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RESEARCH **Open Access**

Damage and Deterioration Mechanism of Coal Gangue Mixed Pumice Aggregate Concrete Under Freeze–Thaw Cycles

Wen Li[1], Hailong Wang[1,2*] and Wenyan Zhang[1]

Abstract

The world is facing the problem of depletion of natural sand and gravel resources, and a large amount of coal gangue solid waste is produced in Inner Mongolia, China, which has low utilization rate and causes ecological pollution. In order to improve the gangue in the mining infrastructure construction of a wide range of application prospects, the use of coal gangue as the coarse aggregate of pumice concrete is of great significance. Inner Mongolia is a cold region, and gangue mixed aggregate concrete (MFC) will certainly face the damage caused by freeze–thaw cycles. Therefore, design gangue by different volume replacement rate (0%, 20%, 40%, 60%, 80%,100%) to replace pumice coarse aggregate. The results show that with the increase of gangue substitution rate, the mass loss rate, relative dynamic elastic modulus, and peak stress of MFC decrease, but the trend of peak strain increases. It is mainly attributed to the less Al_2O_3 and SiO_2 content of gangue, which makes the MFC hydration products decrease with the increase of substitution rate and more original microcracks and pores in the specimens. In addition, the damage model of MFC was established by using Weibull statistical distribution theory and the principle of LEMAITRE equivalent effect variation assumption, and the damage evolution characteristics were explored by combining the experimental results. It can provide the theoretical basis for the application of MFC in cold regions.

Keywords Coal gangue aggregate, Pumice aggregate, Freeze–thaw cycle, Elastic modulus, Damage degradation model

1 Introduction

Inner Mongolia is one of the important provinces with rich coal reserves in China, only in 2020 coal production reached 1.06 billion tons, and the production of coal gangue is even more huge (Derivatives in symbiosis with raw coal). Due to the complex composition and characteristics of gangue itself in different regions, the targeted scientific research and mature application technology in Inner Mongolia Ordos region is relatively less, resulting in relatively low utilization rate of gangue resources, mainly using accumulation treatment, coal gangue hills have small accumulation slope, covering a large area, that easy to spontaneous combustion and releases a large amount of sulfide pollution to the atmosphere, farmland and water. On the contrary, with the rapid development of ecological construction in Inner Mongolia, the speed of mining infrastructure construction and engineering slope protection increased year by year, the demand for concrete increased year by year, conventional gravel concrete is still chosen as the main material of engineering concrete, and a large number of gravel mining aggravate the cost of ecological recovery

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and form a vicious cycle. On the basis of fully analyzing the biochemical physical characteristics of coal gangue in Shenhua Quasi—energy open-pit coal Mine in Zhungeer County, Wuhai City, coal gangue is used to prepare concrete (Fig. 1), which can effectively reduce the cost and realize the differentiated comprehensive utilization of waste. Inner Mongolia has potential development value and broad application prospects. Based on previous research, as a lightweight material, pumice has many excellent characteristics, such as small density, high strength, excellent water absorption rate, strong acid–alkali resistance, strong corrosion resistance, pollution-free, and no radiation activity. In this study, the selection of pumice as a composite admixture not only can improve the performance of concrete but also make full use of regional resources. In order to explore the feasibility of gangue and pumice mixed as coarse aggregate to prepare concrete, the research direction of gangue mixed aggregate concrete is put forward.

Frozen-thaw damage is a common disease of concrete structure in Inner Mongolia and a major factor affecting their durability (Guo et al., 2020; Xie et al., 2021). Based on the chemical and physical properties of coal gangue and pumice, the damage evolution law of concrete under freeze–thaw is studied. At present, there are few researches on freeze–thaw damage evolution of light aggregate concrete mixed with coal gangue and pumice in China. In this paper, the stress–strain relationship of gangue mixed aggregate concrete (MFC) after freezing–thawing cycle is studied. Based on Weibull statistical distribution theory and LEMAITRE equivalent strain assumption principle, the damage constitutive model of MFC under uniaxial compression considering freezing–thawing cycle was established to study the mechanical properties and damage evolution law of MFC after freezing–thawing, providing theoretical and technical reference for the engineering application of MFC in Inner Mongolia.

2 Test Materials and Methods

2.1 Test Materials

In this study, the cement used Jidong P.042.5 ordinary Portland cement. The coarse aggregate was coal gangue from Shenhua Quasi—energy open-pit coal Mine in Zhungeer County, Wuhai city and Ordos City and natural pumice from Wulanchabu city, Inner Mongolia. Physical properties are shown in Table 1. The fine aggregate was Grade medium sand. The water reducing agent is polycarboxylic acid type high efficiency water reducing agent, and its water reduction rate is 20%.

2.2 Mix Proportion

According to JGJ51-2019 "Technical Specification for Light Aggregate Concrete," the water–cement ratio of coal gangue mixed aggregate concrete is a fixed value of 0.43, and coarse aggregate of coal gangue replaces coarse aggregate of pumice with equal volume, with substitution rates of 0%, 20%, 40%, 60%, 80%, and 100%. The group with substitution rate of 0% is marked as MFC-0. A replacement rate of 20% is denoted as MFC-20 and so on. The mix proportions of the concrete are shown in Table 2.

Table 1 Physical properties of the coarse aggregates

Kinds	Accumulating density (kg/m^3)	Apparent density (kg/m^3)	water absorption (%/h)	Crash index (%)
Coal gangue	1150	214	3.24	24
Pumice	715	159	12.78	43

Fig. 1 Coal gangue from Shenhua Quasi—energy open-pit coal Mine dump in Zhungeer County, Wuhai city

Table 2 Mix proportions of the concrete

Groups	Cement (kg/m^3)	River sand (kg/m^3)	Float stone (kg/m^3)	Coal gangue (kg/m^3)	Water (kg/m^3)	Admixture (kg/m^3)
MFC-0	420	706.6	649.4	0	211	4.2
MFC-20	420	706.6	519.5	174.5	211	4.2
MFC-40	420	706.6	389.6	348.9	211	4.2
MFC-60	420	706.6	259.8	523.4	211	4.2
MFC-80	420	706.6	129.9	697.8	211	4.2
MFC-100	420	706.6	0	872.3	211	4.2

2.3 The Experimental Method

Referring to GB/T 50082-2009 "Standard for Test Method of Long-term Performance and Durability of Ordinary Concrete" and GB/T 50082-2009 "Standard for Test Method of Mechanical Properties of Ordinary Concrete," the concrete specimens are soaked for 4 days and subjected to freezing–thawing cycle test by "quick freezing method." After every 25 cycles, the mass and relative dynamic modulus of the specimen (100 mm × 100 mm × 400 mm) were tested. The uniaxial compression stress–strain curves of specimens (100 mm × 100 mm × 300 mm) were tested after 0, 25, 50, 75, and 100 times of freeze–thaw. Furthermore, the conversion coefficient of non-standard test block size is 0.95.

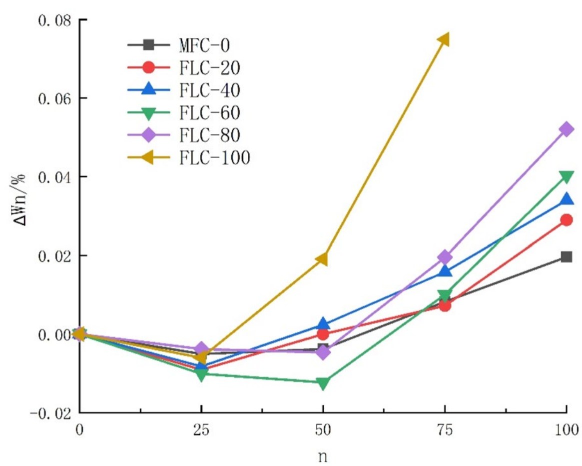


Fig. 2 The relationship curve between the mass loss rate of MFC and the number of freeze–thaw cycles

3 Test Results and Analysis

3.1 Relationship Between Coal Gangue Substitution Rate and Mass Loss Rate and Moving Modulus

The mass loss rate can reflect the surface erosion of the concrete under the freeze-melting cycle (Wang et al., 2018; Wawrzeńczyk & Molendowska, 2017). In this experiment, three specimens were poured at 25, 50, 75, and 100 freeze–thaw cycles, and the average of the mass loss rate of each group of three specimens was taken as the graphical data. The MFC mass loss rate is shown in Fig. 2. With the number of freeze–thaw cycles, the overall quality loss rate of MFC concrete shows a decreasing trend at first and then an increasing trend. Before the freeze–thaw cycle factor of 50 times, the mass of MFC concrete table then reduced by freeze–thaw spalling is less than the mass increased by deep hydration of internal components, and a certain degree of mass increase occurs. When the number of freeze–thaw cycles reached 75 times, the mass loss rate in the MFC-100 group reached 7.5% and more than 5% reached the damage state. When the number of freeze–thaw cycles reached 100 times, quality loss of over 5% in the MFC-80 group reached the destruction status, the minimum mass loss rate of MFC-0 group was 2.0%, the test showed that in the early stage of the freeze–thaw cycle, the mass of MFC concrete surface reduced by freeze–thaw spalling is smaller than the mass of internal components increased by deep hydration, and there is a certain degree of mass increases. But as the freeze–thaw cycle continues to work, MFC concrete surface erosion and internal freeze–thaw damage are intensified and its quality loss rate has gradually increased, and with the increase of coal gangue substitution rate, mass loss rate increases, which shows that the coal gangue intensifies the concrete damage under the freezing and melting cycle. The main reasons are the large porosity of coal gangue coarse aggregate, low density, low strength, large micropowder, and high carbon content than ordinary rock, and in the crushing process, it is more in the needle tablet content. In the mixing process, it is easy to form a needle sheet accumulation, resulting in a cavity phenomenon. Therefore, the number of large pores in the concrete increases accordingly, and the freezing stress produced by the water in the pore also increases accordingly. Therefore, the greater the substitution rate of coal gangue, the freezing resistance performance of concrete is reduced to a certain extent.

The relative dynamic elastic modulus Pi can reflect the changes in the internal freezing-thaw damage except the concrete, the smaller the value, the greater the freeze–thaw damage (Wang et al., 2018; Wawrzeńczyk & Molendowska, 2017). As shown in Fig. 3, with the number of freeze–thaw cycles, the relative elastic modulus of MFC all showed a downward trend. When the number of freeze–thaw cycles reaches 75 times, the relative dynamic elastic modulus of MFC-100 group reaches 62.14%, 60% of the failure condition specified in the critical specification. When the number of freezing–thawing cycles reached 100, the MFC-100 group was damaged, and the dynamic elastic modulus was not suitable for testing. The other five groups did not exceed 60%. The maximum value of the relative dynamic elastic modulus of MF-0 group was 83.32%. The results show that with the increase of coal gangue replacement rate, the freezing–thawing damage in MFC concrete increases under the same number of freezing–thawing cycles.

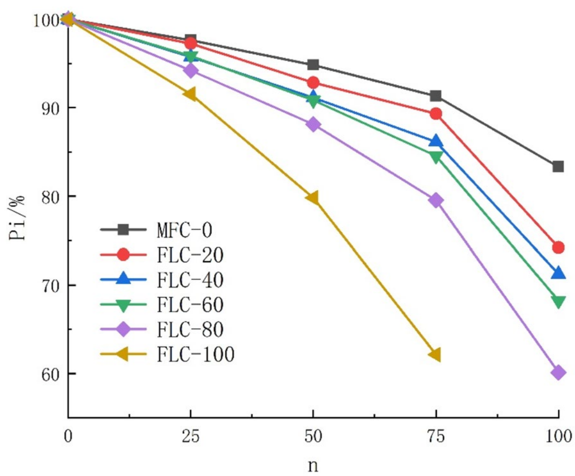


Fig. 3 The relationship curve between the relative dynamic elastic modulus of MFC and the number of freeze–thaw cycles

3.2 Attenuation of Stress–Strain Curve of Coal Gangue Concrete Under Different Freeze–Thaw Cycles

As shown in Fig. 4, this is the full stress–strain curve of different freeze–thaw cycles, and MFC-20-25 in the legend indicates that MFC-20 group undergoes 25 freeze–thaw cycles and so on. As the number of freeze–thaw cycles increases, the sample stress–strain curve deviated from the Y axis, the peak stress was significantly reduced, and the peak strain gradually increased. The peak stresses of MFC-0, MFC-20, and MFC-40 groups decreased by 81.2%, 77.1%, and 76.0% after 100 freeze–thaw cycles, and the corresponding peak stresses increased by 297.5%, 135.4%, and 95.3%; the corresponding modulus of elasticity decreased by 93.5%, 88.5%, and 84.5%. However, the specimens of MFC-60 and MFC-80 groups were destroyed after subjected to freeze–thaw cycles for

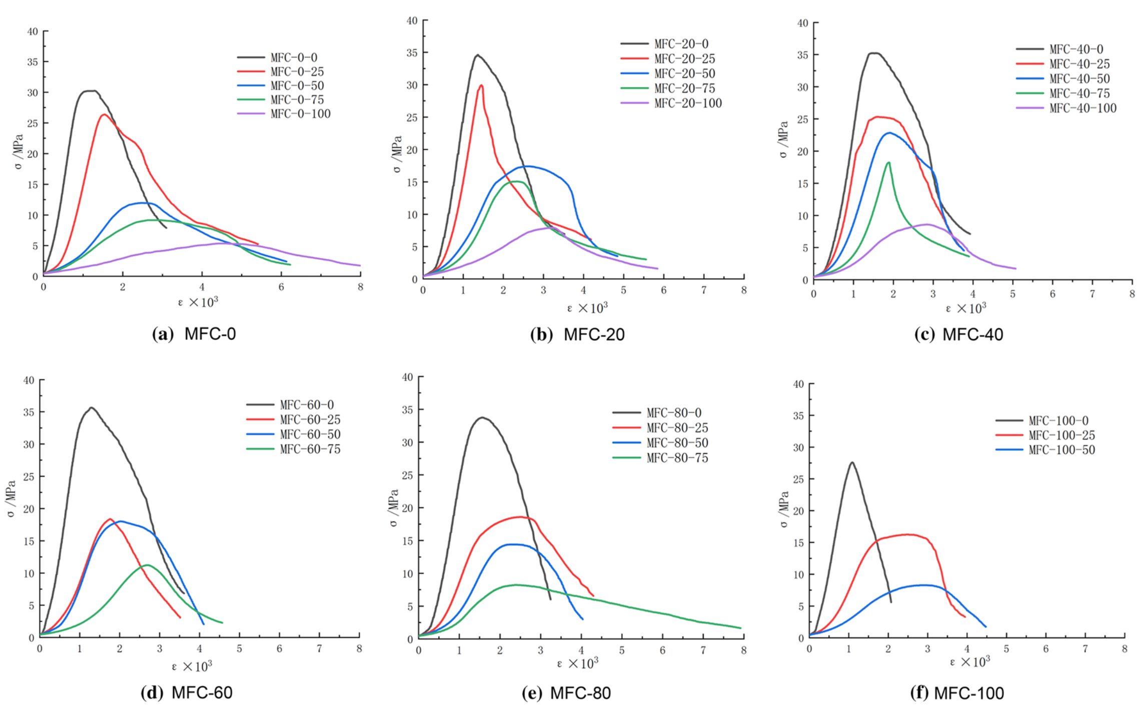


Fig. 4 The stress–strain full curve of different freeze–thaw cycles of MFC concrete

100 times, and the specimens of MFC-100 group were destroyed after subjected to freeze–thaw cycles for 75 times. The changes of the above data indicate that the stress–strain attenuation amplitude of MFC concrete increases with the increase of the substitution rate of coal gangue coarse aggregate after freeze–thaw cycle.

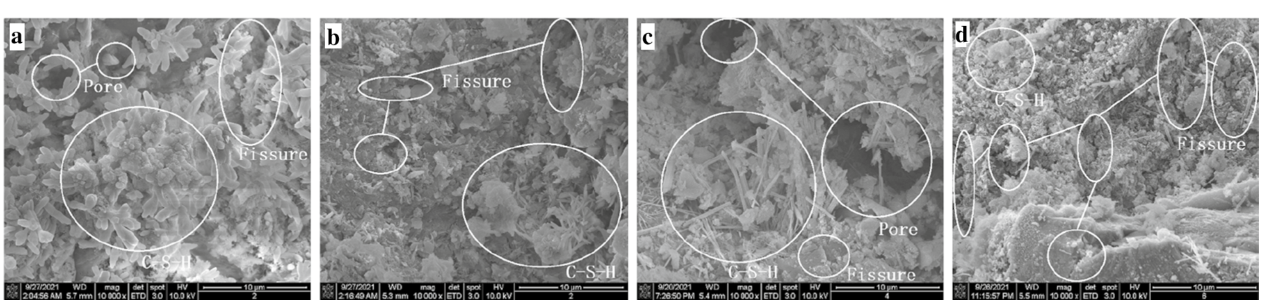
3.3 SEM Analysis

Fig. 5 shows the microscopic comparison of MFC with different substitution rates and SEM at the same multiple after freeze–thaw cycles. According to the determination results of XRF element, the main internal components of pumice and coal gangue are Al_2O_3 and SiO_2, with contents of 36.5% and 36.5% and 17.8% and 50.0%. Among them, the internal Al_2O_3 content of gangue is lower than that of pumice, and the SiO_2 content is higher than that of pumice. The active Al_2O_3 and SiO_2 on the surface of coarse aggregate can react with $Ca(OH)_2$ in cement slurry to generate C–S–H gelation and hydration products containing $Al3^+$ (Yi et al., 2017). From Fig. 5a–c it can be seen that with the increase of coal gangue replacement rate, the hydration reaction of the specimen deepened and the flock-like and acicular hydration reaction products (C–S–H) increase, but the overall hydration reaction products decrease and the filling effect of hydration products decreases. The analysis of the reason is that the content of Al_2O_3 and SiO_2 of coal gangue is lower than that of pumice, the active components that can react with cement mortar decrease, and the hydration reaction products decrease. Cracks and pores appear to increase the phenomenon of becoming larger, which is the same as the reason analysis in Sect. 2.1. With the increase of coal gangue replacement rate, larger pores are more likely to be generated in MFC. After freezing–thawing cycles, due to the higher water absorption rate of pumice and coal gangue, water in aggregate freezes and expands, and mortar on aggregate surface is stripped (Wang et al., 2014). It can be clearly seen from Fig. 5d that the mortar part is separated, resulting in a large number of cracks, which degrades the performance of MFC. With the increase of freezing–thawing times, microcracks do not increase, crack width increases, microcracks further expand into large cracks, and deterioration deepens continuously.

4 Development of the Statistical Damage Degradation Model for MFC

4.1 Model Building

With the material difference of pumice and coal gangue, the water absorption rate is about 4 times and the strength is 0.5 times. As coal gangue substitution rate increases, aggregate strength increases, water absorption decreases, porosity increases, and larger pores increase. So MFC strength showed a trend of first increase and then decrease with the increase of gangue substitution rate. In conclusion, the internal structure of MFC is complex and diverse, in order to deeply study the effect of coal gangue substitution rate on MFC damage deterioration under the load effect of MFC after freeze-melting cycle. The destruction of MFC is regarded as a continuous development process, and the assumption is made: (1) the pumice and coal gangue aggregate are evenly distributed in the concrete. (2) MFCs with different substitution rates are greatly uniform, and the inherent damage caused by its own defects is defined as the first stage, and the inherent damage increases with the coal gangue substitution rate. The damage degree represented by the degradation degree of elastic modulus under freezing–thawing cycle is defined as the second stage. The damage caused by load after freezing–thawing cycle is defined as the third stage. (3) During the loading process of the test piece, the interface bond damage at the loading end appears first and accumulates gradually, which is manifested as interface slip at macro level. With the increase of load, the interface bond damage accumulates continuously and extends to the free end until the specimen is



(a) MFC-0 (b) MFC-60 (c) MFC-100 (d) After freezing-thawing MFC-60

Fig. 5 SEM comparison diagram. The figure (**a**, **b**, **c**) show the SEM images at different substitution rates before the freeze-thaw cycles destroyed. (4) To the interface of concrete under compression load as a network composed of several bonding point system, assuming that each bond force the same point, and after freeze–thaw action is defined as the number of bonding point initial damage, and in the process of continuous load, bonding point of fracture network system by load, create effective bonding point number decreases, and at the same time the stick point number obeys WEIBULL probability distribution.

$$P(F) = \frac{a}{b}\left(\frac{F}{b}\right)^{a-1} \exp\left[-\left(\frac{F}{b}\right)^{a}\right], \tag{1}$$

where F is the distribution variable of the adhesion point; a and b are the WEIBULL distribution parameters, respectively.

The elastic modulus obtained at the first and second stages was defined by the initial damage. According to the damage mechanics theory, the response energy of the macroscopic physical and mechanical properties of concrete represents the degree of deterioration inside the material, so the damage variable D_n after MFC freeze–thaw cycle is

$$D_n = 1 - \frac{E_n}{E_0}. \tag{2}$$

According to LEMAITRE strain equivalence hypothesis (Lemaitre, 1984), the strain produced by the action of nominal stress σ on damaged materials is equivalent to the strain produced by the action of effective stress on non-destructive materials (Long et al., 2018; Ma et al., 2021; Shang et al., 2015). Therefore, the damage constitutive relation of concrete under the loading of freezing–thawing action can be expressed as follows:

$$\sigma = E_0\varepsilon(1 - D). \tag{3}$$

In the formula, $D = D_n + D_c - D_nD_c$, D is the total damage variable and D_c is the damage variable caused by the load action.

Based on the assumption, combining the WEIBULL probability defines the number of adhesion damage N_d and the total adhesion number N_t in the MFC as the damage variable, which can be expressed as

$$N_d = N_t\int_0^{\varepsilon} P(F)dF = N_t\left\{1 - \exp\left[-\left(\frac{\varepsilon}{a}\right)^{b}\right]\right\}. \tag{4}$$

The damage variable D_c under load can be expressed as the ratio of the number of broken adhesion points to the total number of adhesion points:

$$D_C = \frac{N_d}{N_t} = 1 - \exp\left[-\left(\frac{\varepsilon}{a}\right)^{b}\right]. \tag{5}$$

Therefore, the total damage variable D available (6) after the freeze–thaw cycle is indicated as follows:

$$D = 1 - \frac{E_n}{E_0}\exp\left[-\left(\frac{\varepsilon}{a}\right)^{b}\right]. \tag{6}$$

The load-bearing constitutive relation of concrete after n cycles of freezing–thawing can be derived as follows:

$$\sigma = E_n\varepsilon\exp\left[-\left(\frac{\varepsilon}{a}\right)^{b}\right]. \tag{7}$$

4.2 Physical Significance of the Model Parameters

Based on Formula (7), in order to explore the influence relationship of changes in parameters a and b on constitutive model curves, by controlling parameters E_n and b to be arbitrary fixed values, the change law of stress–strain curves of parameters a and b was explored, as shown in Fig. 6.

With the increase of parameter a, the slope of the rising and falling curves has no significant change, but the peak point is increasing and parameter a positively correlated with the peak stress in the stress–strain curve. The greater the peak stress, the greater the peak strain is also increasing.

With the increase of parameter b, the slope of the stress–strain curve decreases significantly, the peak stress increases, and the slope becomes larger, indicating the brittle characteristics of concrete (Qin et al., 2021; Sun et al., 1999). Therefore, the parameter b reflects the fragility characteristics of the concrete, and with the increase of parameter b, the concrete peak stress increases, but the fragility also increases and the limiting strain decreases.

4.3 Model Parameter Calculation and Verification

Based on the characteristic of the slope of 0 at the peak point of the stress–strain curve, at the curve peak point (ε_n, s_n), $\varepsilon = \varepsilon_n$ and $s = s_n$ (Liu et al., 2021; Zhang et al., 2015; Zhu et al., 2019). The calculation formulas of control parameters a and b can be obtained through deduction:

$$a = \frac{\varepsilon_n}{(1/b)^{1/b}}, \tag{8}$$

$$b = \frac{1}{\ln\left(\frac{E_n\varepsilon_n}{\sigma_n}\right)}. \tag{9}$$

The relevant parameters of the constitutive model obtained by calculating a and b are shown in Table 3. The stress and strain results calculated by the model and the test results are shown in Fig. 7.

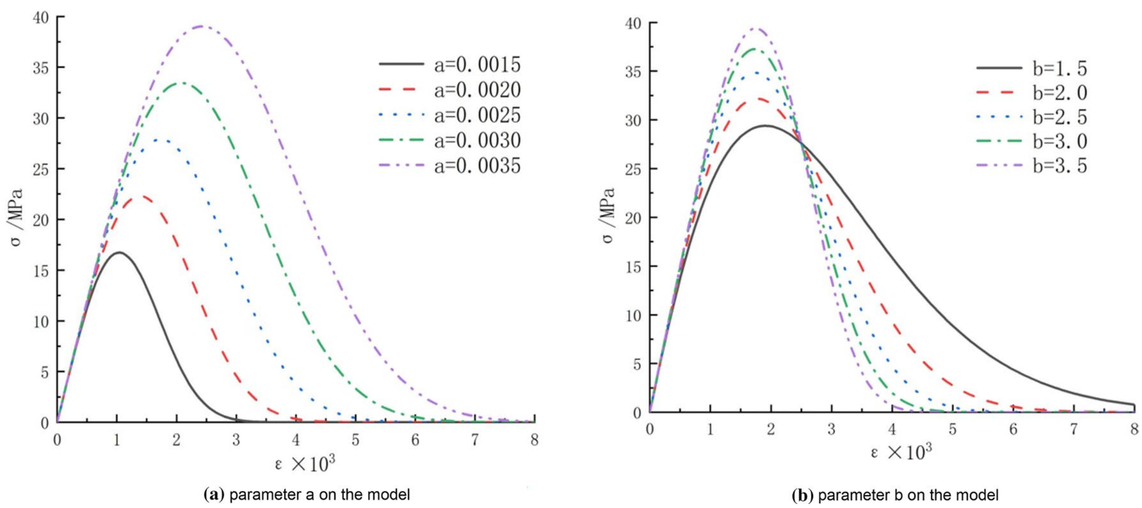


Fig. 6 Effect of parameters *a*, *b* on the model

Table 3 Weibull distribution parameters

Sample number	*a*	*b*	Sample number	*a*	*b*	Sample number	*a*	*b*
MFC-0-0	0.001662	2.8526	MFC-20-0	0.001977	2.5089	MFC-40-0	0.002139	2.7142
MFC-0-25	0.002213	2.4507	MFC-20-25	0.002067	1.9531	MFC-40-25	0.002492	2.6121
MFC-0-50	0.002823	1.5594	MFC-20-50	0.003129	1.7645	MFC-40-50	0.002713	2.5869
MFC-0-75	0.003163	1.1729	MFC-20-75	0.00342	1.483	MFC-40-75	0.002957	1.923
MFC-0-100	0.004355	0.954	MFC-20-100	0.004455	1.4736	MFC-40-100	0.003847	1.5495
MFC-60-0	0.001856	2.7067	MFC-80-0	0.002196	2.9611	MFC-100-0	0.001515	4.6913
MFC-60-25	0.002533	2.3503	MFC-80-25	0.003072	2.4068	MFC-100-25	0.003055	1.3354
MFC-60-50	0.002949	1.721	MFC-80-50	0.003316	2.0967	MFC-100-50	0.00366	1.3142
MFC-60-75	0.003922	1.5777	MFC-80-75	0.003493	1.8762			

We can see from Table 3 that after 100 freeze–thaw cycles, the model parameter a of the MFC-0, MFC-20, and MFC-40 groups increased by 162.0%, 125.3%, and 79.9% relative to the do not freeze–thaw state, respectively, indicating that the peak strain increases in the MFC-0 group under 100 cycles, and the parameter b decreased by 66.6%, 41.3%, and 42.3%, respectively, indicating that the peak strain and limit strain of the MFC-0 group increased significantly under 100 freezing-thaw cycles, and the fragility characteristics are more obvious. Due to the failure of MFC-60, MFC-80, and MFC-100 specimens after 100 freeze–thaw cycles, no comparative analysis under the same number of freeze–thaw cycles is done. However, according to the control parameters a and b, the MFC-100 decreased by 71.9% in parameter b with just 50 freeze–thaw cycles. This is the same as that found in 2.1. The increase of coal gangue replacement rate reduces the frost resistance of MFC. The stress–strain shape variation law and concrete characteristics of MFC reflected by parameters a and b after freezing–thawing cycle are consistent with the change law of test results, indicating that the constitutive model established in this paper can predict the loading performance of MFC after freezing–thawing cycle.

As we can see from Fig. 7, the established damage constitutive model curve has a high fit with the test curve before reaching the peak stress, and the deviation after the peak stress begins. As the peak stress test piece tends to damage and failure, there is no in-depth research in this paper.

4.4 Characteristics of the Changes in the Injury Variables

In order to explore the damage and development characteristics of MFC under loading after freezing–thawing cycle, damage variable D and strain of MFC under loading of 6 groups of samples were calculated according to Eq. (6), and their variation relationship is shown in Fig. 8. It can be seen from Fig. 8 that the total damage variable of MFC increases monotonically with strain. When strain is 0, the initial damage is positively

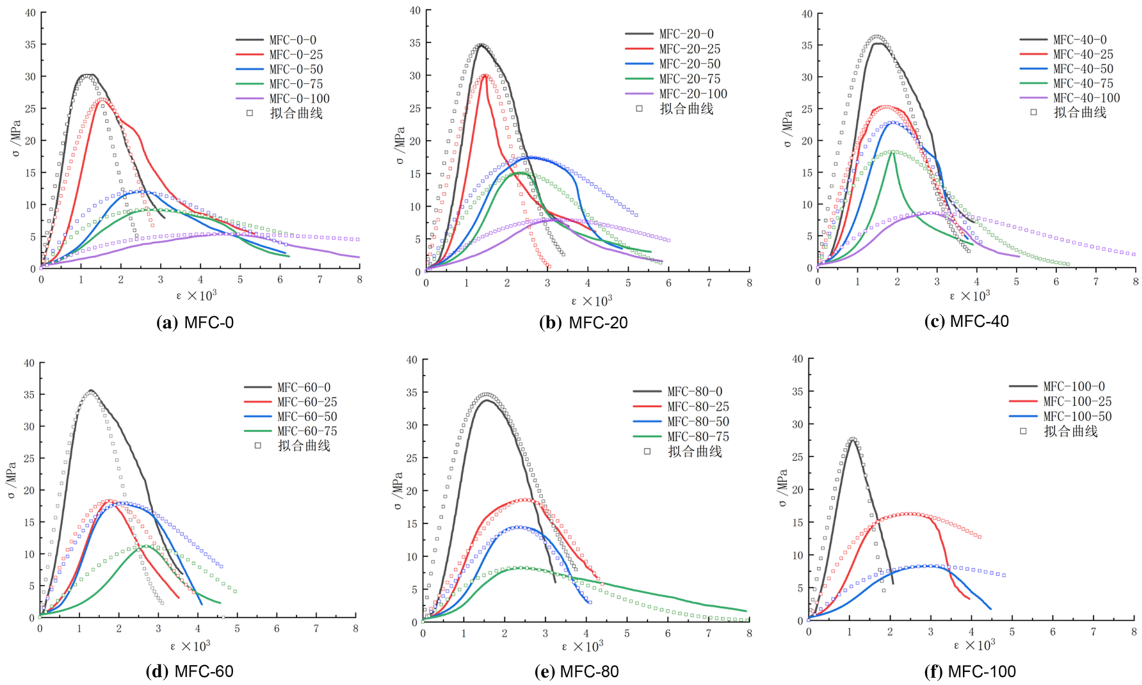


Fig. 7 The fitted results of this model are compared with the test results

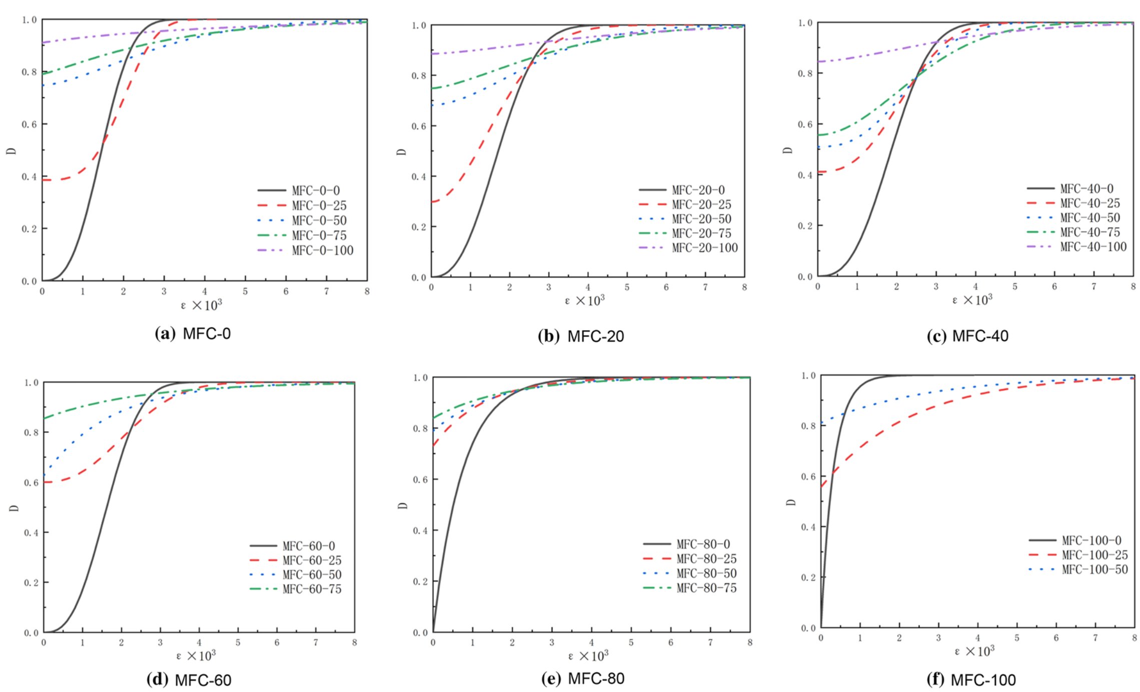


Fig. 8 Relationship between total damage variable *D* and strain under different times of freeze–thaw cycles

correlated with the number of freezing–thawing cycles and the coal gangue replacement rate. MFC with the increase of coal gangue replacement rate, the initial damage value increases, causes the increase improve the concrete porosity and coal gangue, on the one hand, reduce the friction resistance between aggregate and mechanical bite resultant force, on the other hand, coal gangue and slurry form of bonding interface is weaker than the pumice and slurry form bonding interface, the crack propagation probability of aggregate-slurry interface is increased and damage development is faster. MFC after freezing–thawing process. With the increase of freezing–thawing times, the volume of inherent open pores in concrete increases under the action of freezing–thawing cycle, and some closed pores form connected pores under the action of freezing–thawing. Finally, the internal pores of concrete are connected, which increases the damage.

Under the load, with the increase of strain ε, the damage strain curve tends to be gentle, and the increase rate of damage variable decreases. The reason is that with the increase of freeze–thaw cycles, the MFC internal open pore increases. Initial damage increased. However, the vertical deformation under load causes the connected pores to shrink. This process offsets some of the new pores under load. Thus, the damage variable development rate decreases with the increase of freeze–thaw cycles.

The scatter plot is drawn for the damage variable D_n of MFC according to Formula (6). According to point distribution characteristics, logarithmic function was used to fit the functional relationship between MFC damage variable D_n and the number of freezing and thawing action *N*. The fitting results are shown in Fig. 9 and the fitting function is

$$D_n = \ln(c + d \times n). \tag{10}$$

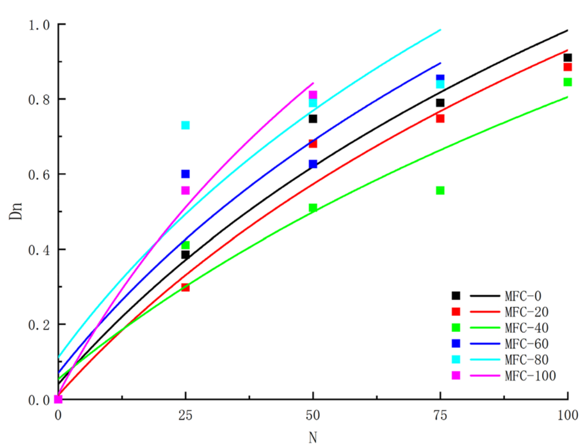


Fig. 9 MFC test values and fitting curves

The corresponding parameters *c* and *d* and the correlation coefficient of the fitting curve are shown in Table 4. MFC-0, MFC-20, and MFC-100 groups have the correlation coefficient above 0.95, and the fitting accuracy is good. The fitting curve can calculate other number of damage variables for MFC except the freeze–thaw cycle design and provide theoretical reference for the damage variables of coal gangue mixed aggregate concrete after undergoing different freezing–thawing cycles.

5 Conclusions

(a) Under the action of freezing–thawing cycle, with the increase of coal gangue substitution rate, the mass loss rate increases, the relative dynamic elastic modulus decreases, and the increase of coal gangue substitution rate deteriorates the MFC freezing resistance to durability.
(b) The damage degradation model was established under different coal gangue substitution rates of MFC based on the action of freezing–thawing. The degradation of coal gangue substitution rate on freezing–thawing was characterized.
(c) The total damage variable *D* of MFC compression load increases monotonically with strain; the initial damage value also increases with the number of freezing and thawing and coal gangue substitution rate.
(d) Scatter plots are drawn according to the number of freeze–thaw damage variables and the number of freeze–thaw cycles. The scatter characteristics fit the function between the D_n of the MFC damage variable and the freeze–thaw times *n*. The fitting accuracy is high, which provides theoretical reference for the damage variables after different freeze–thaw cycles of MFC.

Table 4 Fitting curve parameters

Groups	*c*	*d*	R^2
MFC-0	1.0409	0.0163	0.9561
MFC-20	1.0108	0.0153	0.9711
MFC-40	1.0551	0.0118	0.9242
MFC-60	1.0725	0.0184	0.8981
MFC-80	1.1177	0.0208	0.8080
MFC-100	1.0137	0.0262	0.9908

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Author contributions
WL was involved in conceptualization, writing, and editing. HW contributed to funding acquisition, investigation, and writing—review and editing. WZ was involved in writing—review and editing.

Availability of data and materials
All data generated or analyzed during this study are included in this published article.

Declarations

Competing interests
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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