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Experimental research into the ultrasonic p-wave velocity of slime-based filling materials

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Abstract: To study the influences of different mix proportions of materials on the ultrasonic p-wave velocity of slime-based filling materials, influences of four factors on the p-wave velocity of specimens were investigated by designing orthogonal tests and taking slime as the principal raw material. The four factors included the mass concentration of slime (A), the content of high-water-content material (B), cement content (C), and content of fly ash (D). Test results revealed that: (1) the ultrasonic p-wave velocity is in the range of 3.916 to 8.319 km/s and various factors are listed in a descending order as A, D, B, and C according to their influences on the ultrasonic p-wave velocity; (2) the ultrasonic p-wave velocity is positively correlated with the compressive strength and shear strength, with correlation coefficients separately of 0.87 and 0.65; (3) the equations for variations in the ultrasonic p-wave velocity under influences of different factors are fitted. The ultrasonic p-wave velocity has a quadratic polynomial relationship with factor A, while following exponential relationships with factors B, C, and D. A predictive model for characteristic parameters of the ultrasonic p-wave velocity of slime-based filling materials jointly influenced by the four factors was established based on the fitting equation for variations of single factors and ultrasonic p-wave velocity. The predictive model was then verified.

Keywords: coal slime; backfill material; correlation coefficient; ultrasonic wave speed; formula fitting

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1. Introduction

Slime-based cemented filling materials are filling materials taking slime as the principal raw material and solidified using cementing materials[1]. They are generally applied to filling material areas including mined-out areas and abandoned roadways in the working of coal mines, thus stabilizing the surrounding underground rock mass, improving the safety factor during (and after) the mining process, and increasing the rate of resource utilization[2-5]. Meanwhile, using slime as the principal raw material can also achieve effective utilization of waste arising from coal mining, reduce discharge and stockpiling of slime, mitigate environmental pollution and destruction, and improve the natural environment around coal mines[6-8].

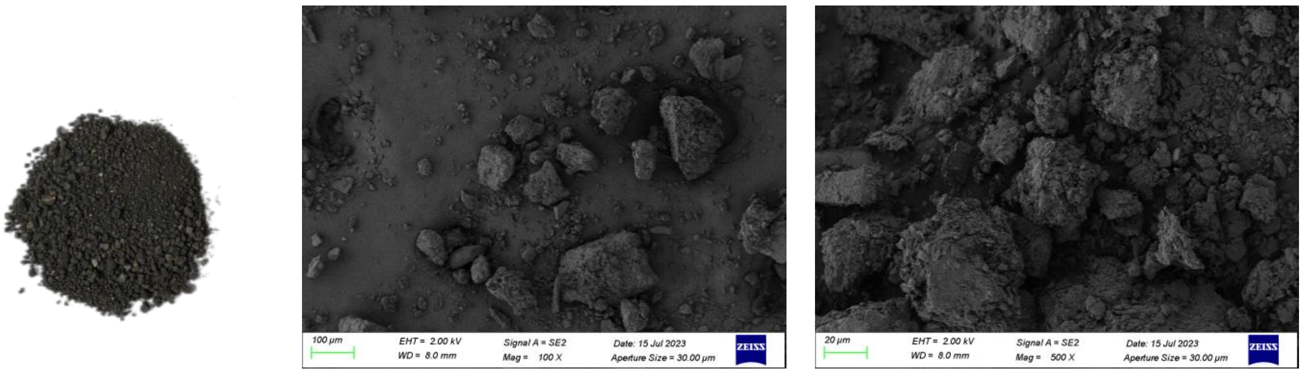
Ultrasonic wave velocity measurement, as a non-destructive measurement method, is used to measure the ultrasonic propagation speed in materials. This method of measurement has been widely applied in materials science, engineering, and medicine to assess the physical properties and quality of materials[9-11]. In application to concrete structures, ultrasonic wave velocity measurement has also found broad use; important information about the quality and performance of concrete can be acquired by measuring the ultrasonic propagation speed in concrete[12-14]. However, ultrasonic wave velocity measurement can only estimate concrete performance and the results are influenced by multiple factors, such as the mix proportions, porosity, water content, and temperature of concrete. The present research applied ultrasonic wave velocity measurement to slime-based filling material specimens to evaluate the quality and performance of the filling material by comparison with measured ultrasonic wave velocities in concrete[15-19].

The proportion and quality of cementing materials may affect the density and strength of filling materials, thus influencing wave-velocity measurement. Generally, a high proportion of cementing materials may lead to a high wave velocity because more cementing materials can improve the strength and density of filling materials. Different proportions of cementing materials cause the ultrasonic wave velocity to change in different manners[20-22]. Besides, the mix proportions of filling materials are correlated with influences of other factors on wave velocity measurement. The variation of one factor may induce variation of other factors, thus affecting the wave velocity measurement results. The tests mainly study the mechanism of influence of mix proportions of slime-based filling materials on the ultrasonic wave velocity and provide a corresponding method of prediction thereof[23-25].

2. Orthogonal test schemes and test results

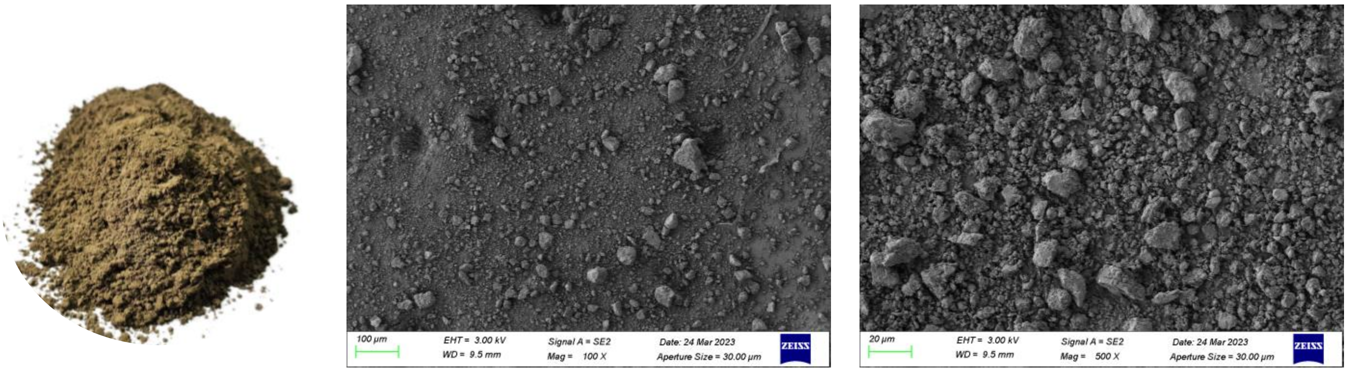
The main material used in the tests was slime discharged during coal washing in Shuiliandong coal mine in Shaanxi Province, China. In addition, high-water-content material, cement, and fly ash were selected as additives, which were used together with slime to prepare the cemented filling materials. Therein, cement was a 42.5# ordinary Portland cement, and fly ash was secondary fly ash discharged from a power plant. The tested raw materials and scanning electron microscope (SEM) images are shown in Figures 1-5. Previous research has shown that specimens have favorable strength and cost when they are prepared with slime at a mass concentration of 28.75% ~ 40.00%, while remaining the high-water-content material, cement, and fly ash being 2.4% ~ 3.9%, 3.3% ~ 10%, and 3.3% ~ 10% of the total mass.

For the convenience of description and calculation, the total mass of slime was fixed as 100 (%) and an orthogonal table with four factors at three levels was set to design test schemes, as listed in Table 1. In the table, (B/A), (C/A), and (D/A) separately represent the mass ratios of high-water-content material, cement, and fly ash to slime.



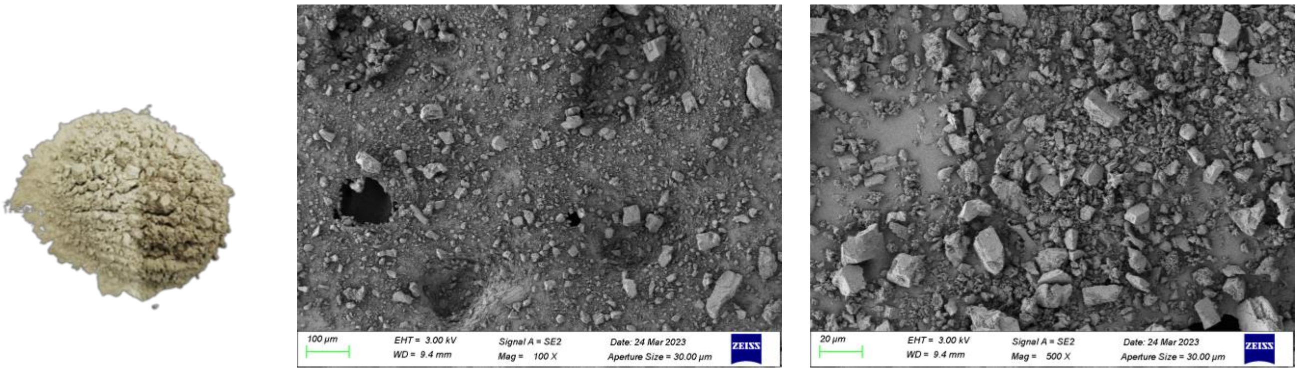
(a) (b)

Figure 1. coal slime and SEM images. (a) 100×; (b) 500×.



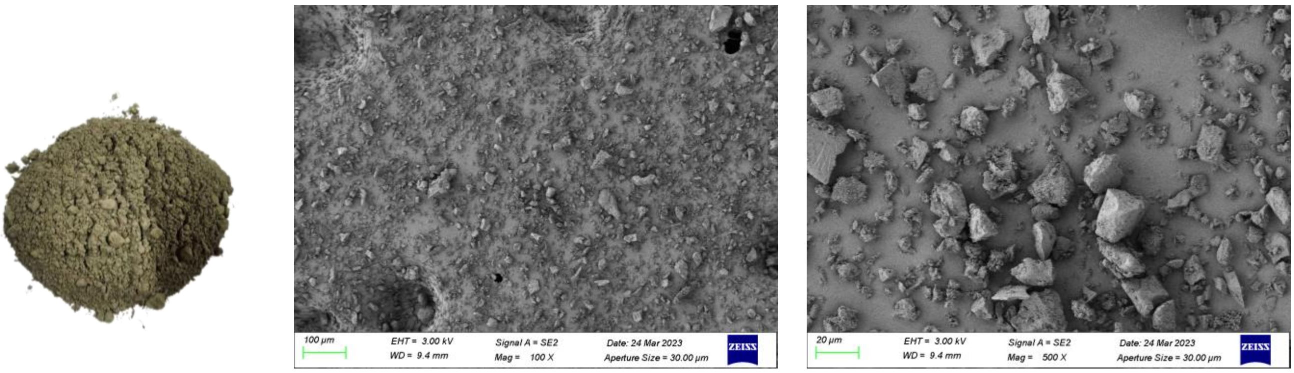
(a) (b)

Figure 2. High-water-material A and SEM images. (a) 100×; (b) 500×.



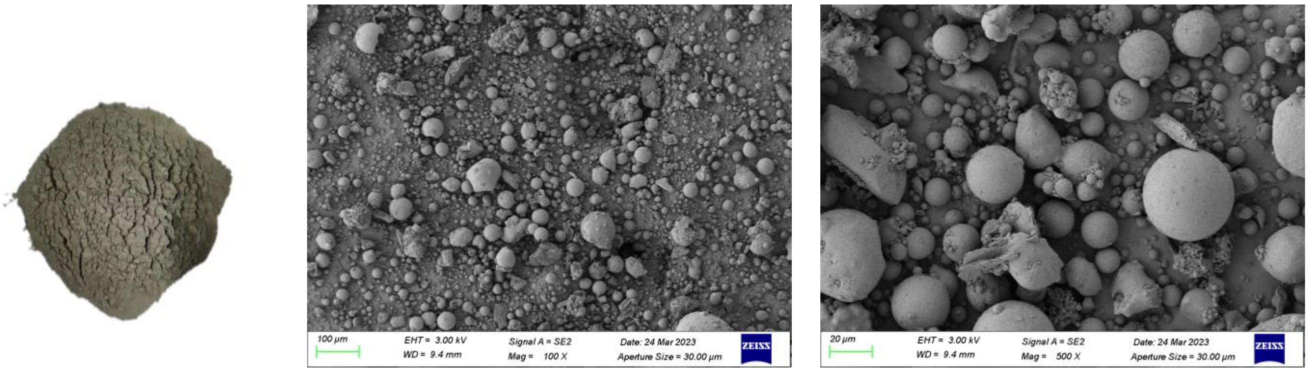
(a) (b)

Figure 3. High-water-material B and SEM images. (a) 100×; (b) 500×



(a) (b)

Figure 4. Cement and SEM images. (a) 100×; (b) 500×



(a) (b)

Figure 5. Fly ash and SEM images. (a) 100×; (b) 500×.

Table 1. Factor(designed in proportion to mass)

Levels	**A. Mass concentration of slime (A)**	**B. Content of high-water-content material (B/A)**	**C. Cement content (C/A)**	**D. Content of fly ash (D/A)**
1	0.4000	0.0300	0.04	0.04
2	0.3375	0.0375	0.08	0.08
3	0.2875	0.0450	0.12	0.12

The material prepared at the room temperature of 25 ℃ was poured in a standard triplet mold measuring 70.7 mm × 70.7 mm × 70.7 mm. After initial setting and demolding, the specimens were placed in a standard YH-60B curing box to be cured for 28 d. After 28 d, the specimens were removed to measure their ultrasonic p-wave velocities, followed by measurement of the uniaxial compressive strength and uniaxial shear strength (Table 2). The ultrasonic p-wave velocities were measured using a HICHANCE HC-U81 concrete ultrasonic detector, and the strength was tested on an MTS electro-hydraulic servo-motor machine.

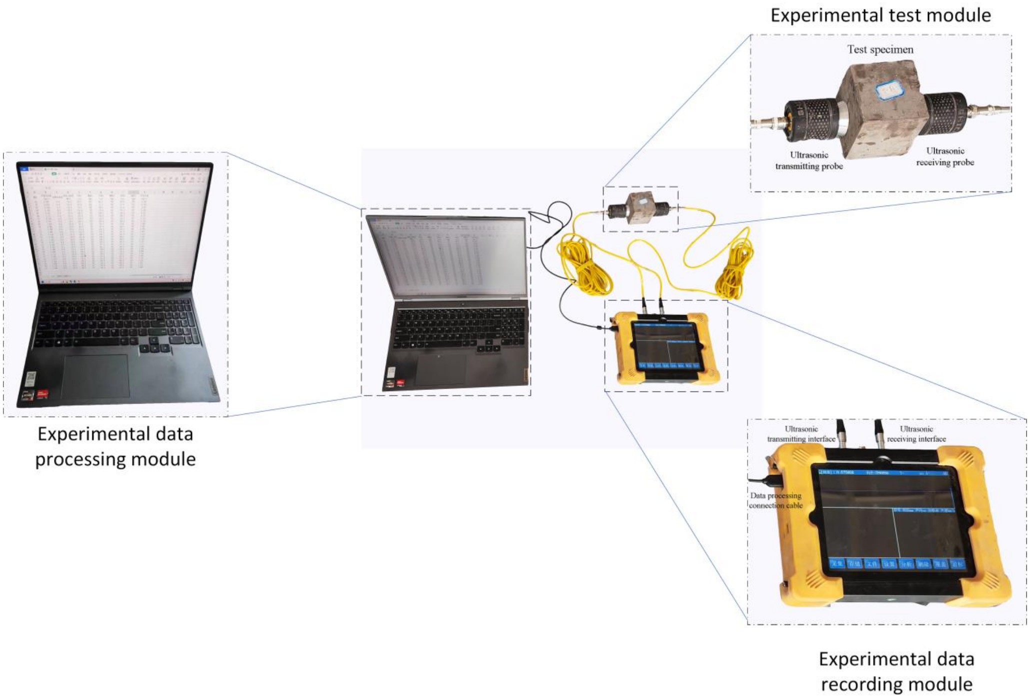
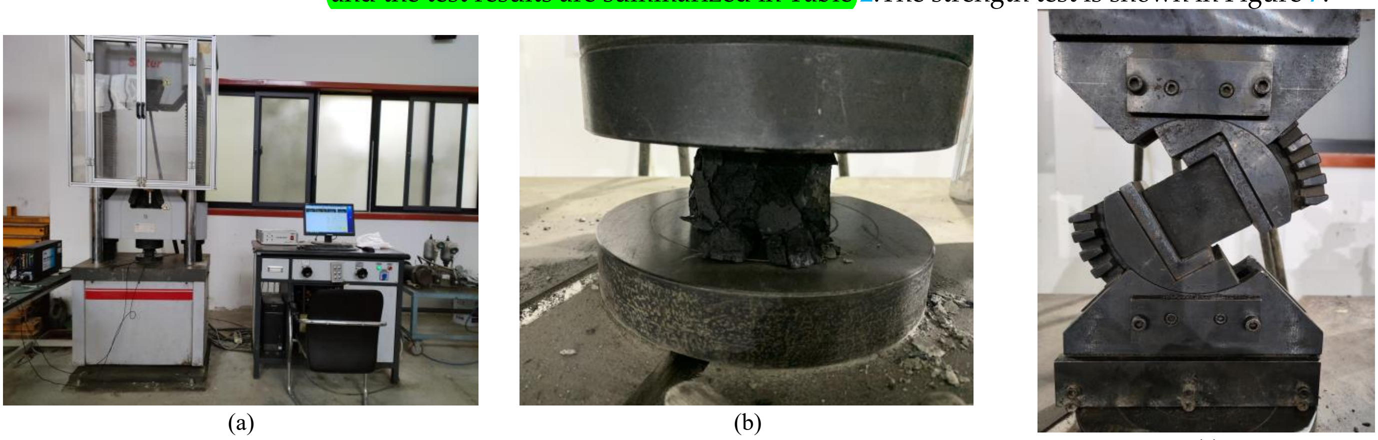


Figure 6. Ultrasonic p-wave velocity measurement of specimens

The ultrasonic p-wave velocity was measured using the following method: a standard specimen was put between two probes coated with the coupling agent. One of the probes was used as the pulsed acoustic emission source and the other as the pulsed acoustic receiver. Results of the received pulses were displayed on the screen of a microcomputer equipped with an ultrasonic detector. After finishing all tests, the computer was used to process the measurement data. If the long axis along the height of the specimen is L and the arrival time is t, then the ultrasonic p-wave velocity V in the specimen is given by L/t, as shown in Figure 6. Each specimen was tested three times and the arithmetic mean was adopted as the final test value. The ultrasonic testing is illustrated in Figure 6 and the test results are summarized in Table 2.The strength test is shown in Figure 7.



(a) (b) (c)

Figure 7. Strength test of specimens.(a) MTS electro-hydraulic servo-motor testing machine; (b) Compression tests; (c) Shear tests.

Table 2. Summary of orthogonal test results

Influencing factors	Test results

Test groups	**A**	**B**	**C**	**D**	**Wave velocity/ km·s⁻¹**	**Compressive strength/ MPa**	**Shear strength/ MPa**
1	0.400	0.0300	0.04	0.04	5.992	0.107	0.056
2	0.400	0.0375	0.08	0.08	5.783	0.161	0.095
3	0.400	0.0450	0.12	0.12	8.319	0.234	0.369
4	0.3375	0.0300	0.08	0.12	5.205	0.247	0.109
5	0.3375	0.0375	0.12	0.04	3.916	0.086	0.043
6	0.3375	0.0450	0.04	0.08	4.706	0.076	0.052
7	0.2875	0.0300	0.12	0.08	5.741	0.115	0.139
8	0.2875	0.0375	0.04	0.12	3.936	0.102	0.036
9	0.2875	0.0450	0.08	0.04	4.076	0.105	0.033

Table 3. Ranged analysis of test data

Factor	**Ultrasonic p-wave velocity/ km·s⁻¹**			
	A	**B**	**C**	**D**
k1	6.698	5.646	4.878	4.661
k2	4.609	4.545	5.021	5.41
k3	4.584	5.700	5.992	5.82
R	2.114	1.155	1.114	1.159

As shown in Table 2, the p-wave velocity, compressive strength, and shear strength of the slime-based filling materials are 3.916 ~ 8.319 km/s, 0.076 ~ 0.247 MPa, and 0.033 ~ 0.139 MPa, respectively, each of which is distributed across a broad range. Range analysis was performed on the test results. Data in Table 3 indicate that various factors are listed in descending order as A, D, B, and C according to their influences on the p-wave velocity.

3 Correlation between strength and p-wave velocity

3.1 Correlation between compressive strength and p-wave velocity

Variations in the ultrasonic p-wave velocity in the filling materials are correlated with the strength of the filling materials. Generally, the strength of filling materials is related to their density and texture. The ultrasonic propagation speed in different materials is related to the material density, elastic modulus, and internal defects.

Therefore, variations in the ultrasonic p-wave velocity and strength of slime-based filling materials were subjected to correlation analysis. In correlation analysis, the correlation coefficient is commonly used to measure how closely variables are correlated. The most common correlation coefficient is the Pearson correlation coefficient, which measures the linear correlation between two variables. The Pearson correlation coefficient lies within the range from -1 to 1. A Pearson correlation coefficient approaching 1 or -1 indicates positive or negative correlations, while it means that there is no correlation if the coefficient approximates to 0.

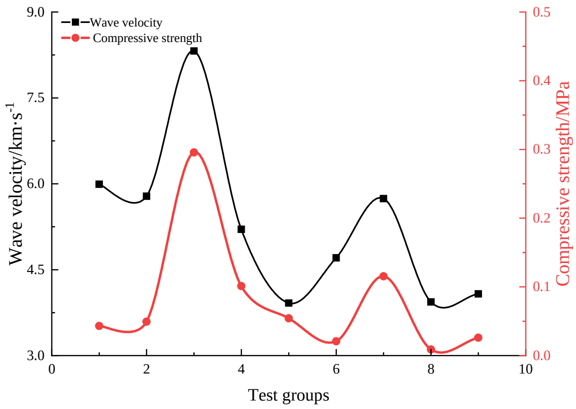


Figure 8. Changes in the ultrasonic p-wave velocity and compressive strength under different mix proportions.

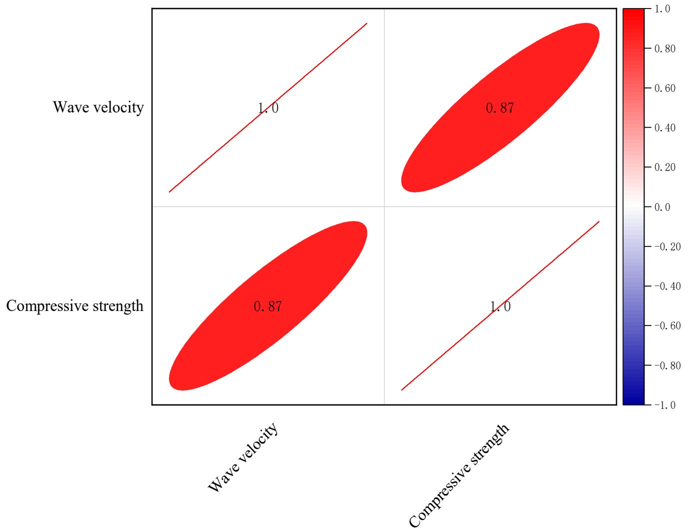
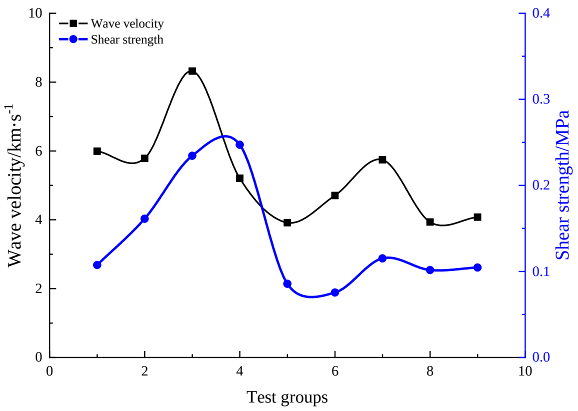


Figure 9. Heat map for the correlation coefficient between the ultrasonic p-wave velocity and compressive strength.

As shown in Figure 8, the ultrasonic p-wave velocity varies in a manner similar to the strength of the filling materials. In the first several groups of tests, the ultrasonic p-wave velocity increases with the strength. From the third to the sixth group, the strength and ultrasonic p-wave velocity begin to decline simultaneously, and they decrease at the same time after small increments in the seventh group of tests. After correlation analysis on the ultrasonic p-wave velocity and compressive strength, a correlation heat map was drawn (Figure 9). In the figure, the color of ellipses indicates the symbol of correlation coefficients: red indicates that two factors are positively correlated, and the more oblate the ellipse is, the higher the correlation. The correlation coefficient between the ultrasonic p-wave velocity and compressive strength is 0.87, which means that they have a strong positive correlation. These results suggest that in these tests, specimens with a high (low) ultrasonic p-wave velocity generally show a high (low) compressive strength.

3.2 Correlation between shear strength and ultrasonic p-wave velocity

Figure 10 shows that the ultrasonic p-wave velocity changes with the shear strength of the filling materials. In the first several groups of tests, the ultrasonic p-wave velocity increases with the shear strength. From the third to the sixth group, the strength and wave velocity begin to decrease at the same time; in the seventh group of tests, they decrease simultaneously after slight increments.



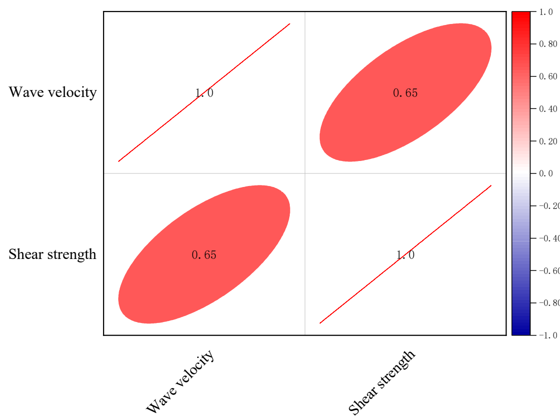


Figure 10. Changes in the ultrasonic p-wave velocity and shear strength under different mix proportions.

Figure 11. Heat map for the correlation coefficient between the ultrasonic p-wave velocity and shear strength

As can be seen from Figure 11,the ultrasonic p-wave velocity and the shear strength have a correlation coefficient of 0.65, indicative of a positive correlation, suggesting that, in these tests, specimens with a high (low) ultrasonic p-wave velocity generally show a high (low) shear strength.

4 Influences of mix proportions of materials on the ultrasonic p-wave velocity

4.1 Factors influencing the ultrasonic p-wave velocity

Based on orthogonal test results, influences of multiple factors on the ultrasonic p-wave velocity are displayed in Figure 12. Various factors are found to differ in their influences on the ultrasonic p-wave velocity. According to analysis of test results, the content of fly ash was listed in the error row and significance testing was conducted at each level. The results of analysis of variance (ANOVA) are listed in Table 4.

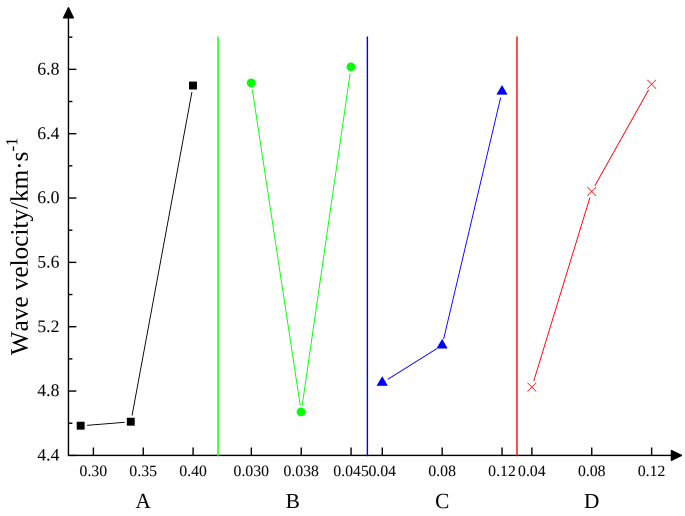


Figure 12. Relationship between the ultrasonic p-wave velocity and multiple factors.

Table 4. Analysis of variance of test data

Source of variation	Sum of squared deviations	Degree of freedom	Variance	F-value	Fα	Significant level
A	8.8312	2	4.4156	3.884		*[1]
B	2.5488	2	1.2744		F0.01(2,6)=10.925	
C	2.2027	2	1.1013		F0.05(2,6)=5.143	
Error e	2.0701	2	1.0351		F0.1(2,6)=3.463	
Correction error e	6.8216	6	1.1369		F0.25(2,6)=1.762	
Sum	15.653					

[1] "*" in the table means that the factor has a certain impact on the wave speed

ANOVA shows that, within a given range, factor A significantly influences the ultrasonic p-wave velocity, while factors B and C exert slight influences. The values of variance suggest that factor B exerts a greater influence than factor C, and the mass concentration of slime is the main factor affecting the ultrasonic p-wave velocity of specimens.

5. Parametric analysis model of ultrasonic p-wave velocity

5.1 Variation of characteristic parameters under the influences of single factors

Influences of various factors on the ultrasonic p-wave velocity of specimens were revealed through the above tests with different mix proportions. The results are displayed in Figure 13. The ultrasonic p-wave velocity tends to decrease, then increase with the increments of factors A and B, while it increases with factors C and D.

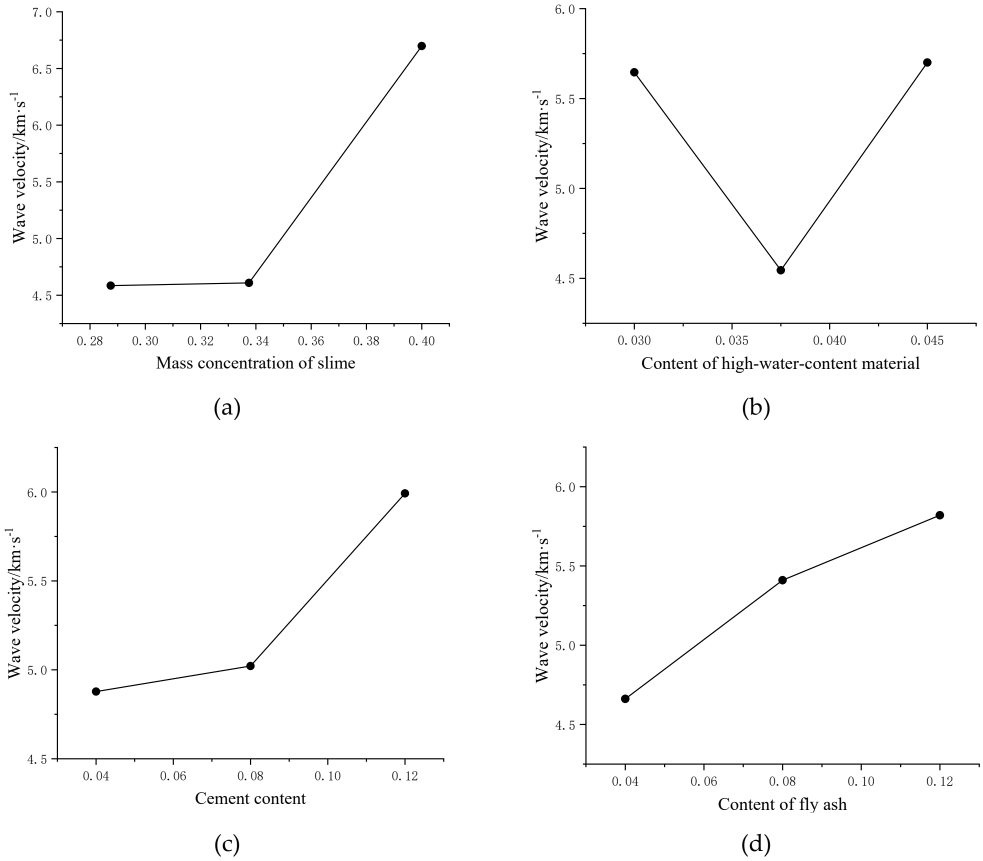


Figure 13. Ultrasonic p-wave velocities of materials under influences of different factors.(a)The effects of coal slime; (b)The effects of high-water-content material; (c)The effects of cement; (d)The effects of fly ash.

5.2 Parametric predictive model

Various influencing factors and parametric curves of materials in Figure 13 were fitted. In this way, the relationships of the mass concentration of slime, content of high-water-content material, cement content, and content of fly ash with the ultrasonic p-wave velocity were acquired (Table 5).

Table 5. Fitting relationship

Factor	Index	Fit relation	General formula	Degree of fit R^2
Mass concentration of slime(P_A)	Wave velocity	$v_1 = 292.71704 P_A^2 - 182.45481 P_A + 32.8452$	$v_1 = aP_A^2 + bP_A + c$	0.99
High-water content(P_B)	Wave velocity	$v_2 = 5.67317 - 1.12815 e^{-\frac{(P_B-0.0375)^2}{2\times0.00299^2}}$	$v_2 = y_0 + Ae^{-\frac{(P_B-x_c)^2}{2w^2}}$	0.99
Cement content(P_C)	Wave velocity	$v_3 = 4.85317 + 0.02483 e^{(P_D-0.04)/0.02091}$	$v_2 = y_0 + A_1 e^{(P_C-x_0)/t_1}$	0.99

Fly ash content(P_D)	Wave velocity	$v_4 = 6.31636 - 3.0221 e^{-P_D/0.06643}$	$v_2 = y_0 + A_1 e^{-P_D/t_1}$	0.99

The data show that the ultrasonic p-wave velocity has a quadratic polynomial relationship with factor A, while it follows an exponential relationship with factors B, C, and D. The fitted curves of the four factors were plotted, as illustrated in Figure 14.

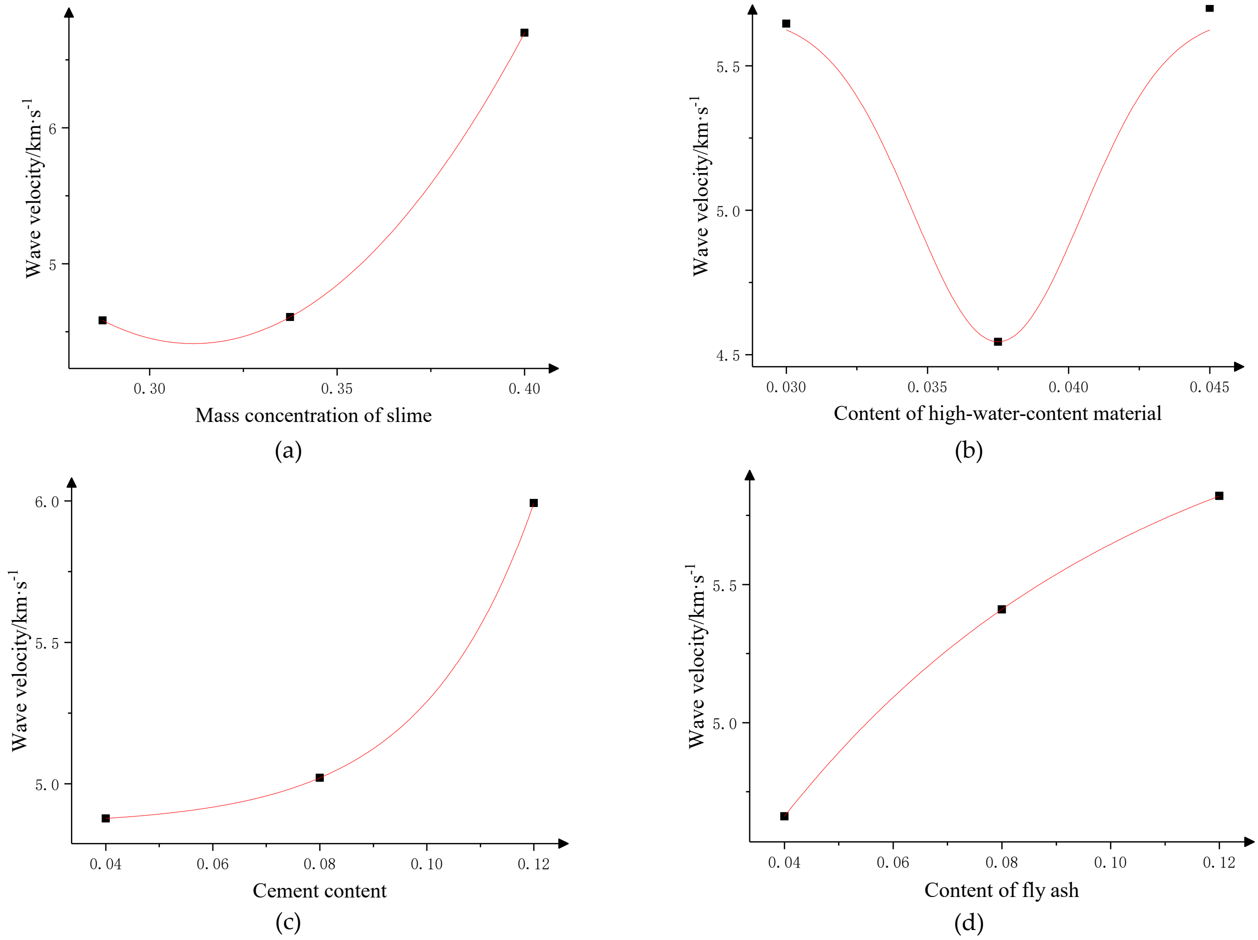


Figure 14. For the wave velocity fitting curves of different factors.(a)The effects of coal slime; (b)The effects of high-water-content material; (c)The effects of cement; (d)The effects of fly ash.

5.3 Establishment of the predictive model

According to relationships of the ultrasonic p-wave velocity with various factors, a synthetic forecasting model for characteristic parameters of the material was constructed combining with the principle for developing multiple linear models, as expressed by Formula (1).

$$\{v = a_1 v_1 + a_2 v_2 + a_3 v_3 + a_4 v_4 + a_5 \quad (1)$$

According to numerous test results pertaining to each of the characteristic parameters, Eq.(1) is subjected to multiple linear regression, thus acquiring the relationships of the ultrasonic p-wave velocity with various factors, as expressed by Formula (2).

$$\{v = 0.97093\, v_1 + 1.00732\, 4v_2 + 0.95037\, v_3 + 0.99147\, v_4 - 15.43465 \quad (2)$$

5.4 Verification of the synthetic forecasting model

Formula (2) was verified: the filling material slurry and specimens were prepared by selecting two groups of mix proportions (Table 6), the ultrasonic p-wave velocity of which was measured. The predicted values obtained by using the predictive model were compared with the test values (comparisons of results and errors are listed in Table 7).

Table 6. Design of verification schemes

The specimen number	A.Mass concentration of slime	B.Content of high-water-content material	C.Cement content	D.Content of fly ash
C1	0.35	0.037	0.06	0.10
C2	0.31	0.039	0.07	0.11

Table 7. Analysis of verification results

The specimen number	Materials	Wave velocity/ km·s^{-1}
C1	Predicted value	4.133
	Experimental value	4.324
	Error /%	4.413%
C2	Predicted value	3.965
	Experimental value	4.231
	Error /%	6.270%

Analysis unveils that the parameters calculated using the synthetic forecasting model well conform to test values. The relative errors of various indices are in a range of 4% to 6%, with an average value of 5.3%.

6. Conclusions

(i) Orthogonal tests of the ultrasonic p-wave velocity were conducted on four factors including the mass concentration of slime, content of high-water-content material, cement content, and content of fly ash that were set at three levels using the orthogonal test method. Afterwards, analysis shows that the ultrasonic p-wave velocity of the slime-based filling materials is in the range of 3.916 ~ 8.319 km/s.

(ii) Various factors are listed in descending order as A, D, B, and C according to their influences on the ultrasonic p-wave velocity, suggesting that the mass concentration of slime and content of fly ash are main factors influencing the diffusivity.

(iii) The mathematical relationships of factors A, B, C, and D with the ultrasonic p-wave velocity were fitted. The wave velocity has a quadratic polynomial relationship with factor A while being governed by exponential relationships with factors B, C, and D. The synthetic forecasting model was obtained through the multiple regression equation, and the ultrasonic p-wave velocity calculated thereby has an average error of 5.3% with the test data.

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References

1. An, B.F.; Yi, Q.M.; Zhao, X.; Yu, W.J.; Wang, D.D. Experimental study on fluidity and strength characteristics of slime based filling materials. *J. Coal Sci. Technol*, **2023,** 2022-1733
2. Xiang, J.Z.;Song, H.; Leng, M.H.; Gui, F.L. Effect of aggregate particle size on ultrasonic wave velocity of pervious concrete. *J. Concretei*, **2022**, 396, 106-111.
3. Cheng, X.H.; Song, D.Z.; Qiu, L.M.; Wang, H.L.; Wang, A.H. Study on coupling relationship between longitudinal wave velocity and stress in gas outburst coal. *J. Coal mine safe*, **2021**, 52, 36-41
4. Wang, X.J.; Zeng, J.; Qin, Y.H.; Li, C.L. Experimental study on filling mass measurement based on acoustic wave velocity. *J. Sci.Technol.Eng*, **2007**, 23, 6180-6181+6186.
5. Mu, G.C.; Deng, F.; Lai, Z.H. Study on the relationship between the strength of cemented backfill and ultrasonic wave velocity. *J. Chem. Miner. Proce.* **2020**, 49, 1-3+53.
6. Cheng, A.P.; Dong, F.S.; Zhang, Y.S.; Wang, P.; Dai, S.Y. Cemented filling body wave - density double parameters strength prediction model the study. *J. Metal Mine*, **2019**, 517, 26-31.
7. Bayram Ercikdi, Tekin Yilmaz, Gokhan Kulekci. Strengthand ultrasonic properties of cemented paste backfill. *J. Ultrasonics*, **2014**, 54, 195-204.
8. Gao, H.; Hao, W.X.; Li, L.; Wang, J.L.; Sun, J.H. Experimental study on working stress state detection of concrete by ultrasonic method. *J. Build. Sci*, **2017**, 33, 94-99.
9. Gao, H.; Hao, W.X.; Bi, C.K.; Sun, J.H. Experimental study on combined ultrasonic and acoustic emission characteristics of concrete under uniaxial loading. *J. Agric. Univ. Hebei*, **2017**, 40, 119-123.
10. Xu, X.L.; Zhang, R.; Dai, F.; Yu, B.; Gao, Mi.Z. Experimental study on the influence of coal and rock characteristics on ultrasonic wave velocity. *J. China Coal Soc.*, **2015**, 40, 793-800.
11. Li, S.H.; Study on strength and acoustic characteristics of stratified cemented backfill. Master's thesis, Jiangxi University Of Science And Technology, Ganzhou City, Jiangxi Province, 2021.
12. Cheng, A.P.; Zhang, Y.S.; Dai, S.Y.; Dong, F.S. Ultrasonic wave velocity damage evolution test of cemented backfill. *J. Metal Mine*, **2018**, 507, 41-46.
13. Yu, K.P.; Ren, F.Y,; Liu, D.; Study on ultrasonic test and compressive strength test of cemented backfill. *J. Min. Res. Dev.* **2020**, 40, 38-42.
14. Yilmaz T,Ercikdi B. Predicting the uniaxial compressive strength of cemented paste backfill from ultrasonic pulsevelocity test. *J. Nondestruct. Test. Eval.* **2016**, 31, 247-266
15. Gregor Trtnik,Franci Kavcit,Goran Turk. Prediction of concrete strength using ultrasonic pulse velocity and artificialneural networks .*J. Ultrasonics*,**2009**,499,53-60
16. Wang, Q. Bearing performance characterization and prediction of lane side filling concrete based on ultrasonic wave. Master's thesis, Xi`an University Of Science And Technology, Xi 'an City, Shaanxi Province, 2022.
17. Feng, G.R.; Li, Q.D.; Qi, T.Y.; Guo, Y.X.; Ran, H.Y. Evaluation of static anti-segregation performance of gangue by ultrasonic method. *J. Min. Saf. Eng.*, **2019**, 37, 1231-1237.
18. Wu, J.Y.; Macro and Micro mechanical properties and creep model of cemented backfill. Doctoral thesis, China University of Mining and Technology, Xuzhou City, Jiangsu Province, 2020.
19. Xu, M.F.; Gao, Y.T.; Jin, A.B.; Zhou, Y.; Guo, L.J. Strength prediction of cemented backfill based on ultrasonic wave velocity and BP neural network. *J. Chin. J. Eng.*,**2016**, 38, 1059-1068.
20. CHANDRAPPA A K; BILICIRI K P.Influence of mix parameters onpore properties and modulus of pervious concrete :an application ofultrasonic pulse velocity. *J. Materials & Structures*, **2016**, 49, 5255-5271.

21. Wang, X.P.; Liu, S.R.; Yang, J.J.; Wang, Z.Z.; Rong, H.; Zhang, L. Experimental study on the relationship between ultrasonic wave velocity and compressive strength of concrete. *J. Concrete*, **2015,** 12, 34-37.

22. Cheng, Z.X.; Xu, Y.F.; Wang, Y.X. Development of the strength by ultrasonic method. *J. Huazhong Univ. Sci. Technol*, **2003**, 20, 95-98.

23. Zheng, B.C.; Zhou, H.Q.; He, R.J. Experimental study on the filling material of coal gangue paste. *J. Min. Saf. Eng*, **2006,** 04, 460-463.

24. Shi, J.W.; Wei, Z.J.; Liu, Q.L.;Li, Y.Y.; Chen, J.T. Study on the ratio optimization of filling materials for coal mine safety mining based on orthogonal test. *J. Chin. J. Saf. Sci*. **2011**, 21, 111-115.

25. Sun, Q.W.; Zhu, H.; Cui, Z.L. Study on the preparation and properties of fly ash-gangue based cement filling material. *J. Chinese Journal of Safety Science*, **2012**, 22, 74-80.