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Research Article

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Experimental research on the physical and mechanical properties and freeze-thaw resistant performance of rice straw-mortar composite materials

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Abstract

Straw has relatively high tensile strength, as a kind of aggregate, combined with cement, gypsum and other bonding materials, which can improve the strength of the benchmark materials and control the initiation and propagation of microcracks. The physical and mechanical properties of straw-mortar composite materials are determined by the property of straw itself, the bonding capacity of matrix materials, the environment and other factors. In this study, three-factor and three-level regression analysis experiment was carried out by using Design-Expert software to obtain the optimal physical and mechanical properties and mix ratio of rice straw-mortar composite materials. The highest compressive strength, dry density and the lowest water absorption rate of rice straw-mortar composite materials were 3.06 MPa, 1.340 $g \cdot cm^{-3}$ and 20.8%, respectively, and the corresponding gypsum content, straw length, and straw content were 15%, 20 mm, and 15%, respectively. After 15 times freeze-thaw (F-T) between -20 ℃ and 20 ℃, the strength loss rate, mass loss rate and water absorption rate of rice straw-mortar composites produced in different conditions varied from 32.2% to 47.3%, from 2.1% to 4.5%, and from 22.6% to 42.2%, respectively. The above experimental results indicated that the rice straw-mortar composite materials could meet the actual using demand of self-bearing wall whether to experience F-T, which laid a certain foundation for the popularization and application of rice straw-mortar composite materials in non-bearing wall.

Keywords: Rice straw-mortar composite materials, Physical and mechanical properties, Freeze-thaw resistant performance, Influencing factors

1 Introduction

As a major grain producing country, China's total annual crop straw is 1.04 billion tons and the collectable and available quantity of straw resources are about 900 million tons [1]. The traditional method of crop straw treatment is mainly burning in the field, which will result in waste of resources and affect the normal traffic, human health and atmospheric environment for the smoke and dust generated by the combustion of straw [2,3]. Therefore, people has made comprehensive utilization of straw resources, and the main utilization methods are feed, base material, energy, fertilizer and raw material [4,5].

Compared with traditional cement brick walls, straw brick walls bundled with straw saved about 40% of the total construction cost [6]. The grass-brick wall has many advantages, such as, lightweight, earthquake-resistant, and energy-saving, but it is always has not enough bearing loads capacity. Fu et al. [7] finds that the maximum compressive strength of cotton straw bricks is 0.34 MPa. The bearing capacity of the straw brick lime wall is about 30% of the lime soil wall with same thickness [8]. By adjusting the ratio of straw and other materials, the strength of straw composite wall materials can be improved. Zhang [9] produces type I straw block with compressive strength of 0.1 MPa, and the original material mass ratio of straw, cement and sand is 1 to 9 to 24. The back wall of the greenhouse built with this kind of blocks can bear the weight of greenhouse structure and loads of rain and snow. Gonzalez [10] obtains that family residential buildings with exterior walls which made of gypsum straw-clay mortar can meet normal load-bearing capacity. The straw composite materials wall can withstand an earthquake of magnitude 8.5 or above and has obvious improvement in thermal insulation [11,12]. Presently, straw composite materials are mainly used to construct buildings and roads [13]. The compressive strength of straw composite materials decreases with the increase of plant fiber additive amount. It is very important to obtain the straw composite materials with good properties for its practical utilization.

In this study, the physical and mechanical properties of rice straw-mortar composite materials, such as, compressive strength, dry density and water absorption rate are improved by adjusting the influencing factors such as gypsum content, straw length and straw content. Meanwhile, winter temperature in north of China is relatively low, and the average temperature of most area is as low as -20 ℃. Because of composite materials contain a lot of liquid and gas water, repeated freeze-thaw (F-T) action will lead to strength loss and structural damage of straw composite materials. Therefore, it is meaningful to study the action rules of various influencing factors on the physical and mechanical properties indexes of rice straw-mortar composites, reach the optimal values of relevant indexes and corresponding influencing factors, and analyze the F-T action mechanism of the composite materials from a macro perspective for the application and promotion of straw-mortar composite materials as building wall blocks in north of China.

2 Materials and methods

2.1 Experimental materials

According to "Method of Testing Cements-Determination of Strength" (GB/T17671-2020), the mortar ratio of rice straw-mortar composite materials was determined as M (cement): M (sand) = 1: 3 to ensure that the composite materials meet the strength requirements.

2.1.1 Portland cement

The cement used in this experiment was ordinary Portland cement produced by Shenyang Sunnsy Gongyuan Cement Co, Ltd. The physical and mechanical properties of cement are shown in Table 1.

Table 1 Physical and mechanical properties of Portland cement used in the experiment

Specific surface area	Density	Setting time (min)		Compressive strength (MPa)	
($cm^2 \cdot g^{-1}$)	($g \cdot cm^{-3}$)	Initial setting time	Final setting time	3d	28d
3245	3.15	183	237	25.4	49.5

2.1.2 Sand

The fine aggregate used in the experiment was natural river sand, which was taken from the Hun River in Liaoning Province.

2.1.3 Gypsum

Yao [14] found that when the mixture ratio of gypsum and cement was 1 to 9, the cement gypsum based cotton straw polystyrene composite block had the highest density and high compressive strength. Therefore, gypsum content (the mass ratio of gypsum to cement) selected for this experiment was 5%, 10% and 15%.

The gypsum was white powder produced by Liaoning 303 Decoration Material Co., Ltd. The density of gypsum was about 2.60-2.75 $g \cdot cm^{-3}$, and the packing dry density was 0.8-1.1 $g \cdot cm^{-3}$. The main physical and mechanical properties of gypsum are shown in Table 2.

Table 2 Gypsum specification

Technical specification		Values
Strength (MPa)	Flexural strength	≥1.8
	Compressive strength	≥2.9
Fineness (%)	0.2 mm square sieve residue	≤15.0
Coagulation time (min)	Initial setting time ≥, Final setting time ≤	6, 30

2.1.4 Rice straw

Benmansour et al. [15] concluded that fiber cement mortar composite materials had higher compressive strength when jujube fiber content was 5%-15%. Feng [16] obtained that the straw cement composite mortar had better performance when the wheat straw content was 10%-20%. Therefore, the rice straw content (the mass ratio of rice straw to cement) was selected as 15%, 20% and 25% in this study. Jian et al. [17] found that the straw cement composite materials had good mechanical properties when the length of rice straw was 20-30 mm. Wang et al. [18] believed that the straw cement composite materials with fiber length of 5-10 mm had the highest strength of 5.5 MPa. In this study, three test levels of rice straw length were selected as 10, 15 and 20 mm.

The rice straw ‘Shennong 511’ was gathered from the rice field in Shenyang Agricultural University, Liaoning China (123°33′51″ E, 41°49′8″ N) in October 2019. The plant height was about 110 cm. Firstly, exposed rice straw to the sun to control the moisture content no more than 10%. Then, chose rice straw which was not rotten and moldy, and removed the excess roots and ears. Finally, cut straw samples into strips of 10, 15 and 20 mm, and put them into moisture-proof bags.

2.1.5 Water

Under the same water-cement ratio condition, with the increase of straw length and content, the fluidity of slurry and adhesion between aggregates became poor, which was not conducive to the formation of slurry. Oppositely, the fluidity of slurry was stronger and the slurry was thinner, which led to small holes on the surface of testing block after drying. In order to ensure a good water-cement ratio, the addition of water could be controlled to mix the fluidity of mortar during the process of preparing mortar. The experimental water was ordinary tap water.

2.2 Experimental design

In this study, Design-Expert software was used to design 17 sets of three-factor and three-levelBox-Behnken central combination experiments, which were carried out at room temperature with different levels of influencing factors. Main influencing factors and coded factor levels of independent variables are given in Table 3. The influence of various factors on the compressive strength, dry density and water absorption rate were investigated. The optimal values of the related physical and mechanical properties of rice straw-mortar composite materials and the values of each factor were determined.

Table 3 Coded levels for independent variables used in the experiment

Factors / Levels	x_1 (%)	x_2 (mm)	x_3 (%)
1	15	20	25
0	10	15	20
-1	5	10	15
Δ_j	5	5	5

Where, x_1 is gypsum content (%); x_2 is straw length (mm); x_3 is straw content (%).

The regression equation of each target value was calculated according to the formula (1):

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 \quad (1)$$

Where, x_1 is gypsum content, % (the mass ratio of gypsum to cement); x_2 is straw length, mm; x_3 is straw content, % (the mass ratio of rice straw to cement); y is predicted target value of each factor level combination; b_0 is constant term; b_1, b_2, and b_3 are linear influence coefficients; b_{12}, b_{13}, and b_{23} are linear interaction influence coefficients; b_{11}, b_{22}, and b_{33} are quadratic regression coefficients.

Meantime, carrying out F-T experiments to determine the mass and compressive strength of testing blocks after 5, 10, and 15 times F-T, then compared the testing blocks before and after F-T to obtain the compressive strength and mass loss rate. Analyzing the influence of freeze-thaw cycles (FTC) on water absorption rate of composite blocks, which was related to block's compression failure.

2.3 Experimental methods

2.3.1 Preparation of rice straw-mortar composite material samples

Firstly, poured the right amount of cement, rice straw, sand and gypsum into the mortar mixer. After dry mixing for 3 minutes, added water after stirring more than 5 minutes until the viscous slurry appeared. Then, the mixed mortar was injected into a mortar triplet mold in size of 70.7×70.7×70.7 mm. The mold was removed after standing 48 hours in a ventilated and dry room. Finally, the testing blocks were placed into the SJ-40A mortar curing box (above 90% relative humidity, 20±2 °C temperature) for 28 days.

2.3.2 Measurement of physical and mechanical property parameters

(1) Determination of compressive strength

Compressive strength is the most basic mechanical property of composite materials in the application process, which reflects the load capacity of materials. Compressive strength was determined according to "Standard for Test Method of Performance on Building Mortar" (JGJ/T70-2009). The average values of each set of three testing blocks were determined as the final compressive strength of the block. The compressive strength of testing block was calculated according to formula (2):

$$f_{m,cu} = 1.35\frac{N_u}{A} \quad (2)$$

Where, $f_{m,cu}$ is compressive strength of testing block, MPa; N_u is failure load of testing block, N; A is pressure bearing area of failure load of testing block, mm^2.

(2) Determination of dry density

28-day-old testing blocks were placed in electricity hot blast drying oven. Refer to experimental method of Dry Mix Thermal Insulating Composition for Buildings (GB/T20473-2006), adjusted the temperature in drying oven to 60±5 °C, and took out the specimen to observe and record its mass every 3 hours, until the mass change rate of the testing block weighed twice within 3 hours was less than 0.2%, it was considered that the block reached a constant quality, and then moved it out of oven and cooled in room temperature. The dry density of testing block was calculated according to formula (3):

$$\rho_0 = \frac{m_0}{V} \tag{3}$$

Where, ρ_0 is dry density of testing block, g·cm^{-3}; m_0 is constant mass of testing block after drying, g; V is volume of testing block after drying, m^3.

(3) Determination of water absorption rate

Placed 28-day-old testing block in water for 2 days and weighed its mass, then put it in a constant temperature drying oven at the temperature of 60±5 °C and recorded the data every 3 hours until it reached a constant mass. The water absorption rate of testing block was calculated according to formula (4):

$$w_1 = \frac{m_1 - m_0}{m_0} \times 100\% \tag{4}$$

Where, w_1 is water absorption rate of testing block in normal temperature condition, %; m_1 is mass of testing block after absorbing water for 2 days, g; m_0 is constant mass of testing block after drying, g.

2.3.3 Measurement of F-T resistant performance parameters

F-T resistant performance refers to the ability of saturated materials to resist destroy in condition of repeated F-T. Compressive strength loss rate and mass loss rate are two important indicators reflected the F-T resistant performance of materials. Quick freezing method was used to study the damage acted on the rice straw-mortar composite materials placed in the outdoor environment in winter. The experiment was referred to the relevant test methods of Dry Mix Thermal Insulating Composition for Buildings (GB/T20473-2006) and Standard for Test Method of Performance on Building Mortar (JGJ/T70-2009).

The compressive strength loss rate of testing block was calculated according to formula (5):

$$\Delta f_n = \frac{f_0 - f_n}{f_0} \times 100\% \tag{5}$$

Where, Δf_n is compressive strength loss rate of testing block after *n* times F-T, %; f_0 is compressive strength of testing block without F-T, MPa; f_n is compressive strength of testing block after *n* times F-T, MPa.

The mass loss rate of testing block was calculated according to formula (6):

$$\Delta m_n = \frac{m_0 - m_n}{m_0} \times 100\% \tag{6}$$

Where, Δm_n is mass loss rate of testing block after *n* times F-T, %; m_0 is constant mass of testing block after drying without F-T, g; m_n is drying mass of testing block after *n* times F-T, g.

The water absorption rate of testing block was calculated according to formula (7):

$$w_n = \frac{m_n' - m_0'}{m_0'} \times 100\% \tag{7}$$

Where, w_n is water absorption rate of testing block after *n* times F-T, %; m_n' is mass of testing block after *n* times F-T and absorbing water for 2 days, g; m_0' is constant mass of testing block after F-T and drying, g.

3 Experimental results and discussion to the composite materials tested in condition of normal temperature

In condition of normal temperature, the experimental results are shown in Table 4. Compressive strength ranged between 0.55 MPa and 3.0 MPa, the range of dry density was from 0.921 g·cm^{-3} to 1.549 g·cm^{-3}, and water absorption rate varied from 18.2% to 36.3%.

Table 4 Physical and mechanical properties of composite materials based on the Box-Behnken experimental design

Number	x_1 (%)	x_2 (mm)	x_3 (%)	y_f (MPa)	y_ρ (g·cm^{-3})	y_w (%)
1	15	20	20	2.60	1.206	28.6
2	15	10	20	2.35	1.394	23.5
3	10	15	20	1.90	1.432	25.3
4	10	15	20	2.17	1.473	24.9
5	5	10	20	1.65	1.375	22.6
6	5	20	20	2.00	1.329	23.1
7	5	15	25	0.55	1.075	34.2
8	10	20	15	3.00	1.549	18.2
9	10	10	25	0.85	1.256	26.8

10	15	15	15	2.53	1.331	21.8
11	10	20	25	2.15	1.130	32.4
12	10	10	15	2.70	14.87	20.2
13	10	15	20	2.10	1.452	25.9
14	10	15	20	1.85	1.446	26.7
15	15	15	25	0.95	0.921	36.3
16	5	15	15	1.10	1.418	22.6
17	10	15	20	1.97	1.369	25.3

Where, x_1 is gypsum content (%); x_2 is straw length (mm); x_3 is straw content (%); y_f is compressive strength in normal temperature condition (MPa); y_ρ is dry density in normal temperature condition (g·cm^{-3}); y_w is water absorption rate in normal temperature condition (%).

3.1 Modeling and analysis of variance (ANOVA)

Used Design-Expert software to perform regression analysis on the data of Table 5, and fitted regression equations with gypsum content, straw length, and straw content as independent variables, compressive strength, dry density and water absorption rate as dependent variables. R^2_{Adj} is an important coefficient standard for checking the reliability and accuracy of the regression model. The value is closer to 1, and the model fits better.

Table 5 Regression equations for physical and mechanical properties of rice straw-mortar composite materials

Physical and mechanical properties	Response surface model	R^2_{Adj}
Compressive strength	$y_f = 1.29075 + 0.59545x_1 - 0.7617x_2 + 0.38465x_3 - 0.0103x_1x_3$ $+0.01x_2x_3 - 0.01481x_1^2 + 0.02089x_2^2 - 0.01381x_3^2$	0.919
Dry density	$y_\rho = -607.775 + 137.135x_1 + 7.84x_2 + 174.745x_3$ $-5.553x_1^2 - 4.373x_3^2$	0.930
Water absorption rate	$y_w = 38.3175 - 2.3395x_1 + 1.547x_2 - 2.7015x_3 + 0.076x_2x_3$ $+0.0631x_1^2 - 0.1099x_2^2 + 0.0611x_3^2$	0.944

Where, x_1 is gypsum content (%); x_2 is straw length (mm); x_3 is straw content (%); y_f is compressive strength in normal temperature condition (MPa); y_ρ is dry density in normal temperature condition (g·cm^{-3}); y_w is water absorption rate in normal temperature condition (%).

Variance analysis results of the regression equations in Table 5 are shown in Table 6. *P* values and lack of fit values of fitting models were less than 0.01 and more than 0.05, respectively, which indicated that the regression analysis reached significant level and the model was high reliability. In terms of compressive strength, gypsum content, straw length, and straw content had extremely significant effects; the interaction between gypsum content and straw content, as well as the interaction between straw length and straw content had significant effects. For dry density, gypsum content and straw content had extremely significant effects, straw length had significant effects. In terms of water absorption rate, straw length and straw content had extremely significant effects; gypsum content and interaction terms straw length and straw content had significant effects.

Table 6 Coefficient values and variance analysis of the fitting model for different responses of composite materials

Response	Source	*df*	Sum of squares	Mean squares	*F* value	*P* value	Significance
Compressive strength	Model	9	7.39	0.82	21.05	0.0003	**
	x_1	1	1.22	1.22	31.40	0.0008	**
	x_2	1	0.60	0.60	15.51	0.0056	**
	x_3	1	2.92	2.92	74.77	<0.0001	**
	x_1x_2	1	2.5×10^{-3}	2.5×10^{-3}	0.064	0.8074	
	x_1x_3	1	0.27	0.27	6.80	0.0350	*
	x_2x_3	1	0.25	0.25	6.41	0.0391	*
	x_1^2	1	0.58	0.58	14.80	0.0063	**
	x_2^2	1	1.15	1.15	29.45	0.0010	**
	x_3^2	1	0.50	0.50	12.87	0.0089	**
	Lack of fit	3	0.20	0.067	3.70	0.1192	
Dry density	Model	9	4.268×10^{5}	47420.33	24.58	0.0002	**
	x_1	1	14878.12	14878.12	7.71	0.0274	*
	x_2	1	11100.5	11100.5	5.75	0.0475	*
	x_3	1	2.461×10^{5}	2.461×10^{5}	127.56	<0.0001	**
	x_1x_2	1	5041	5041	2.61	0.15	
	x_1x_3	1	1122.25	1122.25	0.58	0.4705	
	x_2x_3	1	8836	8836	4.58	0.0696	
	x_1^2	1	81146.87	81146.87	42.07	0.0003	**

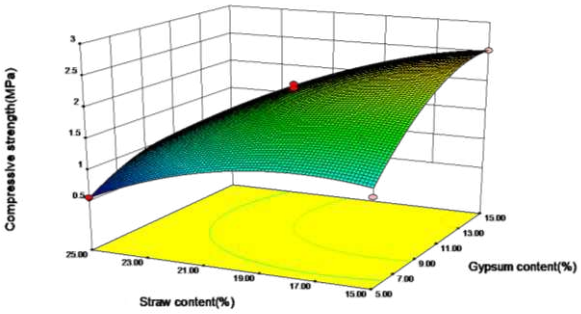
	x_2^2	1	3897.6	3897.6	2.02	0.1982	
	x_3^2	1	50324.02	50324.02	26.09	0.0014	**
	Lack of fit	3	7284.75	2428.25	1.56	0.33	
Water absorption rate	Model	9	364.29	40.48	30.7	<0.0001	**
	x_1	1	7.41	7.41	5.62	0.0496	*
	x_2	1	10.58	10.58	8.02	0.0253	*
	x_3	1	274.95	274.95	208.51	<0.0001	**
	x_1x_2	1	5.29	5.29	4.01	0.0853	
	x_1x_3	1	2.1	2.1	1.59	0.2471	
	x_2x_3	1	14.44	14.44	10.95	0.0130	*
	x_1^2	1	10.48	10.48	7.95	0.0258	*
	x_2^2	1	31.78	31.78	24.10	0.0017	**
	x_3^2	1	9.82	9.82	7.45	0.0294	*
	Lack of fit	3	7.26	2.42	4.92	0.0789	

Where, x_1 is gypsum content (%); x_2 is straw length (mm); x_3 is straw content (%); "**" means extremely significant difference at P<0.01 level; "*" means generally significant difference at P<0.05 level.

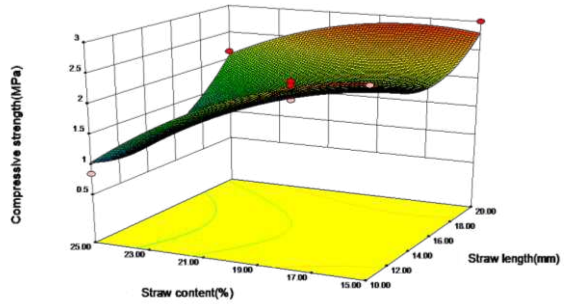
3.2 Influence of interaction of various factors on the physical and mechanical properties

Based on the results of Tables 5 and 6, interaction effects of independent variables on all responses of composite materials generated by Design Expert software are shown in Figs. 1, 2 and 3.

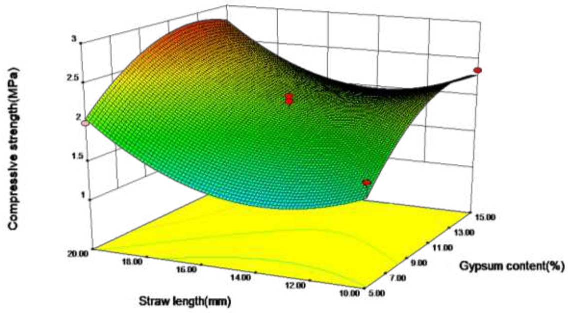
It could be seen from Fig. 1 that compressive strength of the composite materials increased with the increase of gypsum content and straw length, or decrease of straw content. The slope of the curved surface was most obviously affected by straw content. Festugato et al. [19] found that compressive strength of fiber-reinforced soil mixture increased linearly with the increase of fiber length. Zeng et al. [20] studied the rape straw concrete testing block in the size of 100×100×100 mm, with the fiber length increased from 10-20 mm to 20-30 mm, its compressive strength was continuously improved. The compressive strength of the composite materials decreased with the increase of straw content addition [21]. These were consistent with the results of this study. Increase of gypsum content caused the straw with "dissolution phenomenon" on surface to be filled by the gypsum matrix, which improved the bonding between the interfaces and the expansion, and was effect to improve the mechanical properties. Because the fiber length range of rice straw was smaller than the size of testing block, the rice fiber was used as aggregate component, which could be evenly dispersed in cement paste by fully mixing. With the increase of straw content, the cement paste component played the main mechanical role in the testing block decreased. In addition, the smooth waxy layer on the surface of the straw made the adhesion between the straw and the cement matrix worse, retarding phenomenon appeared after the straw was mixed into the basic matrix which would lead to reduction of strength.



$f(x_1, 15, x_3)$



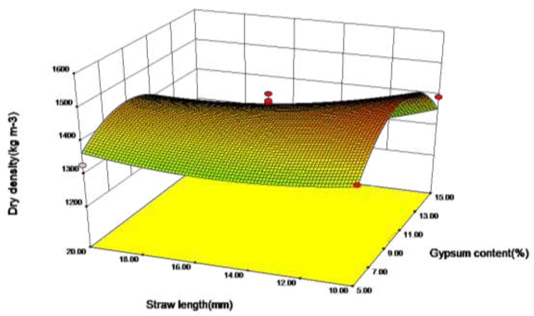
$f(10, x_2, x_3)$



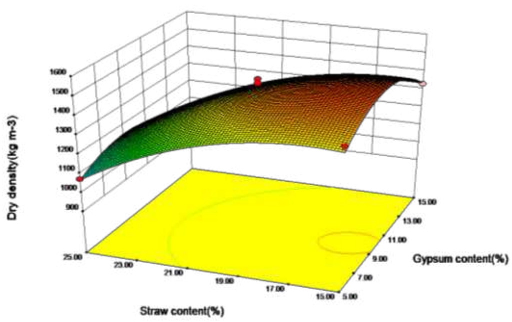
$f(x_1, x_2, 20)$

Fig. 1 Influence of interaction factors on compressive strength

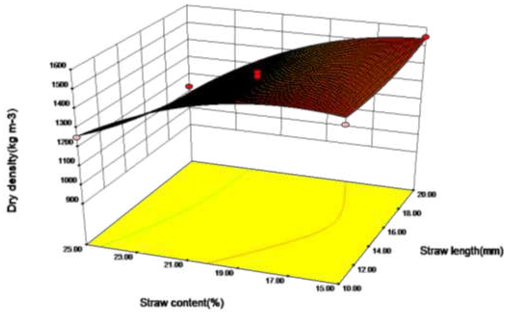
It could be seen from Fig. 2 that dry density firstly increased and then decreased with the increase of gypsum content, and decreased smoothly with the increase of straw content and length. The reason for this phenomenon may be the light weight characteristics of the building gypsum, and the increase of straw length and straw content caused the straw volume increased in composite materials, which led to the dry density of the composite materials kept decreasing [22].



$f(x_1, x_2, 20)$



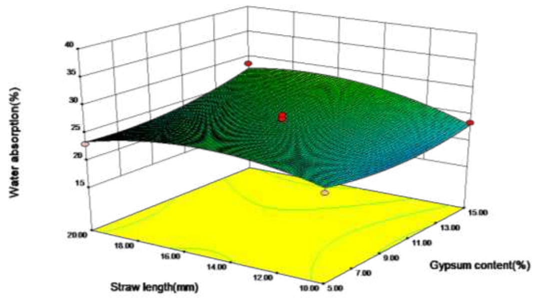
$f(x_1, 15, x_3)$



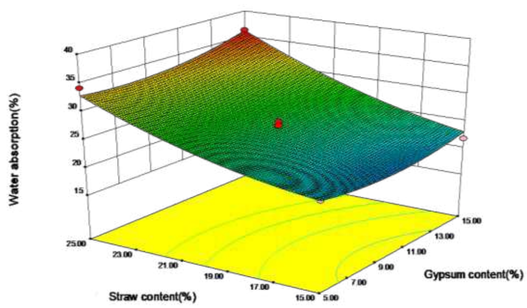
$f(10, x_2, x_3)$

Fig. 2 Influence of interaction factors on dry density

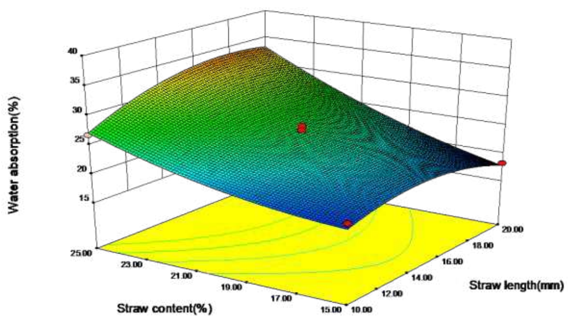
It could be seen from Fig. 3 that water absorption rate increased with the increase of gypsum content, straw length and content. Migneault et al. [23] found that water absorption rate of the composite materials increased when the straw size aspect ratio gradually increased from 8.3 to 21.3 mm. With the increase of straw content, the water absorption rate gradually increased [24]. The reason for this phenomenon may be that the water consumption in mixing process of gypsum and cement was greater than theoretical value, and after the excess water evaporated, the water seepage channel was formed inside the composite materials, which increased the water absorption rate. Meantime, the porous structure of straw led the volume increase for the void and pore inside the composite materials, which was benefit for water absorption of composite materials.



$f(x_1, x_2, 20)$



$f(x_1, 15, x_3)$



$f(10, x_2, x_3)$

Fig. 3 Influence of interaction factors on water absorption rate

3.3 Optimization and model validation

The optimized goals are shown in Table 7. The compressive strength can reflect the load-bearing capacity of materials, which should be maximized. High compressive strength of composite materials is the most important mechanical performance (response variable), as it is a good indicator of the mechanical resistance of composite materials to the forces during working process. It is followed by water absorption rate, because a low value of water absorption rate indicates good waterproof and the resistant ability of F-T destroy. Low values of dry density are also desirable properties in terms of decreasing the weight of building wall and bearing requirement of foundation. For the goals related to variables, the gypsum content, straw length and straw content were placed “in range”.

Table 7 Optimization goal of variables during the experimental production rice straw-mortar composite materials

Variable	Goal	Level of importance
Independent		
Gypsum content (%)	In range (5-15)	
Straw length (mm)	In range (10-20)	
Straw content (%)	In range (15-25)	
Response		
Compressive strength (MPa)	Maximize	First
Dry density ($g \cdot cm^{-3}$)	Minimize	Third
Water absorption rate (%)	Minimize	Second

According to Design Expert software analysis, the best optimization of materials is shown in Table 8. The composite materials produced with m (cement): m (sand) = 1: 3, 15% gypsum content, 20 mm of straw length, and 15% straw content were chosen to be the best set of condition due to the high compressive strength of 3.06 MPa, low dry density of 1.340 $g \cdot cm^{-3}$, and low water absorption rate of 20.8%.

Table 8 Optimum conditions for producing rice straw-mortar composite materials

Gypsum content (%)	Straw length (mm)	Straw content (%)	Compressive strength (MPa)	Dry density ($g \cdot cm^{-3}$)	Water absorption rate (%)
15	20	15	3.06	1.340	20.8

4 Experimental results and discussion to the composite materials after FTC

4.1 Performance parameters after FTC

According to the experimental results of Table 4, the quick freezing method was selected to simulate the outdoor F-T environment in winter, discussed the influence of winter rain and snow erosion and the frost heave damage on the rice straw-mortar composite materials. All 17 sets of experimental results after 5, 10 and 15 times F-T were tested in turn, and the physical and mechanical properties of testing blocks in Table 4 were used as the comparison values to calculate the compressive strength loss rate and mass loss rate of the testing blocks, respectively.

4.1.1 Compressive strength loss rate

After 5, 10 and 15 times F-T, the compressive strength loss rates of 17 sets of testing blocks were calculated, and the testing results are shown in Fig. 4.

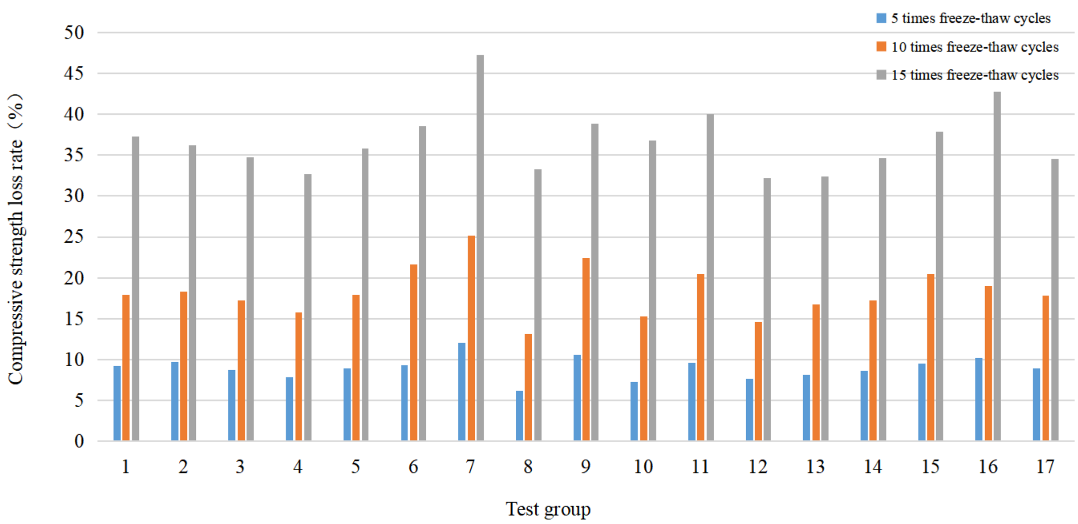


Fig. 4 The compressive strength loss rate of testing blocks after FTC

After FTC, compressive strength loss rate of the composite materials would occur [25]. In this study, compressive strength loss rate of all 17 sets composite material blocks increased with the increase of F-T times. Li [26], Kim et al. [27] and Richardson et al. [28] obtained the consistent research conclusions. After 5th, 10th, and 15th F-T, the compressive strength loss rate varied from 6.2% to 12.1%, 13.11% to 25.2%, and 32.2% to 47.3%, respectively. The compressive strength of straw mortar composite testing block was tested after 15 times F-T, and the maximum compressive strength of the testing block was 2.01 MPa. Although the strength loss rate of the testing block was more than 25%, which meant that the frost resistance of rice straw-mortar composite materials was not good according to the regulation of “Dry Mix Thermal Insulating Composition for Buildings” (GB/T20473 -2006). But the testing block after F-T still retained a certain strength, which could be applied to non-bearing walls and partitions [29].

The fundamental reason for the F-T failure of the testing block was that the internal pore stress of the materials exceeded its limit value, which led to material deformation and cracking (Walker, 2014). After F-T, the original moisture and water vapor inside the testing block condensed into ice, which made the testing block appear relatively small cracks. The existence of straw increased the porosity of the composite testing block, provided more developed water vapor channel and water storage space, improved the moisture content, and intensified the generation of cracks. Under the action of FTC, the original small cracks continued to expand and connect with each other, which made the mortar or paste fall off, resulting in a significant reduction in the mechanical properties of the composite materials. Comparing testing results of the compressive strength loss rate of the 7th and 16th sets, the 9th and 12th sets, and the 10th and 15th sets composite materials, we found that the composite materials with more straw content had a larger compressive strength loss rate (as shown in Fig. 5).

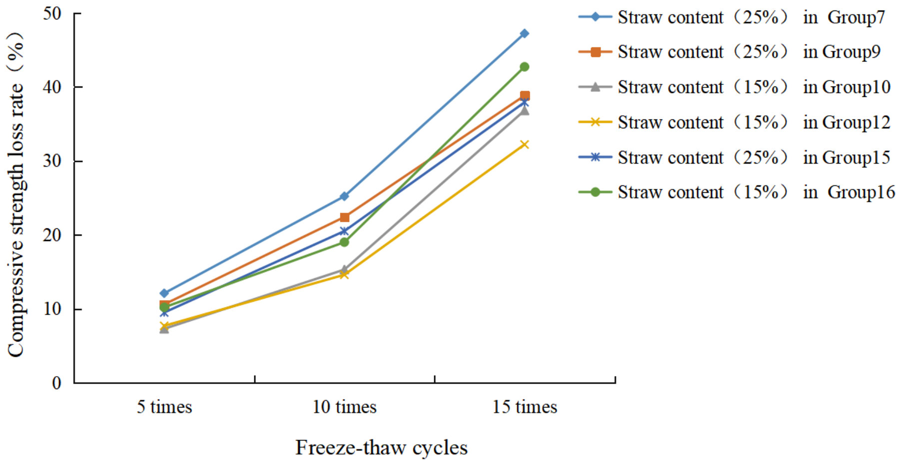


Fig. 5 Effect of straw content on compressive strength loss rate under FTC

4.1.2 Mass loss rate

After 5, 10 and 15 times F-T, the mass loss rates of 17 sets testing blocks were calculated, and the testing results are shown in Fig. 6.

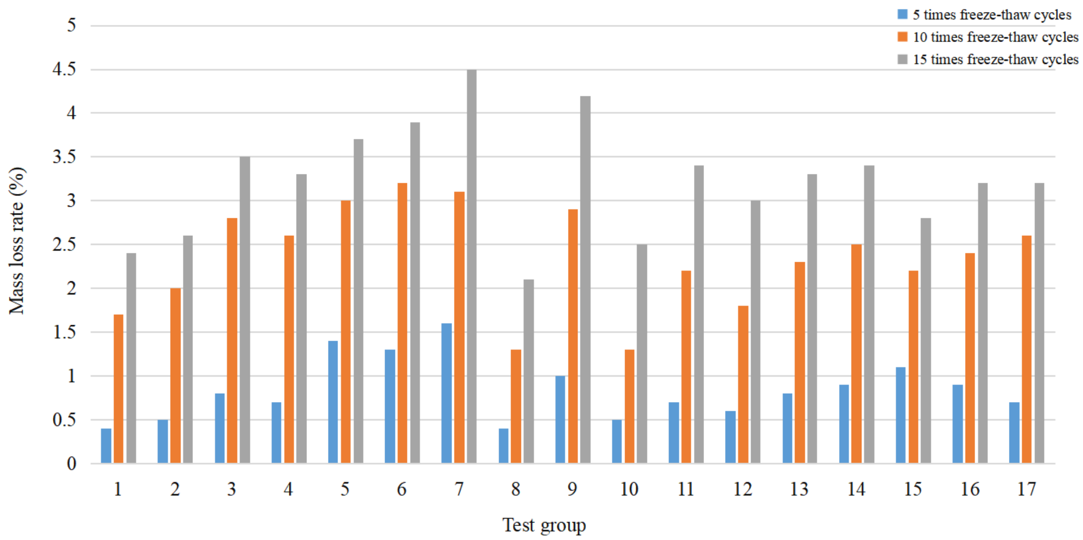


Fig. 6 The mass loss rate of testing blocks after FTC

According to the Fig. 6, the mass loss rate was positively connected with the number of FTC, and the mass loss rate of all 17 sets rice straw-mortar composite blocks all increased with the increase of the F-T times. After 5th, 10th, and 15th F-T, the mass loss rate varied from 0.4% to 1.6%, 1.3% to 3.0%, and 2.1% to 4.5%, respectively. The maximal mass loss rate of testing blocks was not bigger than 5%, which meant that the composite materials has good frost resistance according to the regulation of "Dry Mix Thermal Insulating Composition for Buildings" (GB/T20473-2006).

In order to better reflect the influence of straw content on the mass loss rate of rice straw-mortar composite materials under the action of F-T, comparing the experimental results of the 7th and 16th sets, the 9th and 12th sets, and the 10th and 15th sets in Table 4 (as shown in Fig. 7), we found that the composite materials with bigger straw content had a larger mass loss rate after F-T. Zhou [30] studied the phosphogypsum-straw composite wall materials, and observed that the straw content had a significant effect on the mass loss rate of mortar samples under the condition of FTC, which was consistent with the conclusion of this study.

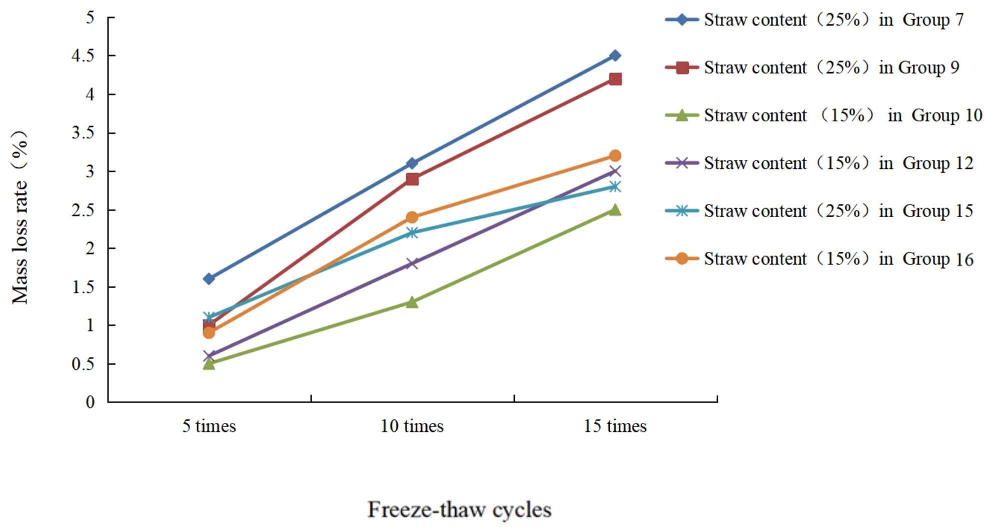


Fig. 7 Effect of straw content on mass loss rate under FTC

4.2 Analysis of testing results after 15 times F-T

The following mainly analyzed and studied the compressive strength loss rate, mass loss rate, and water absorption rate of the testing block after 15 times F-T. The testing results are shown in Table 9, the range of compressive strength loss rate was between 32.2% and 47.3%, the mass loss rate varied from 2.1% to 4.5%, and the water absorption rate was ranged from 22.6% to 42.2%. According to the testing results of 7th and 16th sets, the 9th and 12th sets, and the 10th and 15th sets composite materials in Table 9, it could be found that the compressive strength loss rate increased with the increase of water absorption rate, and the water absorption rate increased with the increase of straw content of the composite materials with the same values of gypsum content and straw length after FTC. After the data analyzed of FTC, it could be found that the minimum compressive strength loss rate of the rice straw-mortar composite materials after 15 times F-T exceeded 30%. Yi [29] obtained that the stress at the bottom of 5 meters high non-load-bearing wall constructed with straw-mortar composite materials was 0.066 MPa, and the maximum compressive strength of straw-mortar composite block almost with the same density in this test was 2.01 MPa after 15 times F-T, much higher than 0.066 MPa. Therefore, the straw-mortar composite materials in this test could meet the actual using requirement of non-load-bearing wall.

Table 9 The performance change of composite materials after 15 times F-T

Number	x_1 (%)	x_2 (mm)	x_3 (%)	y_f (MPa)	$y_{f_{15}}$ (MPa)	Δf_{15} (%)	y_m (g)	$y_{m_{15}}$ (g)	Δm_{15} (%)	y_w (%)	$y_{w_{15}}$ (%)
1	15	20	20	2.60	1.63	37.3	426	416	2.4	28.6	33.2
2	15	10	20	2.35	1.50	36.2	493	480	2.6	23.5	27.9
3	10	15	20	1.90	1.25	34.2	506	488	3.5	25.3	30.0
4	10	15	20	2.17	1.46	32.7	521	503	3.3	24.9	28.3
5	5	10	20	1.65	1.06	35.8	486	468	3.7	22.9	27.1
6	5	20	20	2.00	1.23	38.5	470	451	3.9	23.1	29.2
7	5	15	25	0.55	0.29	47.3	380	363	4.5	34.2	42.2
8	10	20	15	3.00	2.01	33.3	547	536	2.1	18.2	22.6
9	10	10	25	0.85	0.52	38.8	444	425	4.2	26.8	34.0
10	15	15	15	2.53	1.60	36.8	470	459	2.5	21.8	26.0
11	10	20	25	2.15	1.29	40.0	399	386	3.4	32.4	36.8
12	10	10	15	2.70	1.83	32.2	526	510	3.0	20.2	25.3
13	10	15	20	2.10	1.42	32.4	513	496	3.3	25.9	28.8
14	10	15	20	1.85	1.21	34.6	511	494	3.4	26.7	27.1
15	15	15	25	0.95	0.59	37.9	326	316	2.8	36.3	40.6
16	5	15	15	1.10	0.63	42.7	501	485	3.2	22.6	25.9
17	10	15	20	1.97	1.29	34.5	484	468	3.2	25.3	29.5

Where, x_1 is gypsum content (%); x_2 is straw length (mm); x_3 is straw content (%); y_f is compressive strength in normal temperature condition (MPa); y_m is dry mass in normal temperature condition (g); y_w is water absorption rate in normal temperature condition (%); $y_{f_{15}}$ is compressive strength after 15 times F-T (MPa); $y_{m_{15}}$ is dry mass after 15 times F-T (g); $y_{w_{15}}$ is water absorption rate after 15 times F-T (%); Δf_{15} is compressive strength loss rate of testing block after 15 times F-T (%); Δm_{15} is mass loss rate of testing block after 15 times F-T (%).

4.2.1 Compressive failure of testing block under FTC

Comparing Figs. 8 and 9, when the ultimate pressure reached, there was no obvious cracks on the surface of the testing block at room temperature, while the surface of the testing block after 15 times F-T had obvious vertical cracks and a large amount of sand and gravel fell down. The whole testing blocks no longer maintained a complete cube shape, but the block did not collapse completely. This might due to the reason that there were uniformly dispersed straw in the composite materials, which could still play a role of mechanical support to a certain extent. The strength of the composite materials changed and the degree of damage increased under the action of FTC [31].

Fig. 8 Compression failure of specimens in normal temperature

Fig. 9 Compression failure of specimens after 15 times F-T

4.2.2 Water absorption rate after FTC

Water absorption rate affected the durability of materials, and partly reflected the compactness and strength of materials. Ishigami [32] found that the water absorption rate of concrete continuously increased in the process of F-T. Dean [33] obtained that the internal structure of the aggregate damaged due to the increase of frost heave and pore volume when lightweight aggregates with high moisture content subjected to F-T action. These were consistent with the results which could be gotten from the Figs. 8 and 9. The tightness of material's internal connection played a decisive role in the compressive strength. If the water absorption rate was large, the internal connection was relatively loose, and the mechanical properties of the material would reduce. According to the testing results of Table 9, the water absorption rate of most testing blocks generally increased after 15 times F-T under room temperature conditions. The reason might be that with the internal temperature of the freezer device decreased, the internal temperature of the testing block continued to decrease, and the straw structure contained a large amount of unfrozen water, which caused the internal volume to expand and generated pressure after freezing. When the solution in the rice straw-mortar composite materials thawed, the volume expansion caused by icing would remain unchanged, and the newly expanded volume space would be filled with water again when the testing block was soaked in water once more. After repeated F-T, the water absorption rate increased. Therefore, in some special environmental conditions, waterproof measures must be adopted to reduce the damage of F-T to the strength and structural stability of rice straw-mortar composite materials.

5 Conclusions

In this study, the response surface test was designed through Design-Expert software to analyze the physical and mechanical properties and F-T resistant performance of the composite materials with different gypsum content, straw length, and straw content. The main conclusions were as following:

(1) The straw content had extremely significant influence on the compressive strength, dry density, and water absorption rate. With the continuous increase of straw content, the compressive strength and dry density of the rice straw-mortar composite materials continued to decrease, and the water absorption rate continued to increase.

(2) The optimal values of compressive strength, dry density and water absorption rate of rice straw-mortar composite materials were 3.06 MPa, 1.340 $g \cdot cm^{-3}$, and 20.8%, respectively, when the gypsum content, straw length, and straw content were 15%, 20 mm, and 15%, respectively. Under these conditions, the rice straw-mortar composite materials had good physical and mechanical properties.

(3) The compressive strength loss rate, mass loss rate and water absorption rate increased with the increase of F-T times and straw content. After 15 times F-T, the compressive strength loss rate of rice straw-mortar composites materials varied from 32.2% to 47.3%, the mass loss rate varied from 2.1% to 4.5%, and the water absorption rate varied from 22.6% to 42.2%. Although the compressive strength loss rate of the testing block was more than 25%, the rice straw-mortar composite materials after F-T still retained a certain strength, which could be applied to non-bearing walls and partitions with certain waterproof measures.

Data Availability Statement All data generated or analysed during this study are included in this published article.

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