to repair the concrete structure than to rebuild it. The chemical grouting technique is widely used to repair pavement for its ecological safety and resource-saving from the viewpoint of global environment protection [3-5].

The polymer materials for repairing of the pavement are received increasing attention. Much work was conducted on polymer materials for asphalt pavement

[6-9] and cement concrete pavement [10-11]. OHAMA [12] used polymer-based materials to repair concrete structure and improve its durability. PAWAN et al [13] presented laboratory investigations on the performance of the styrene butadiene styrene (SBS) and linear low-density polyethylene (LLDPE) modified mixtures, and determined the optimum dose of the modifier on the basis of empirical tests. They pointed out that the polymer has an important effect on the characteristics of the mixtures. SHEN et al [14] prepared the cement concrete for repairing the structure macrocrack using polymer modified superfine cement, which modified by the polymer latex S400 with properties of flexibility, waterproofness, and cohesiveness. The polymer can improve the interface cohesiveness between the new material and the old one. It can also increase the flexibility and enhance the waterproofness and corrosion resistance. KOSEDNAR and MAILVAGANAM [15] introduced the polymers into the repairing of concrete structure. All above studies provide a theoretic foundation for the application of the polymer material to the pavement. However, in the current design and con-

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struction, the components and the strength of grouting materials are mainly determined by engineers' experience, which probably leads to quality problems in engineering and high lost as a result of an unreasonable ratio design.

The objective of the present study was to find a kind of copolymer grouting material for quick repairing of cement concrete pavement.

2 Experimental

2.1 Materials

According to the working conditions of grouts concretion body, the polymer grouting material should possess the following basic requirements: good impermeability, good groutability, high bond strength, good stability, low volume shrinkage, good physical and mechanical properties and being distributed in water.

(1) Cement (P.O 32.5): Ordinary Portland cement 32.5R was manufactured by Hunan Shaofeng Cement Group Co., Ltd. Its chemical components are listed in Table 1. The measured 3-day flexural strength of cement used in the test is 3.0 MPa and the 28-day flexural strength is up to 5.9 MPa. The measured 3-day compressive strength is 15.3 MPa and the 28-day compressive strength is 41.7 MPa.

(2) Bisphenol-A epoxy resin (DDR331): The resin manufactured by Shanghai Lujia Water Coating Co., Ltd is milk-white latex with solid content of 40%–43%, density of 1.06-1.15 g/cm³, and pH value of 6–7.

(3) Waterborne epoxy curing agent (GCA01): The agent produced by Shanghai Lujia Water Coating Co., Ltd is a glutinous fluid with yellow and transparent appearance. Its solid content is 45%-47%, the density is 1.08-1.12 g/cm³, and the pH value is in the range of 8-9.

Table 1 Chemical components of cement

Component	Mass fraction/%	Component	Mass fraction/%
SiO ₂	23.42	CaO	55.97
Al_2O_3	7.18	MgO	2.26
Fe ₂ O ₃	3.18	f-CaO	0.85
Alkali	0.54	Insoluble	0.07
Ignition loss	2.73		

2.2 Orthogonal experimental design

The pumping area can be filled with copolymer grouts by stuffing, infiltrating, and compaction grouting. The grouts concretion bodies can improve load-carrying capacity of concrete panel base and recover the continuity between concrete panel and basement. According to the stress characteristics of the cement concrete pavement, the concretion bodies should possess high flexural strength and high compressive strength. Regularly, the compressive strength is higher than the flexural strength for concrete. As the compressive strength is easier to meet the requirement of the construction, the flexural strength is chosen as the controlling index and the compressive strength as the reference index in this work.

There are many factors influencing the performance of copolymer concretion body. Among these, water-cement ratio, epoxy resin content and waterborne epoxy curing agent content are important ones. The water-cement ratio is mass fraction of water to cement, and the epoxy resin content and the waterborne epoxy curing agent content are mass fractions of epoxy resin or agent to cement, respectively. The orthogonal table $L_9(3^4)$ [16] was used to array the factors. The orthogonal factorial design is shown in Table 2.

Table 2	Factors and	levels i	for	orthogonal	test (mass fract	tion)
				0			

	Factor								
Level	Water-cement ratio (A)	Epoxy resin content (B)/%	Waterborne epoxy curing agent content (C)/%						
1	0.6	6	1						
2	0.5	8	2						
3	0.4	15	4						

2.3 Experimental method

Firstly, the cement was put into water according to the designed mass fraction and blended to form pure cement grouts. Secondly, the epoxy resin DDR331 and the waterborne epoxy curing agent GCA01 were mixed according to the designed formula, and proper water was added to resin/agent mixtures to bring out copolymer chemical grouts after being stirred uniformly. Lastly, the grouts were put into the standard model of 40 mm×40 mm×160 mm, and the grouts concretion bodies were demoulded, while the grouts were solidified after curing for 24 h. The concretion bodies were cured at 20 °C for 3–28 d according to Testing Methods of Concrete for Highway Engineering (JTJ053–94).

The flexural tests on copolymer concretion bodies were carried out by lever testing machine at an overloading speed of 50 N/s. The broken bodies were taken to perform compressive tests at an overloading speed of 5 kN/s.

The flexural strength was calculated by the following equation:

$$R_{\rm f} = \frac{3PL}{2bh^2} \tag{1}$$

where $R_{\rm f}$ is the flexural strength, MPa; P is the failure

load, N; *L* is the support span, L=100 mm; *b* is the width and *h* is the height, and here they are equal to 40 mm.

The compressive strength was calculated by the following equation:

$$R_{\rm c} = \frac{P}{S} \tag{2}$$

where R_c is the compressive strength, MPa; and S is the compression area, S=40 mm × 40 mm=1 600 mm².

3 Results and discussion

3.1 Results

The flexural strength and the compressive strength of the samples are listed in Table 3. It is noteworthy that K, k, R, and r are calculated and listed in Table 4, where K is the sum of experimental value for each factor in every level, k is the average value, R, and r are the

Table 3 Results of orthogonal experiments on copolymer grouting material

Trial No.		Factor		<i>R</i> _f /MPa			<i>R</i> _c /MPa		
IIIai INO.	А	B / %	C / %	3-day	7-day	28-day	3-day	7-day	28-day
1	0.6	6	1	3.92	4.76	6.43	6.64	9.64	17.23
2	0.6	8	2	5.71	7.13	9.23	5.84	12.71	24.92
3	0.6	15	4	5.16	6.59	9.07	11.23	15.91	29.93
4	0.5	6	2	5.66	6.31	8.99	7.84	14.88	26.52
5	0.5	8	4	7.43	9.27	12.46	15.23	21.88	40.74
6	0.5	15	1	6.12	7.38	9.87	10.87	16.04	29.59
7	0.4	6	4	7.35	9.61	12.67	15.70	21.06	41.30
8	0.4	8	1	8.21	10.21	13.68	12.70	21.15	40.86
9	0.4	15	2	7.74	10.26	13.40	14.21	21.83	41.14

Table 4 Range analysis on strength of copolymer grouting material

F ester	3-day strength /MPa			7-da	ay strength /I	МРа	28-day strength /MPa		
Factor	А	В	С	А	В	С	А	В	С
$K_{f1} = \sum_{i=1}^{3} K_{f1i}$	14.11	17.27	15.85	18.48	20.68	22.35	24.73	28.09	29.98
$K_{f2} = \sum_{i=1}^{3} K_{f2i}$	18.32	20.52	19.11	22.96	26.61	23.70	31.32	35.37	31.62
$K_{f3} = \sum_{i=1}^{3} K_{f3i}$	23.49	18.13	20.96	30.08	24.23	25.47	39.75	32.34	34.20
$R_{\rm f} = K_{\rm fmax} - K_{\rm fmin}$	9.38	3.25	5.11	11.60	5.93	3.12	15.02	7.28	4.22
$k_{\rm fl} = K_{\rm fl}/3$	4.70	5.76	5.28	6.16	6.89	7.45	8.24	9.36	9.99
$k_{f2} = K_{f2}/3$	6.11	6.84	6.37	7.65	8.87	7.90	10.44	11.79	10.54
$k_{f3} = K_{f3}/3$	7.83	6.04	6.99	10.03	8.08	8.49	13.25	10.78	11.40
$r_{\rm f} = R_{\rm f}/3$	3.13	1.08	1.70	3.87	1.98	1.04	5.01	2.43	1.41
$K_{\rm cl} = \sum_{i=1}^{3} K_{\rm cli}$	23.71	30.18	30.21	38.26	45.58	46.83	72.08	85.05	87.68
$K_{c2} = \sum_{i=1}^{3} K_{c2i}$	33.94	33.77	27.89	52.8	55.74	49.42	96.85	106.52	92.58
$K_{c3} = \sum_{i=1}^{3} K_{c3i}$	42.61	36.31	42.16	64.04	53.78	58.85	123.3	100.66	111.97
$R_{\rm c} = K_{\rm cmax} - K_{\rm cmin}$	18.9	6.13	14.27	25.78	10.16	12.02	51.22	21.47	24.29
$k_{c1} = K_{c1}/3$	7.90	10.06	10.07	12.75	15.19	15.61	24.03	28.35	29.23
$k_{c2} = K_{c2}/3$	11.31	11.26	9.30	17.60	18.58	16.47	32.28	35.51	30.86
$k_{c3} = K_{c3}/3$	14.20	12.10	14.05	21.35	17.93	19.62	41.10	33.55	37.32
$r_{\rm c} = R_{\rm c}/3$	6.30	2.04	4.76	8.59	3.39	4.01	17.07	7.16	8.10

ranges of *K* and *k*, respectively.

3.2 Range analysis

As seen from Table 4, the influence on the 3-day flexural strength is in the order: A > C > B, and the influence on the 7-day and 28-day flexural strength is in the order: A > B > C according to the *R* or *r* values. These suggest that waterborne epoxy curing agent has an outstanding influence on the early flexural strength of the samples. The optimal mixing proportion can be obtained to be A3B2C3 when water-cement ratio, epoxy resin content and waterborne epoxy curing agent content are 0.4, 8% and 4%, respectively. But for *R*_f of factor C, *K*₂ is almost equal to *K*₃. In other words, the two levels have little influence on the flexural strength. So, the optimal mixing proportion can be adjusted to A3B2C2 for saving cost.

It can also be seen from Table 4 that the influence on the compressive strength is in the order: A > C > B. In comparison with the influence on the flexural strength, the influence of waterborne epoxy curing agent increases and the optimal mixing proportion is A3B2C3.

3.3 Variance analysis

To verify the distinctiveness of each factor consulting to the *F*-distribution laws, $F(a, f_1, f_2)$, where f_1 and f_2 are the freedoms of the numerator and denominator in the ratio of variance, respectively. The consulting value of *F*-distribution used here included $F_{0.01}(2, 2)=99.0$, $F_{0.05}(2, 2)=19.0$, $F_{0.10}(2, 2)=9.0$ [17]. According to the distinctiveness verification shown in Table 5, the most sensitive factors influencing the flexural strength are water-cement ratio and epoxy resin content, while waterborne epoxy curing agent content is only a marked factor.

The compressive strength is also influenced by waterborne epoxy curing agent content at the highly marked level to some degree, but epoxy resin content and waterborne epoxy curing agent content are only marked factors.

3.4 Empirical formula of regression analysis

According to the methods in Refs.[18–19], it is assumed that there exists linear relationship among the

strength of copolymer grouting material, water-cement ratio, epoxy resin content, and water borne epoxy curing agent content. Therefore, the regression model can be presumed as

$$y = e_0 + e_1 x_1 + e_2 x_2 + e_3 x_3 + e \tag{3}$$

where *y* stands for the flexural strength or the compressive strength, e_i (*i*=0, 1, 2, 3) is the regression coefficient, x_1 stands for the water-cement ratio, x_2 stands for the epoxy resin content, x_3 stands for the water borne epoxy curing agent content, and *e* is the error.

The regression equations of the flexural strength in different curing ages are obtained by the least-squares method as follows:

 $y_3=12.764-14.183x_1+0.028x_2+0.181x_3$, $R^2=0.89$, n=9 (4) $y_7=16.169-19.333x_1+0.680x_2+0.339x_3$, $R^2=0.90$, n=9 (5) $y_{28}=21.320-25.033x_1+0.079x_2+0.463x_3$, $R^2=0.91$, n=9 (6) where y_3 , y_7 , y_{28} stand for the 3-day, 7-day, and 28-day flexural strength, respectively, and R^2 is the correlation coefficient.

The regression equations of the compressive strength in different curing ages are as follows:

$$y'_3 = 21.515 - 31.500x_1 + 0.199x_2 + 1.478x_3, R^2 = 0.94, n = 9$$

(7)

 $y'_1 = 23.588 - 42.967x_1 + 0.200x_1 + 0.137x_2$

$$y_7 = 55.588 - 42.907x_1 + 0.200x_2 + 0.157x_3,$$

$$R^2 = 0.94, n = 9$$
(8)

$$y'_{28} = 65.254 - 85.367x_1 + 0.354x_2 + 2.775x_3,$$

$$R^2 = 0.94, n = 9$$
(9)

where y'_3 , y'_7 , and y'_{28} stand for the 3-day, 7-day, and 28-day compressive strength, respectively.

When the confidence degree is 1%, the critical value of R^2 equals 0.875. All the regression coefficients, not only of flexural strength but also of compressive strength, are larger than the critical values. Therefore, the achieved linear regression equations are of valuable. The variance analytical results of the regression equation are shown in Table 6.

3.5 Relationship between flexural strength and compressive strength

The compressive strength and the flexural strength

Table 5 Variance analysis on 28-day flexural strength and 28-day compressive strength

		,		5	0	, j 1		0			
Factor	28-day flexural strength					28-day compressive strength					
	SS	DOF	MS	F	Significance	SS	DOF	MS	F	Significance	
	А	37.79	2	18.89	592.70	Highly marked	437.40	2	218.70	240.33	Highly marked
	В	8.92	2	4.40	139.84	Highly marked	82.11	2	41.05	45.11	Marked
	С	3.02	2	1.51	47.32	Marked	110.00	2	55.00	60.44	Marked
	Error	0.06	2	0.03			1.82	2	0.91		
	Total	49.78	8				631.33	8			

Note: SS stands for sum of squares, DOF stands for degree of freedom, MS stands for mean square, and F represents F-value.

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Table 6 Variance analytical results on regression equations of strength											
Factor	SS	DOF	MS	F	F _C	Significance					
$R_{\rm f3}$	12.634	3	4.211	6.619	$F_{0.01}(3, 5)=12.06$	Marked					
$R_{ m f7}$	24.649	3	8.216	6.842	$F_{0.05}(3, 5)=5.41$	Marked					
$R_{\rm f28}$	41.434	3	13.811	8.269		Marked					
R_{c3}	95.436	3	31.812	12.000	$F_{0.01}(3, 5)=12.06$	Marked					
R_{c7}	142.377	3	47.459	13.828	$F_{0.05}(3, 5)=5.41$	Highly marked					
<i>R</i> _{c28}	561.873	3	187.291	13.482		Highly marked					

Note: R_{13} , R_{17} and R_{128} are the 3-day, 7-day and 28-day flexural strength, respectively; R_{c3} , R_{c7} and R_{c28} are the 3-day, 7-day and 28-day compressive strength, respectively; and F_C is critical value of F.

of grouting material mainly depend on the factors including cement activity, cement dosage and watercement ratio. Generally, the flexural strengths closely associated with the compressive strength. The flexural strength increases with increasing compressive strength, but the flexural strength is much lower than the compressive strength for the same concretion body.

The relationship between the flexural strength and the compressive strength is shown in Fig.1, and the regression equation is listed as follows:

$$Y = 3.868X - 11.899, R^2 = 0.938, n = 27$$
(10)

where Y and X are the compressive strength in MPa and the flexural strength in MPa, respectively.



Fig.1 Relationship between compressive strength and flexural strength

The ratios of the flexural strength to the compressive strength for the testing samples are between 1.0 and 3.3, which are little lower than the values in Ref.[20], indicating that the flexural strength of copolymer grouting material used in this work has been greatly increased, the pavement performance has been obviously improved, and the flexibility has been remarkably strengthened.

4 Conclusions

(1) The copolymer grouting material is very suitable for repairing the cement concrete pavement. The optimum proportion of the copolymer grouting material is obtained by orthogonal experiment.

(2) The empirical formulae of the flexural strength and the compressive strength with water-cement ratio, epoxy resins content and waterborne epoxy curing agent content are established.

(3) The ratio of compressive strength to flexural strength indicates that the copolymer grouting material has good pavement performance.

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