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PREPARATION AND STUDY OF A PU-MODIFIED EPOXY RESIN COLD PATCHING MIXTURE

PRIPRAVA IN RAZISKAVA EPOKSIDNE MEŠANICE MODIFICIRANE S POLIURETANSKIM PREDPOLIMEROM ZA HLADNO KRPANJE LUKENJ

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Focusing on the problems of low strength and insufficient water stability of the cold patch mixture for asphalt pavement, a polyether polyurethane prepolymer with high activity was prepared in this experiment, and then the epoxy resin was modified with the prepolymer to prepare a kind of epoxy-resin-mixture pothole-repair material with high strength and high toughness. The content of polyurethane prepolymer was optimized using a low-temperature tensile test and a normal-environment-condition mechanical test, and the mechanical properties and road performance of the PU-modified epoxy-resin mixture were studied with a rutting test, bending test, Cantabro scattering and a splitting test. The results show that PU prepolymer-modified epoxy resin can effectively increase the toughness and strength of the binder, and that the toughness and mechanical properties of 20 phr PU prepolymer are the best. The road performance of the PU-modified epoxy-resin cold patch mixture is excellent. There is little difference between the immersion scattering loss rate and the standard loss rate, and both are less than 4 %, indicating that the mixture has excellent anti-scattering and water-loss performance. It is very suitable as a cold patch material for pavement pits. Due to its comprehensive road performance and reasonable price, the use of the PU-modified epoxy resin binder content of 4 % is recommended.

Keywords: asphalt pavement, pothole repair, polyurethane prepolymer, epoxy resin, road performance

V članku je opisano reševanje problemov, ki se nanašajo na slabo trnost in nezadovoljivo odpornost proti vodi oz. slabo stabilnost mešanic za krpanje lukenj v asfaltiranih pločnikih in cestah. V ta namen so pripravili poliuretanski predpolimer (PU) z visoko aktivnostjo. Epoksidno smolo so modificirali s poliuretanskim predpolimerom z namenom pridobitve materiala z visoko trdnostjo in žilavostjo, primernega za krpanje lukenj na asfaltiranih cestah in pločnikih. Vsebnost poliuretanskega predpolimera so optimizirali s pomočjo eksperimentalnih preizkusov določevanja natezne trdnosti pri nizkih in normalnih temperaturah okolice, kakor tudi določevanja mehanskih lastnosti povezanih s karakteristikami cestišč. Epoksidno mešanico modificirano s poliuretanskim predpolimerom so analizirali s pomočjo testov izmeničnega tlačnega in upogibnega obremenjevanja, testa abrazivne obrabe (Cantabro test) in cepilnega testa. Rezultati so pokazali, da epoksidna smola modificirana s poliuretanskim predpolimerom učinkovito poveča žilavost in trdnost veziva. Najboljše mehanske lastnosti so dosegli z 20 phr poliuretanskim predpolimerom. Stabilnost izdelane mešanice za hladno krpanje lukenj na cestišču je bila odlična. Opazili so rahlo razliko med abrazivnimi izgubami med potaplajanjem in standardnimi izgubami zaradi obrabe (v obeh primerih manj kot 4 %), kar kaže na to da ima izdelana mešanica dobro odpornost proti izpiranju z vodo. Mešanica je tako zelo primerna tudi za hladno krpanje lukenj nastalih na asfaltiranih pločnikih. Tudi cenovno je izdelana mešanica s 4 % veziva sprejemljiva glede na dosežene lastnosti popravljenega cestišča.

Ključne besede: asfaltni pločnik; popravilo lukenj na cestah; poliuretanski predpolimer; epoksidna smola; lastnosti cestišča

1 INTRODUCTION

Asphalt pavement is a common form of pavement. However, the temperature stability of asphalt is poor, and asphalt pavement is susceptible to cracks, potholes and ruts due to the influence of external environment. Pothole is typical damage of asphalt pavement with a great influence on the pavement performance and driving safety. Once a pothole is formed, it must be repaired in time.[1,2]

Asphalt pavement potholes are one of the most common and difficult problems of daily maintenance. Especially in the low-temperature and rainy season, conventional hot mixes cannot repair potholes in a timely and effective manner,[3] so the repair material used is still mainly cold patch material. However, in practical applications, it is found that the potholes repaired with a cold patch mixture have low forming strength under traffic load, insufficient durability, and are prone to loosening again, thus often requiring new repairs.[4,5] Therefore, a high-performance pothole repair material is urgently needed in the field of road engineering.

Epoxy resin materials exhibit advantages such as good chemical stability, strong viscosity, high mechanical strength and low shrinkage due to their unique three-dimensional network structure and special functional groups.[6,7] However, a cured epoxy resin adhesive has insufficient toughness and poor impact resistance, which limit the application of epoxy resin in road con-

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crete.[8] Therefore, a toughening modification of epoxy resin is an important way of improving its comprehensiveness and expand its application. Polyurethane materials are widely used in road engineering due to their excellent physical and chemical properties.[9,10] Polyurethane also has a unique three-dimensional network structure of a polymer. Because of this special structure polyurethane can have a toughening effect due to the absorption and dispersion of energy,[11,12] while polyurethane and epoxy resin exhibit compatibility so that their mixture can modify and improve the toughness of a cured epoxy resin adhesive.[13] M. Liu et al.[14] prepared a PU prepolymer-modified epoxy polymer with the microcapsule technology and polymer reinforcement technology. It was found that the cold patch material had a good repair effect in winter and rainy seasons, and could allow rapid repair of pits under low temperatures and humid conditions. Therefore, PU prepolymer-modified epoxy resin can be considered as a cold patch raw material for research.

In this research, a polyether polyurethane prepolymer with high activity was first prepared with the blending method, and then the epoxy resin was modified with the prepolymer to obtain a modified epoxy resin with a polyurethane branched-chain structure. At the same time, appropriate curing agents, a diluent and toughening agents were selected to obtain a kind of asphalt-mixture pothole repair material with high strength and high toughness. The mechanical properties and road performance of the PU(polyurethane)-modified epoxy resin mixture were studied with a rutting test, bending test, Cantabro test, and splitting test. This material has an important engineering practical value and broad application prospects, promoting the development of the asphalt pavement maintenance technology, improving pavement performance, extending pavement service life, and reducing pavement maintenance costs.

2 RAW MATERIALS AND TESTING

2.1 Preparation of PU-prepolymer

First, a certain amount of polyether polyols was weighed, heated in an oil bath to 100 °C, and stirred for 1 h at a stirring rate of 80 min^{-1}; then methylene diphenyl diisocyanate (MDI) was added under the protection of nitrogen, and after cooling down to room temperature the mixture was kept for 1 h at a constant temperature; finally, a certain amount of chain extender butanediol and crosslinking agent trimethylolpropane were added and stirred evenly to prepare the thermoplastic PU-prepolymer.

2.2 Preparation of the PU-modified epoxy resin binder

In accordance with the specific ratio, a certain amount of epoxy resin E-51 was weighed, PU prepolymer was added, and PU-modified epoxy resin was prepared by heating the mixture at 80 °C for 2 h. Then the butyl glycidyl ether active diluent, polythiol curing agent, and catalyst DMP-30 were added, and the PU-modified epoxy resin binder was prepared by stirring. The obtained binder was PU-modified epoxy resin binder.

2.3 Preparation of the PU-modified epoxy resin cold patch mixture

The gradation design (AC-13) shown in **Figure 1** was adopted. The coarse and fine aggregates were limestone, and the mineral powder was a ground limestone powder, which was stirred evenly to prepare a gravel mixture. Then the prepared PU-modified epoxy resin binder and gravel mixture were stirred for 6 min, and finally the PU-modified epoxy resin mixture was obtained.

2.4 Preparation of the PU-prepolymer

Dumbbell tensile specimens were prepared according to T 2567-2008. The prepared PU-modified epoxy resin binder was slowly poured into a mould coated with a release agent, and demoulded after curing. The tensile test was carried out with a universal testing machine at a temperature of –10 °C and a tensile speed of 5 mm/min. Three parallel specimens were used in each group, and the test results were averaged.

2.5 Uniaxial compression and oblique shear tests

According to T 0713-2000, a cylindrical specimen with a diameter of 100 mm and a height of 100 mm was formed with the static pressure method. After forming, the demoulded specimen was kept in an environmental box at 25 °C until it was completely cured. Then the specimen was placed in a constant-temperature water tank at 15 °C for 4 h. Finally, a uniaxial compression test was carried out using the universal testing machine. The loading rate was 2 mm/min and the test temperature was

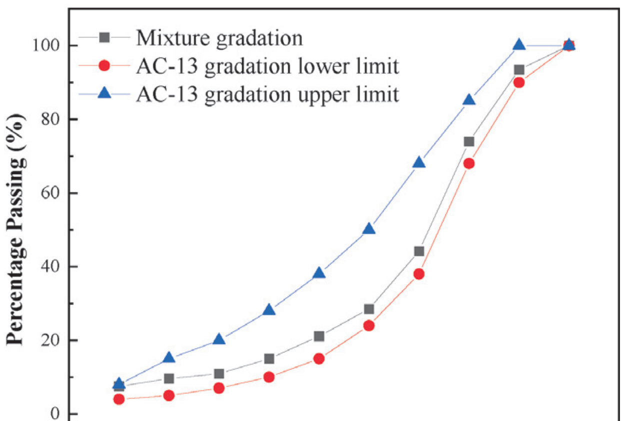

Figure 1: Mixture gradation curve

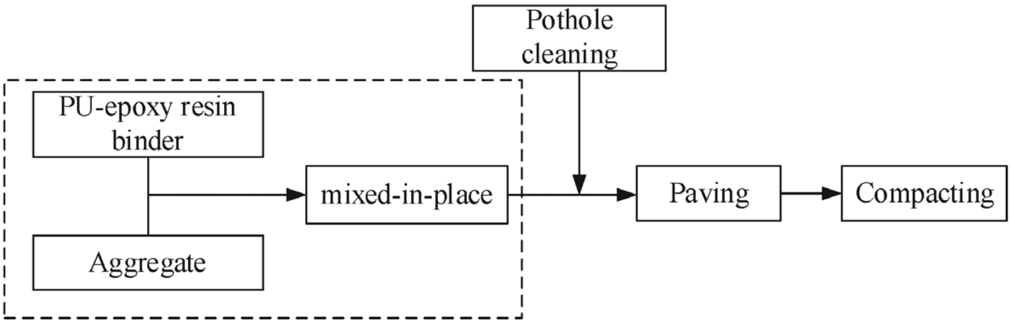

Figure 2: Construction of PU-modified epoxy resin cold patch mixture

15 °C. Three parallel specimens were used in each group, and the test results were averaged.

The oblique shear test was used to cut an asphalt concrete rut plate specimen into (50 × 50 × 50) mm cube specimens. The roll-formed surfaces of two cube specimens were spliced, and a 5-mm crack was kept in the middle so that the PU-modified epoxy resin binder was poured into it. After the binder was completely cured, the specimen was placed in the environmental box at 25 °C for 4 h. The 45 ° oblique shear test was used to test the interfacial shear-bonding performance. The test temperature was 25 °C and the loading rate was 10 mm/min.

2.6 Rutting test

According to T 0719-2011, another rut-plate specimen (300 × 300 × 50) mm was formed and cured in the 25 °C environmental box for 28 d. The test temperature was 60 °C, the wheel pressure was 0.7 MPa, and the dynamic stability of the mixture specimen was calculated after the rutting deformation formed in 45 min and 60 min.

2.7 Low-temperature bending test

According to T 0715-2011, rutting-plate specimens were formed and cured at 25 °C for 28 days. After a rutting plate was demoulded, it was cut into (250 × 30 × 35) mm trabecular specimens and kept at –10 °C for 4 h. The test temperature was –10 °C and the loading rate was 50 mm/min.

2.8 Splitting test

Standard Marshall specimens were prepared according to T 0716-2011. They were compacted 50 times on both sides. The diameter of the specimens was ϕ101.6 ± 0.2 mm and the height was 63.5 mm ± 1.3 mm. A Marshall test block was immersed in water for 1 h and then split by the universal testing machine.

2.9 Cantabro test

According to T 0733-2011, the Marshall specimens were immersed in the constant-temperature water tank at 20 °C for 20 h. During a standard scattering test, the specimens were immersed in the constant-temperature water tank at 60 °C for 48 h, and then kept at room temperature for 24 h. The Los Angeles wear test was used to test the standard-scattering loss rate and immersion-scattering loss rate. The Los Angeles testing machine made 300 revolutions at a speed of 32 min^{-1}.

2.10 Construction process

The construction process for the prepared pothole cold filling mixture is shown in **Figure 2**.

3 RESULT AND DISCUSSION

3.1 Determination of PU Content

According to the previous experiments of the research team, the amount of epoxy resin was determined to be 100 phr, the amount of the curing agent was 50 phr, the amount of the diluent was 60 phr, and the amount of the accelerator was 4 phr. Considering the tensile strength and elongation at break at –10 °C (**Figure 3**) and the compressive strength and oblique shear bond strength at 25 °C (**Figure 4**), the amount of PU was optimized.

It can be seen from **Figure 3** that with an increase in the polyurethane prepolymer content, the tensile strength

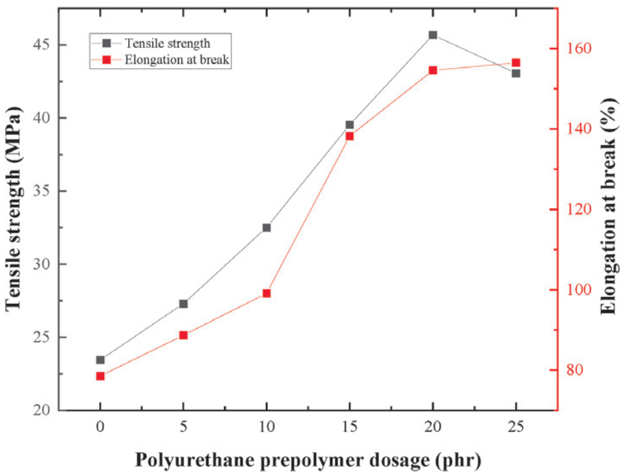

Figure 3: Tensile strength and elongation at break at –10 °C

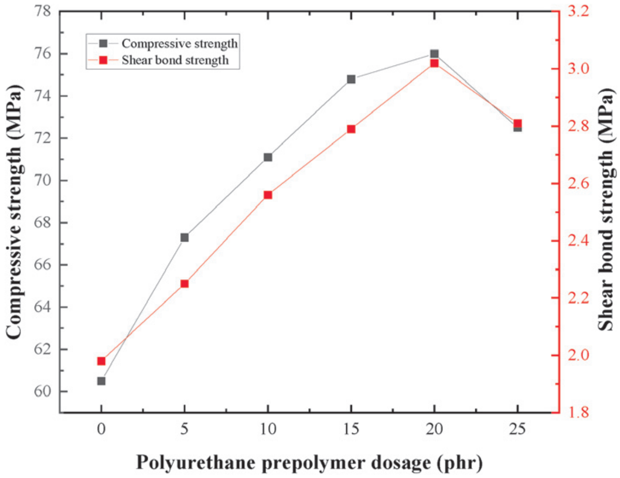

Figure 4: Compressive strength and oblique shear bond strength

of the binder increases first and then decreases. When the amount of polyurethane prepolymer is 20 %, the tensile strength peaks at 45.68 MPa. The elongation at break increases with the content of polyurethane prepolymer, but when the content of polyurethane prepolymer exceeds 20 phr, the elongation at break increases slightly. After adding polyurethane prepolymer-modified epoxy resin, the tensile strength and elongation at break of the binder are greatly improved. Obviously, the unmodified epoxy resin binder shows that the use of polyurethane prepolymer-modified epoxy resin can greatly enhance the toughness of the epoxy resin binder. However, the toughness improvement of adhesive materials should not be at the expense of other mechanical properties. In this research, in order to explore whether the addition of polyurethane prepolymer will reduce the mechanical properties and bonding properties of the binder, the compressive strength and oblique shear bonding strength are used for the analysis.

From **Figure 4**, it can be seen that the compressive strength of the cured product of the unmodified epoxy resin is relatively the lowest, which is 60.5 MPa. After adding polyurethane prepolymer, the compressive strength of the modified epoxy resin is higher than 67 MPa, and the compressive strength increases with the increase in the amount of polyurethane. It first increases and then decreases, reaching a maximum of 76 MPa when the amount of PU is 20 phr. It can also be seen from **Figure 4** that the oblique shear bond strength of the unmodified epoxy resin is 1.98 MPa, which is less than the bond strength of the other samples. At the same time, with the increase in the PU prepolymer content, the oblique shear bond strength of the modified epoxy resin also shows a trend of first increasing and then decreasing, reaching a maximum value of 3.02 MPa when the PU prepolymer content is 20 %. That is, with the improvement in the toughness of the epoxy resin adhesive material, its compressive strength and bond strength are not affected, but they are improved to varying degrees.

Combining the tensile strength and elongation at break at –10 °C and the compressive strength and oblique shear bond strength at 25 °C, it can be seen that the polyurethane prepolymer-modified epoxy resin can effectively increase the toughness and strength of the binder. In addition, the toughness and mechanical properties of the 20 phr PU polyurethane prepolymer are the best, so 20 phr polyurethane is used in this research. Finally, the epoxy resin : PU prepolymer : diluent : curing agent : accelerator ratio of 100 : 20 : 60 : 50 : 4 is used.

3.2 Road performance of the cold patch mixture

3.2.1 High-temperature stability

In order to study the high-temperature performance of the PU-modified epoxy resin cold patch mixture, a 60 °C rutting test was used. The PU-modified epoxy resin binder was 3.0 %, 3.5 %, 4.0 %, 4.5 % and 5.0 %. The test results are shown in **Table 1**.

It can be seen from **Table 1** that within the range of the binder content selected for the test, the deformation of the mixture specimen decreases with the increase in the binder content, and the dynamic stability gradually increases. This is because the increase in the binder improves the overall density of the mixture, while the binding effect of the binder gradually increases. However, when the content exceeds 4 %, the increase in the binder content has no significant effect on the high-temperature performance of the mixture. When the binder content is 3 %, the dynamic stability is 25850 times/mm, which is much larger than the requirement that the dynamic stability of the modified SMA mixture is not less than 3000 times/mm, as specified in the Technical Specification for the Construction of Highway Asphalt Pavement (JTG F40-2004).

It can be seen from the above data that, compared with the asphalt mixture, the PU-modified epoxy resin cold patch mixture exhibits a large dynamic stability and small deformation under high-temperature and load, and

Table 1: Rutting test results for PU-modified epoxy resin cold patch mixture

Binder content (%)		3.0	3.5	4.0	4.5	5.0
45 min deformation (mm)		0.521	0.326	0.235	0.215	0.213
60 min deformation (mm)		0.546	0.334	0.257	0.220	0.219
Dynamic stability (times/mm)		25850	53750	74500	86500	98000
Specification requirements (times/mm)	Normal asphalt mixture	1000				
	Modified SMA asphalt mixture	3000				

has excellent high temperature deformation resistance. This is because asphalt materials are temperature-sensitive thermoplastic materials, exhibiting a soft flow when the temperature rises and being prone to high-temperature rutting damage. The resin material is a thermosetting material, and its curing process is irreversible. Even in high-temperature conditions, there is no flow phenomenon. Therefore, the high temperature stability of the PU-modified epoxy resin cold patch mixture is good.

3.2.2 Low-temperature crack resistance

Asphalt materials are brittle in winter, and transverse cracks in asphalt pavement are mostly caused by low-temperature cracking of asphalt. Therefore, the pavement pit repair material must have low-temperature crack resistance to ensure that a repaired pavement has a certain anti-deformation ability and can avoid low-temperature shrinkage cracks. Therefore, this research uses a low-temperature bending test to evaluate its low-temperature performance with flexural strength and flexural strain. The test results are shown in **Table 2**.

Table 2: Low-temperature bending test results

Binder content (%)	Breaking load (kN)	Mid-deflection (mm)	Bending tensile strength (MPa)	Bending tensile strain (με)	Stiffness modulus (MPa)
3.0	2.917	0.389	23.75	2256	10385
3.5	2.798	0.416	23.01	2455	9850
4.0	2.679	0.452	22.11	2694	8075
4.5	2.501	0.472	21.53	2917	7350
5.0	2.422	0.497	19.89	3018	6780

It can be seen from **Table 2** that the low-temperature flexural tensile strength of the PU-modified epoxy resin cold patch mixture decreases with the increase in the binder content, but it is still 19.89 MPa at the 5 % content. On the one hand, this is due to the high strength of the PU-modified epoxy resin binder; on the other hand, it is due to the strong bonding capacity of the PU-modified epoxy resin binder, and the high bond strength between the aggregates, resulting in a high overall strength of the cold patch mixture.

The flexural strain of the mixture increases with the increase in the binder because the higher the binder content, the thicker is the binder film formed on the surface of the mineral. This fully enhances the toughness of the PU-modified epoxy resin, thereby improving the overall deformation capacity. When the content of the binder is 3 %, the bending strain is the smallest, which is 2256 με, but it still meets the requirement of JTG F40-2004 that the bending strain of an ordinary asphalt mixture in a cold winter region is not less than 2000 με. When the binder content is 4.0 %, the flexural strain is 2694 με, which meets the requirement that the flexural strain of an ordinary asphalt mixture in a cold winter region is not less than 2600 με. It can be seen that when the binder content is 4.0 %, the PU-modified epoxy resin cold patch mixture is suitable for most areas.

3.2.3 Water stability

Traditional pavement cold patch materials such as solvent-based cold patch mixtures and emulsion-based cold patch mixtures use asphalt materials as binders. The presence of water affects the adhesion between the aggregate and asphalt, and generates hydrodynamic pressure under the repeated action of the vehicle load, thereby causing water damage. The pavement itself, when needing to be repaired, is the weakest link in the pavement structure. If secondary damage occurs due to water damage, the performance of the pavement is seriously affected. Therefore, the 25 °C splitting strength and Cantabro scattering loss rate were used to evaluate the water stability and water damage resistance of the PU-modified epoxy resin mixture. The test results are shown in **Table 3**.

Table 3: Splitting strength and scattering loss rate for different mixtures

Binder content (%)	Splitting strength (MPa)	Standard scattering loss rate (%)	Immersion scattering loss rate (%)
3.0	0.98	3.94	3.97
3.5	1.27	3.10	3.18
4.0	1.45	2.53	2.56
4.5	1.51	2.32	2.33
5.0	1.56	2.30	2.30

It can be seen from **Table 3** that the splitting strength of the mixtures with different binder contents after being soaked for 1 h is greater than 0.98 MPa, and the splitting strength gradually increases with the increase in the binder content. When the content of the PU prepolymer is 5 %, the splitting strength of the mixture reaches the maximum (1.56 MPa). This shows that the higher the binder content, the higher is the bond strength of the prepared mixture under water.

It can also be seen from Table 3 that the mass loss rate obtained with the Cantabro standard dispersion test and the immersion dispersion test is less than 4 %, and the dispersion loss rate decreases with the increase in the binder content. This is because the greater the amount of the binder, the thicker is the PU-modified epoxy resin binder film after the curing crosslinking reaction, enhancing the bonding with the aggregate, while water molecules are difficult to aggregate, causing the peeling of the binder. On the whole, there is little difference between the immersion loss rate and the standard loss rate, especially when the binder content is more than 4 % when the immersion loss rate is almost equal to the standard loss rate. This is because the mixture porosity decreases with the increase in the binder content, and due to the high overall density of the mixture, water molecules do not easily enter the interior of the specimen, so the immersion scattering loss rate is close to the standard scattering loss rate. Overall, the PU-modified epoxy resin binder has excellent water stability.

Based on the above results, it can be seen that the PU-modified epoxy resin mixture exhibits excellent road performance and good anti-scattering performance. It can also be used in the rainy season, and it is very suitable for cold patching pavement pits. When the content of the PU-modified epoxy resin binder is 4 %, the performance of the mixture is very cost-effective.

4 CONCLUSIONS

In this research, a polyether polyurethane prepolymer with high activity was prepared, and then epoxy resin was modified with the prepolymer. At the same time, a suitable curing agent, diluent, and toughening agent were selected to prepare an asphalt mixture pothole repair material with high strength and toughness. The content of the polyurethane prepolymer in the binder was optimized through a low-temperature tensile test and room-temperature mechanical test. The mechanical properties and road performance of the PU-modified epoxy resin mixture were studied with a rutting test, bending test, Cantabro scattering and splitting test. The main conclusions are as follows:

(1) Combining the tensile strength and elongation at break at –10 °C and the compressive strength and oblique shear bond strength at 25 °C, it can be seen that the PU prepolymer-modified epoxy resin can effectively increase the toughness and strength of the binder. In addition, the toughness and mechanical properties of the 20 phr content of PU prepolymer are the best. The ratio of the components of the PU-modified epoxy resin binder including epoxy resin : PU prepolymer : diluent : curing agent : accelerator is 100 : 20 : 60 : 50 : 4.

(2) The high-temperature stability of the PU-modified epoxy resin cold patch mixture is better with an increase in the binder content, but when the content exceeds 4 %, the increase has no significant effect on the high-temperature performance of the mixture. The low-temperature flexural tensile strength of the mixture decreases with the increase in the binder content, but it is still 19.89 MPa at the 5 % content. The bending strain increases when the binder content is more than 4 %, so the PU-modified epoxy resin cold patch mixture is suitable for most areas. With the increase in the binder content, the immersion splitting strength of the mixture increases gradually. The Cantabro standard scattering loss and immersion scattering loss decrease with the increase in the binder content. On the whole, there is little difference between the immersion scattering loss rate and standard scattering loss rate, and both are less than 4 %, indicating that the mixture has excellent resistance to scattering and water loss.

(3) The road performance of the PU-modified epoxy resin mixture is excellent, and it also exhibits good anti-scattering performance. It is very suitable as a cold patch for pavement potholes. Due to its comprehensive road performance and reasonable price, we recommend the PU-modified epoxy resin binder content of 4 %.

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