磷石膏稳定红黏土的压缩特性及微观机理

罗国夫,陈开圣,骆弟普
(, 550025)

摘要: , , ,
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, 、 。 ,
1∶3 。 > > 。
、 。 , 、
, 1∶3 , 。
, , , , , 。

关键词: ; ; ; ; ;
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Compression Characteristics and Microscopic Mechanism of Phosphogypsum-Stabilized Red Clay

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Abstract: In view of special engineering properties of red clay and the waste of phosphogypsum solid-waste resource, the compression characteristics of phosphogypsum-stabilized red clay under different mixing ratios were studied through consolidation tests with cement as curing agent. And the mechanism of compressive modulus change law was analyzed based on microstructure and mineral composition. The results show that plain red clay presents medium compressibility, while phosphogypsum-stabilized red clay presents medium and low compressibility. The compressive modulus of mixture increases first and then decreases with the increase of phosphogypsum content. The compressive modulus is the largest as the mass ratio of cement to phosphogypsum is 1∶3. The degree of influence on the compressive modulus of mixture is vertical load > cement content > phosphogypsum content. The compressive modulus of mixture can be fitted by a binary cubic function relationship with cement content and phosphogypsum content. With the increase of phosphogypsum content, the hydration reaction, adsorption of cement and the formation of ettringite reduce the porosity of the mixture. When the mass ratio of cement to phosphogypsum is 1∶3, the porosity is minimum. When the content of phosphogypsum continues to increase, the solution is acidic, ettringite is dissolved, the content of free iron oxide decreases, the cementation and mechanical bite of aggregates are reduced, and the compression modulus is reduced.

Key words: phosphogypsum; red clay; cement; compression characteristic; compressive modulus; microscopic mechanism

0 引 言

, 5 ~ 7 m,
20 m, , 、 ,
、 [1-3]。《 》(JTG D30—2015) 3.3.3 ,

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50%、 26 。 ,
, 、 、 、 、 [4-6]。
, 3 , 10% 。
, ,
[7-8]。 [9] 、 、 、
, , , -
- - ( ) 15∶20∶6∶59 15∶25∶8∶52。 [10]
, 、 、 (California bearing ratio, CBR) ,
[11]。 , 、
、 , [12]。
CBR 、 7 d ,
[13]。 ,
, , 。
, 。
、 , , 、
。 , ,
, , 。

1 实 验

1.1 原材料

K14 +000 ~ K16 +000 , 0 ~ 3 m,
, , , , , 1。

表 1 红黏土的基本物理指标

Table 1 Basic physical indexes of red clay

Density/ ($g \cdot cm^{-3}$)	Moisture content/%	Optimum moisture content/%	Maximum dry density/ ($g \cdot cm^{-3}$)	Liquid limit/%	Plastic limit/%	Nonuniform coefficient	Curvature coefficient
1.76	60.03	30.24	1.46	82.13	43.02	10.63	1.085

, , 、 。 2,
3, 4。 4 , 《
》(GB 5085.3—2007) , 《 》
(GB 6566—2010) 。
P·C 32.5R , , , , 5。 ,
《 》(GB 175—2007) 。

表 2 磷石膏的基本参数

Table 2 Basic parameters of phosphogypsum

Specific surface area/ ($m^2 \cdot kg^{-1}$)	Loss on ignition/%	Moisture content/%	Alkali content/%	Density/ ($g \cdot cm^{-3}$)	Fineness/%
102	18.43	5.3	1.31	2.38	44.3

表 3 磷石膏的化学成分

Table 3 Chemical composition of phosphogypsum

Composition	SO_3	CaO	SiO_2	P_2O_5	Na_2O	Al_2O_3	Other
Mass fraction/%	49.070	40.070	5.780	1.350	0.587	0.435	2.708

表 4　磷石膏的重金属及放射性检测结果

Table 4　Test results of heavy metals and radioactivity of phosphogypsum

Category	Test item	Standard limit	Result	Conclusion
Heavy metal	Cu concentration/(mg · L^{-1})	≤100	0.157	Qualified
	Zn concentration/(mg · L^{-1})	≤100	0.051	Qualified
	Cd concentration/(mg · L^{-1})	≤1	0	Qualified
	Pb concentration/(mg · L^{-1})	≤5	0	Qualified
	Cr concentration/(mg · L^{-1})	≤15	0	Qualified
	As concentration/(mg · L^{-1})	≤5	0.035 6	Qualified
	Hg concentration/(mg · L^{-1})	≤0.1	0.000 5	Qualified
Radioactivity	Ra-226/(Bq · kg^{-1})	—	53.94	—
	TH-232/(Bq · kg^{-1})	—	42.13	—
	K-40/(Bq · kg^{-1})	—	52.95	—
	I_{Ra}	≤1.0	0.3	Qualified
	I_{γ}	≤1.0	0.3	Qualified

Note: I_{Ra} is internal exposure index; I_{γ} is external exposure index.

表 5　水泥的基本参数

Table 5　Basic parameters of cement

Item	Index	Item	Index	Item	Index
3 d f_{cf}/MPa	5.0	Loss on ignition/%	1.58	Initial setting time/min	302
28 d f_{cf}/MPa	6.7	Alkali content/%	2.42	Final setting time/min	322
3 d f_{cu}/MPa	24.9	Chloride ion content/%	0.018	Stability	Qualified
28 d f_{cu}/MPa	43.7	Sulfur trioxide content/%	2.87		

Note: f_{cf} is flexural strength; f_{cu} is compressive strength.

《　　》(JTG/T F20—2015)　　,
、　　12　　,　　6% ~14% (　　)。
,　　3%、5%、7%。[14-16]　　,
1∶1、1∶2　1∶3　　,　　1∶1、1∶2
1∶3　　,　　6。　　C　　,　　P　　,　　T　　。

表 6　磷石膏稳定红黏土的配合比

Table 6　Mix proportion of phosphogypsum-stabilized red clay

Item	C content/%	m_P∶m_T	P content/%	T content/%	Maximum dry density/(g · cm^{-3})	Optimum moisture content/%
High content phosphogypsum	3	1∶1	48.50	48.50	1.52	25.47
	3	1∶2	32.33	64.67	1.51	25.84
	3	1∶3	24.25	72.75	1.45	26.96
	5	1∶1	47.50	47.50	1.55	22.88
	5	1∶2	31.67	63.33	1.54	25.92
	5	1∶3	23.75	71.25	1.46	27.11
	7	1∶1	46.50	46.50	1.51	24.00
	7	1∶2	31.00	62.00	1.54	23.93
	7	1∶3	23.25	69.75	1.53	25.21
Item	**C content/%**	**m_C∶m_P**	**P content/%**	**T content/%**	**Maximum dry density/(g · cm^{-3})**	**Optimum moisture content/%**
Low content phosphogypsum	3	1∶1	3.00	94.00	1.50	30.30
	3	1∶2	6.00	91.00	1.50	29.18
	3	1∶3	9.00	88.00	1.54	27.83
	5	1∶1	5.00	90.00	1.51	26.15
	5	1∶2	10.00	85.00	1.52	27.41
	5	1∶3	15.00	80.00	1.54	26.35
	7	1∶1	7.00	86.00	1.52	27.98
	7	1∶2	14.00	79.00	1.49	27.20
	7	1∶3	21.00	72.00	1.52	27.32

Note: % represents mass fraction.

1.2 制备样品

、 2 mm ， 。 ，
3%， ， 24 h。 3%
， 。 ， 68.1 mm
， 90%， 。 1 h 。
(20 ±2) ℃、 ≥95% 7 d， 。

1.3 压缩试验

《 》(JTG 3430—2020) 。 50、100、200、400、800 kPa
， 1 h， ， 1 h ，
24 h ， ， 、 、 。
(1) 。

$$e_0 = \frac{(1+0.01\omega_0)\rho_s}{\rho_0} - 1 \tag{1}$$

：e_0 ， 0.01；ρ_0 ，g/cm^3；ω_0 ，%；ρ_s
，g/cm^3。

(2) (3) 。

$$a_v = \frac{(S_{i+1} - S_i)(1+e_0)/1\,000}{p_{i+1} - p_i} \tag{2}$$

$$S_i = \frac{\sum \Delta h_i}{h_0} \tag{3}$$

：a_v ，MPa^{-1}；p_i ，kPa；S_i ， 0.1 mm；h_0
，mm；$\sum \Delta h_i$ ，mm。

(4) 。

$$E_s = \frac{p_{i+1} - p_i}{(S_{i+1} - S_i)/1\,000} \tag{4}$$

：E_s ，MPa。

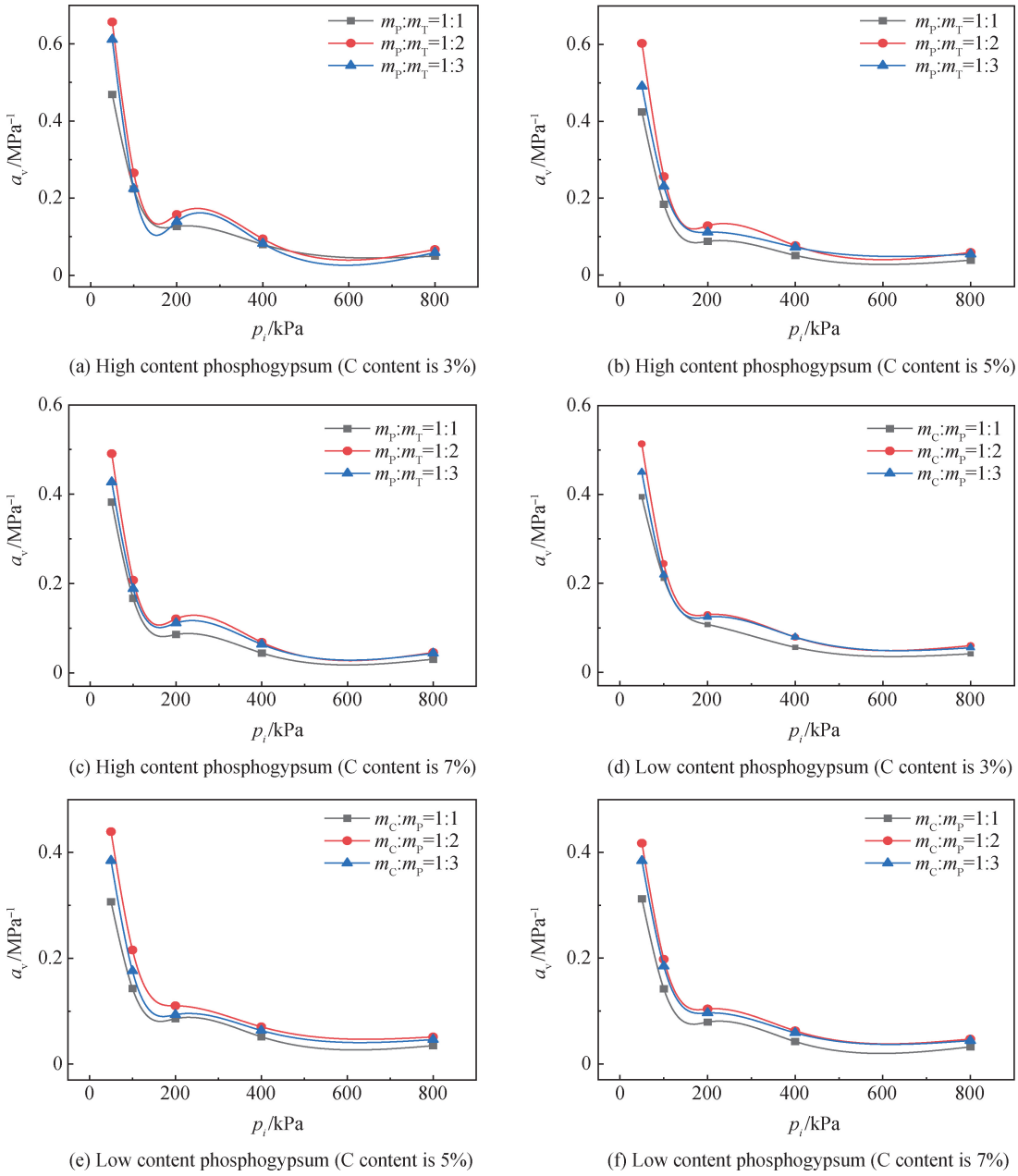
1.4 微观结构试验

， X 。
， ， ， 61.8 mm、 20 mm 。
(20 ±2) ℃、 95% 7 d，7 d 。
HITACHI SU8100， 3.00 kV。 2 cm^3 。
， 0.5 cm^3 。 ，
。 ， 。X
Rigaku SmartLab， 0.02°， 15° ~ 80°， 10 (°)/min。 。

2 结果与讨论

2.1 压缩系数与竖向荷载的关系

1 。 1 ， 200 kPa ，
， 200 kPa ， 。 0 ~ 200 kPa，
， ，
， 81.667% ~ 84.576%； 200 ~ 800 kPa，
， ，
15.423% ~ 18.333%。



(a) High content phosphogypsum (C content is 3%)

(b) High content phosphogypsum (C content is 5%)

(c) High content phosphogypsum (C content is 7%)

(d) Low content phosphogypsum (C content is 3%)

(e) Low content phosphogypsum (C content is 5%)

(f) Low content phosphogypsum (C content is 7%)

1

Fig. 1 Relationship between compressibility coefficient and vertical load of mixture

《 》(GB 50007—2011) , a_v 、 、 。 $a_v<0.1$ MPa^{-1}, ,0.1 MPa$^{-1}\leqslant a_v<0.5$ MPa^{-1}, , $a_v>0.5$ MPa^{-1}, 。 7 。 7 , , 、 。

表 7 磷石膏稳定红黏土压缩性

Table 7 Compressibility of phosphogypsum-stabilized red clay

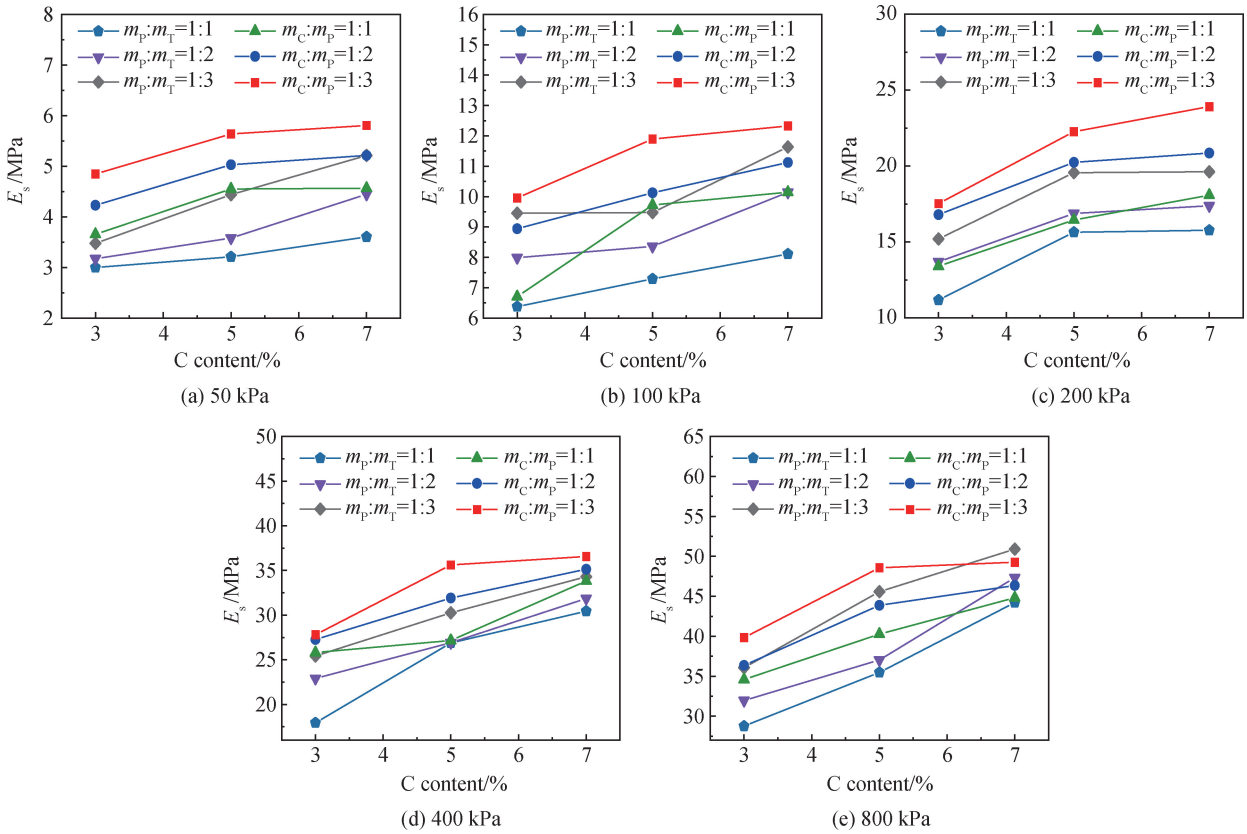
Cement content/%	m_P∶m_T	a_v/MPa^{-1}	Compressibility	Cement content/%	m_C∶m_P	a_v/MPa^{-1}	Compressibility
0% (plain red clay)	—	0.143	Middle	0% (plain red clay)	—	—	—
3	1∶1	0.126	Middle	3	1∶1	0.107	Middle
3	1∶2	0.158	Middle	3	1∶2	0.130	Middle
3	1∶3	0.139	Middle	3	1∶3	0.124	Middle

续表

Cement content/%	m_P∶m_T	a_v/MPa^{-1}	Compressibility	Cement content/%	m_C∶m_P	a_v/MPa^{-1}	Compressibility
5	1∶1	0.087	Middle	5	1∶1	0.086	Low
5	1∶2	0.128	Middle	5	1∶2	0.110	Middle
5	1∶3	0.111	Middle	5	1∶3	0.093	Low
7	1∶1	0.086	Low	7	1∶1	0.079	Low
7	1∶2	0.121	Middle	7	1∶2	0.104	Middle
7	1∶3	0.112	Middle	7	1∶3	0.096	Low

2.2 压缩模量与水泥掺量的关系

2 。 2 ,
, 。 200 kPa m_C∶m_P = 1∶1 , 3% ,
13.41 MPa, 7% , 18.08 MPa, 34.82%。 CaO、
SiO_2 , (C-S-H) 、 (C-A-H) [17]。
, , , ,
。

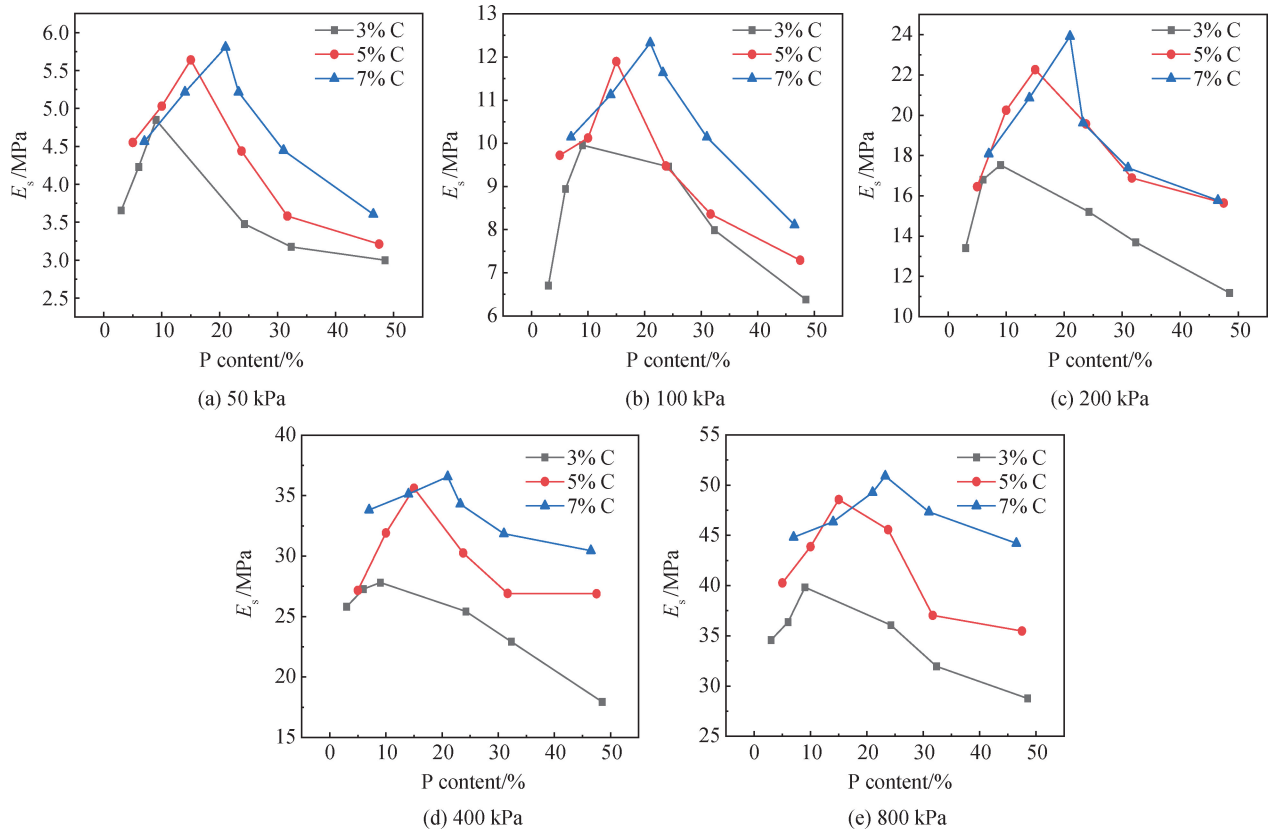


2

Fig. 2 Relationship between compressive modulus and cement content under different vertical loads

2.3 压缩模量与磷石膏掺量的关系

3 。 3 , ,
, , m_C∶m_P = 1∶3 。 200 kPa 5%
,m_C∶m_P = 1∶1 16.45 MPa,m_P∶m_T = 1∶1 15.64 MPa, m_C∶m_P = 1∶3
22.26 MPa, m_C∶m_P = 1∶1 35.32%, m_P∶m_T = 1∶1 42.33%。



3

Fig. 3 Relationship between compressive modulus and phosphogypsum content under different vertical loads

2.4 各因素关联度分析

,

, 。 ,

, [18] 。

$X_i=(x_i(1),\cdots,x_i(n))$ X_i 。 $x_i(k)$ 。

, (5)、(6) , , 。

$$r(x_0(k),x_i(k))=\frac{\min_i\min_k|x_0(k)-x_i(k)|+\rho\max_i\max_k|x_0(k)-x_i(k)|}{|x_0(k)-x_i(k)|+\rho\max_i\max_k|x_0(k)-x_i(k)|} \tag{5}$$

$$r(X_0,X_i)=\frac{1}{n}\sum_{k=1}^{n}r(x_0(k),x_i(k)) \tag{6}$$

:r ;$\min_i\min_k|x_0(k)-x_i(k)|$;$\max_i\max_k|x_0(k)-x_i(k)|$

;ρ , , 0.5。

、 、 , 、 、

, 8 , 、 、 90 。

。

(6), , 9 。

, 。 , , 2

3 , 。 800 kPa , 3% 7% ,

9.99 ~ 15.45 MPa, 53.72%。 3% 47.5% ,

5.05 ~ 13.08 MPa, 36.64%。

表 8　各参考序列与压缩模量的关联系数

Table 8　Correlation coefficient between each reference sequence and compressive modulus

Cement content/%	Phosphogypsum content/%	Vertical load/kPa	Compression modulus/MPa	Correlation coefficient of cement content	Correlation coefficient of phosphogypsum content	Correlation coefficient of vertical load
3	3	800	34.584	0.482	0.396	0.538
5	10	800	43.876	0.469	0.375	0.707
7	21	800	49.269	0.501	0.410	0.867
3	6	400	27.271	0.581	0.489	0.955
5	15	400	35.613	0.577	0.488	0.691
7	46.5	400	30.446	0.915	0.630	0.834
3	24.25	200	15.204	0.876	0.747	0.911
5	31.67	200	16.885	0.858	0.631	0.848
7	23.25	200	19.626	0.702	0.924	0.763
3	3	100	6.705	0.792	0.841	0.995
5	15	100	12.327	0.722	0.936	0.784
7	31	100	10.143	0.532	0.532	0.855
3	6	50	4.229	0.724	0.944	0.958
5	47.5	50	3.211	0.549	0.340	0.999
7	23.25	50	5.214	0.473	0.563	0.917

表 9　压缩模量与各因素的关联度

Table 9　Correlation between compressive modulus and various factors

Evaluation item	Correlation	Priority ordered
Vertical load	0.836	1
Cement content	0.661	2
Phosphogypsum content	0.621	3

2.5　压缩模量的非线性拟合

2　3　，　、

，　，　4　10，　(7)。

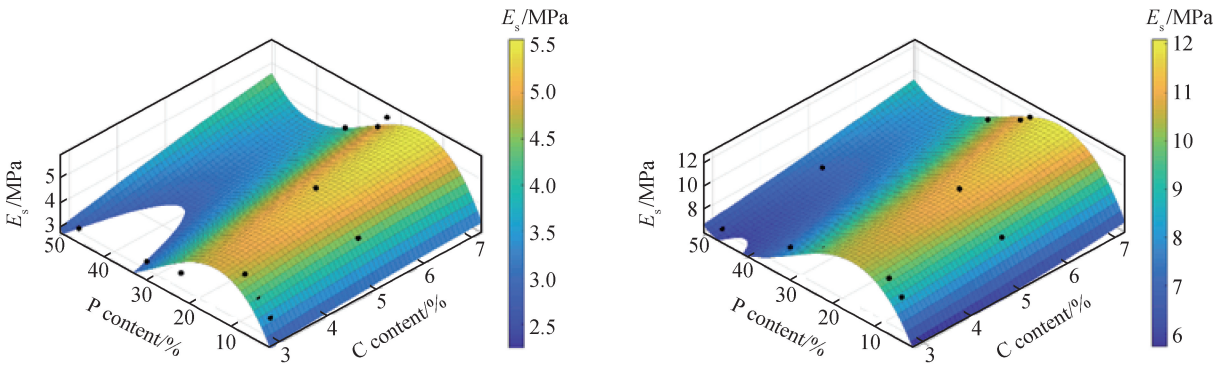
$$f(x,y)=a_1y^3+a_2(y+bx)^2+a_3y+a_4x^2+a_5x+c \tag{7}$$

:x　;y　;$f(x,y)$　;a,b,c　。

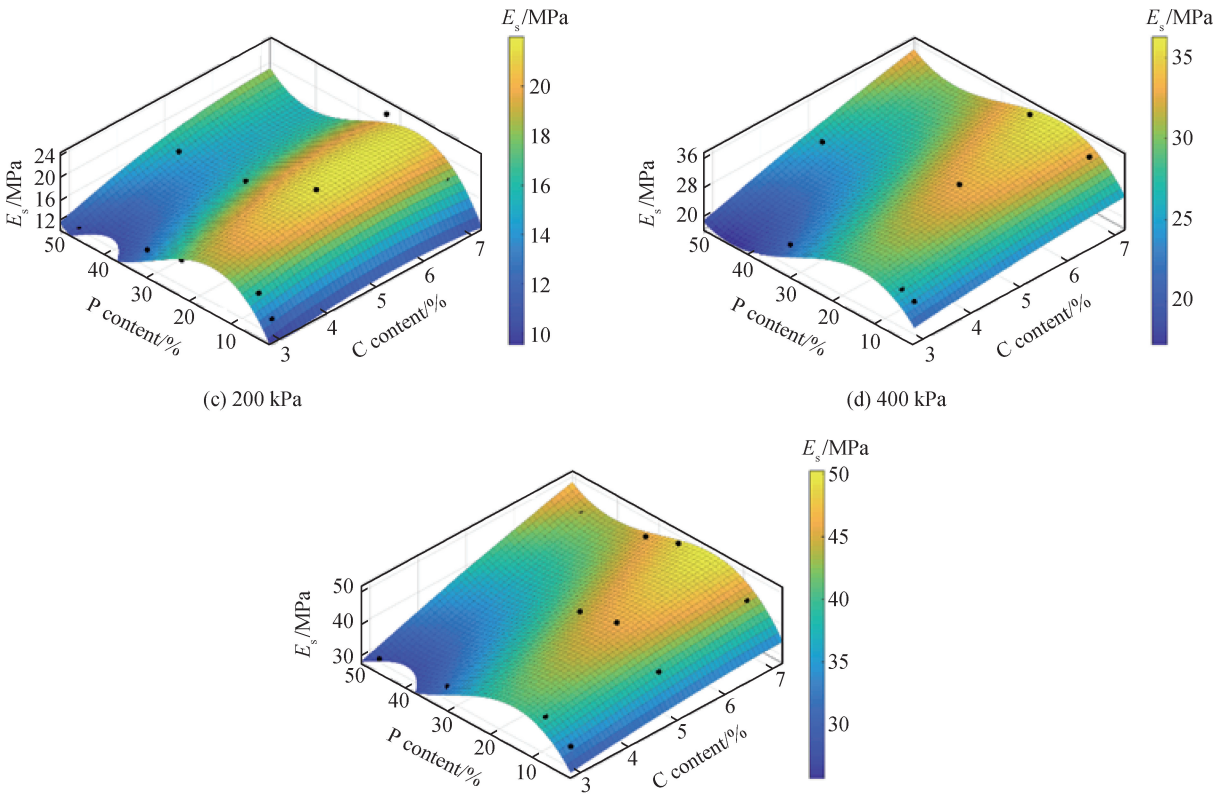
R^2　，　$R^2\geqslant 0.9$　。　10　，　R^2　0.9。

，　4　，　，　，

，　。



(a) 50 kPa　　(b) 100 kPa



(c) 200 kPa

(d) 400 kPa

(e) 800 kPa

4

Fig. 4 Compression modulus sufrace fitting

表 10 待定系数与拟合效果

Table 10 Undetermined coefficients and fitting effects

Vertical load/kPa	50	100	200	400	800
a_1	217.2×10^{-6}	379.0×10^{-6}	805.0×10^{-6}	810.2×10^{-6}	103.6×10^{-5}
a_2	-0.182 2	-0.034 0	-0.069 8	-0.069 7	-0.091 3
a_3	0.358 9	0.763 0	1.454 0	1.284 0	1.754 0
a_4	-0.016 3	-0.022 7	-0.319 4	-0.114 8	-0.159 2
a_5	0.256 2	0.045 1	3044 6	2.147 0	2.808 0
b	-0.143 7	-0.084 9	-0.190 2	-0.349 9	-0.389 7
c	2.006	4.886 0	1.657 0	15.570 0	21.790 0
R^2	0.897 9	0.916 6	0.916 3	0.946 1	0.946 3

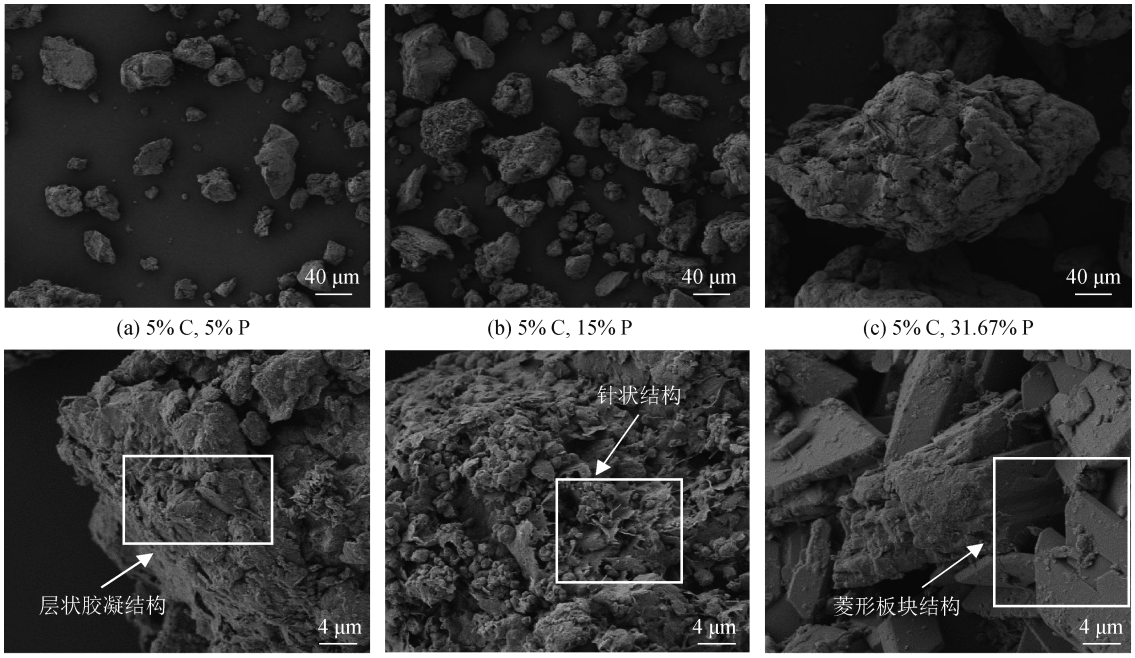
2.6 机理分析

, $m_C : m_P = 1:3$ 。

5%, SEM XRD 。SEM 5, 11。

:1) ;2) ,

。 , 。



(a) 5% C, 5% P (b) 5% C, 15% P (c) 5% C, 31.67% P

(d) 5% C, 5% P (e) 5% C, 15% P (f) 5% C, 31.67% P

5 SEM

Fig. 5 SEM images of mixtures with different phosphogypsum content

表 11 不同磷石膏掺量下矿物含量

Table 11 Mineral content under different phosphogypsum content

Phosphogypsum content/%	Mass fraction/%						
	SiO_2	$CaCO_3$	$(K,Na)Al_2(Si,Al)_4O_{10}(OH)_2$	$Al_2(OH)_4Si_2O_5$	$CaSO_4 \cdot 2H_2O$	$FeO \cdot OH$	$3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$
0	42.8	—	29.7	18.7	—	8.8	—
5	39.1	3.2	30.7	—	13.2	13.8	—
10	30.2	2.4	17.1	17.0	23.2	8.5	1.6
15	28.3	2.4	21.6	4.3	33.2	8.4	1.8
23.8	23.2	0.8	14.1	15.6	40.0	5.3	1.0
31.7	14.5	2.0	14.2	9.1	56.2	4.0	—
47.5	4.9	—	3.9	3.0	87.5	0.8	—

2.6.1

, , , 。

, , 。 , ,

。

5 :5% -5% , 10 ~ 30 μm;5% -15%

, 10 ~ 80 μm;5% -31.67% , 200 μm 。

, ; , , ;

, 。

。 , $CaSO_4 \cdot 2H_2O$,

SiO_2、 $Al_2(OH)_4Si_2O_5$, ,

[19]。 ,

。

: , Ca^{2+} ,

,Ca^{2+} , 。

, Capotosto [20] $CaCl_2$, Ca^{2+} 。
: SiO_2、 $Al_2(OH)_4Si_2O_5$ 。
, H^+ Ca^{2+}, 。 (8) ~ (10)。

$$\equiv SiO^- + H^+ \longrightarrow \equiv SiOH \tag{8}$$

$$> AlO^- + H^+ \longrightarrow > AlOH \tag{9}$$

$$2 > AlOH + Ca^{2+} \longrightarrow \begin{matrix} > Al\text{-}O \\ > Al\text{-}O \end{matrix} > Ca^{2+} + 2H^+ \tag{10}$$

, ,Ca^{2+}
。 , 。 , 。
, 。 $m_C : m_P = 1:3$, - - 、
、 、 。 , , ,
, 。

2.6.2

, 。 ,
, , , 。 ,
。 , :
C-S-H : , , (C-S-H) (
(11))。C-S-H , , ,
。

$$3CaO \cdot SiO_2 + nH_2O \longrightarrow xCaO \cdot SiO_2 \cdot (n-3+x)H_2O + (3-x)Ca(OH)_2 \tag{11}$$

: $CaSO_4 \cdot 2H_2O$ $Ca(OH)_2$、$3CaO \cdot Al_2O_3$
, (12)、(13)。

$$3Ca(OH)_2 + Al_2O_3 + (n-3)H_2O \longrightarrow 3CaAl_2O_3 \cdot nH_2O \tag{12}$$

$$3CaO \cdot Al_2O_3 + 3(CaSO_4 \cdot 2H_2O) + 2Ca(OH)_2 + 24H_2O \longrightarrow 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O \tag{13}$$

C-S-H , 。 11 ,
, , $m_C : m_P = 1:3$, 。
, pH , , [21-23], (14)。
, , 。

$$3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O + 3CO_2 \longrightarrow 3CaCO_3 + 3(CaSO_4 \cdot 2H_2O) + Al_2O_3 \cdot xH_2O + (26-x)H_2O \tag{14}$$

Ca^{2+} : ,
Ca^{2+} , K^+、Na^+、Fe^{3+} , [24]。
, Fe_2O_3、 FeO(OH)、 γ-FeO(OH), ,
[25]。 11 , ,Fe^{3+} ,
Ca^{2+} Fe^{3+}。 , 。
: , 。
, [26]。
, , ,
, 。 , ,
, ,
。 $m_C : m_P = 1:3$, , , ,
, , , 。

3 结 论

1) , 、 。 ,

, 1∶3 。

2) > > 。

3) 、 $f(x,y)=a_1y^3+a_2(y+bx)^2+a_3y+a_4x^2+a_5x+c$。 、

。

4) , 、 ,

, 1∶3 , , 。 ,

, , , , 。

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