Study on the performance of environmental micro-surfacing for exhaust purification

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Abstract

In order to endow pavement materials with environmental protection properties such as automobile exhaust purification, an environmentally friendly micro-surfacing material with exhaust purification was prepared. Functional materials with photocatalytic activity were prepared by sol-gel method. The best preparation scheme of composite modified asphalt was determined by comparative test. The mix proportion of micro-surfacing mixture was designed to determine the optimal preparation process of micro-surfacing. Based on rutting test, wet wheel wear test and pendulum test, the road performance of protective environmental micro-surfacing with exhaust purification function is studied. An exhaust gas purification test equipment is improved to determine the test method and analyze the exhaust gas purification performance of protective environmental micro-surfacing materials. The results showed that when the molecular weight ratio of Ti : N : Fe was 1 : 0.7 : 0.005, the photocatalytic performance of the prepared environmental functional material with tail gas purification effect was the best. The availability of photocatalytic materials is increased, and the photocatalytic performance is the optimum. When 3% functional materials and 5% SBR latex are used as composite modifier of emulsified asphalt, the consistency, cracking resistance and high temperature performance of composite modified emulsified asphalt are the optimum. The micro-surfacing mixture prepared with 7% oil-stone ratio and 6%–7% external water has good high temperature stability, water stability and skid resistance. When the molecular weight ratio of Ti : N : Fe is 1 : 0.7 : 0.005, the HC purification efficiency reaches 12%, the CO purification efficiency reaches 15%, and the NO\textsubscript{X} purification efficiency reaches 30%, which has good road performance and exhaust purification effect.

1. Introduction

With the rapid increase of car ownership in China, the problem of environmental pollution caused by exhaust emission is becoming more and more serious. It is of great significance to develop effective tail gas treatment methods to solve this practical problem, prevent air pollution and improve air quality \cite{1}. At present, the solution of exhaust pollution is mainly through the following aspects: clean energy is developed and used; engine performance and adoption of internal purification technology are improved; road materials with exhaust degradation properties are developed. The addition of nano-TiO\textsubscript{2} and other catalytic materials in the pavement material can give the pavement material the ability to directly purify the exhaust gas discharged by the vehicle, so as to realize the purpose of exhaust purification \cite{2}.
More and more researchers have explored the function of adding environmental protection materials to various surface materials in the field of roads to endow roads with air pollutants purification. As a mature preventive maintenance technology, micro-surfacings are widely used in the field of road maintenance due to its good anti-skid, wear resistance, waterproof and rutting repair. However, there are few reports on the research of tail gas purification by micro-surfacings. Therefore, the research and development of environmentally friendly micro-surfacings with the function of purifying exhaust gas is not only beneficial to improve the performance of asphalt pavement, but also has important significance for changing the road development mode, promoting road construction and maintaining green and healthy development. TiO$_2$ is a kind of commonly used protective environmental photocatalyst, which can improve the mechanical properties and durability of asphalt pavement to a certain extent [3]. However, a single TiO$_2$ is restricted by a wide band gap ($E_g \approx 3.2$ eV), which has low utilization efficiency of solar energy and limited catalytic performance [4]. Doping metal ions in TiO$_2$ can prolong the recombination time to improve its photocatalytic activity [5, 6]. The doping of non-metal in TiO$_2$ can generate photogenerated carriers to improve its photocatalytic activity and absorb visible light [7, 8]. N-doped TiO$_2$ plays a role in narrowing the band gap and photocatalytic activity [9]. Synergistic effect exists in co-doped and tri-doped catalysts, which helps to enhance photocatalytic activity [10]. At present, the common research on asphalt pavement materials for catalytic purification of automobile exhaust mainly includes coating pavement and incorporation pavement. Mitsubishi Corporation of Japan will prepare nano photocatalytic materials coated on the road surface, the nitrogen oxide has obvious adsorption effect [11], but the coating process is long, the degradation effect is limited by the wet environment. Nano TiO$_2$ coating on rubber powder provides a new way to improve the efficiency and durability of photocatalyst for automobile exhaust degradation [12]. The visible-light-responsive and durable photocatalytic degradation coating for automobile exhaust was successfully prepared, but the research on the basic performance of pavement was lacking [13]. The use of TiO$_2$ in slurry seal materials can achieve the purpose of persistent degradation of NO$_x$ [14]. When doped iron enters the TiO$_2$ lattice, impurity energy levels will be formed above the valence band, which improves the utilization of sunlight by reducing the apparent band gap [15]. The wide-area composite nano-TiO$_2$ material is applied to the ultra-thin overlay asphalt mixture, which can degrade more than 10% of the main pollutants in automobile exhaust [16]. The fog seal material containing nano-TiO$_2$ can not only maintain the pre-curing function of the general fog seal layer, but also achieve the photocatalytic effect [17], which has certain promotional significance.

In summary, N and Fe co-doped TiO$_2$ photocatalytic materials to improve the photocatalytic properties of TiO$_2$ are less studied, and it has wide application prospect in pavement sealing materials. At the same time, the difference in experimental conditions and material dosage will cause the difference in photocatalytic performance. Therefore, the preparation of N, Fe co-doped Anatase TiO$_2$ photocatalytic materials based on sol-gel method provides an effective way to study and solve the preparation of efficient photocatalytic materials for road. Therefore, in this paper, the preparation conditions were optimized, the dosage ratio of different raw materials was determined, and the micro-surfacings with protective environmental exhaust purification function were prepared. The photocatalytic and road performance were systematically analyzed to realize the efficient utilization of photocatalytic materials for sunlight and the exhaust purification function of protective environmental micro-surfacings materials.

2. Raw material

2.1. The raw material of modified emulsified asphalt

2.1.1. Functional materials

There are six kinds of raw materials used for functional materials, namely, butyl titanate, ethanol, ferric nitrate, ammonium nitrate, ice ethanol and acetylacetone, and the purity is analytically pure. The specific content is shown in table 1, and the water used is distilled water.

2.1.2. Emulsified asphalt

In this study, the slow-cracking and fast-setting cationic emulsified asphalt BC-1 is used, B is the mixing type, and C is the cationic emulsified asphalt. The penetration of emulsified asphalt is 70.7 mm (0.1 mm at 25 °C, 100g and 5s), and the ductility is 88.5cm (5cm min$^{-1}$ at 15 °C).

2.1.3. SBR latex modifier

In this study, SBR latex modifier produced by Xinxiang Boxu Highway Engineering Co., Ltd. was used, and the model was JY-SBR 65 cationic styrene-butadiene latex. The latex modifier is milky white liquid modifier with certain viscosity. SBR latex performance indicators are shown in table 2.
Table 1. The raw materials of functional materials.

| Order number | Name               | Purity            | Molecular formula   | Manufacturer                                      |
|--------------|--------------------|-------------------|---------------------|--------------------------------------------------|
| 1            | Butyl titanate     | Analytically pure | C₁₆H₃₆O₄Ti         | Tianjin Comiou Co., Ltd                          |
| 2            | Ethanol            | Analytically pure | C₂H₁₀O₂             | Yantai Shuanghuang Chemical Co., Ltd             |
| 3            | Ferric nitrate     | Analytically pure | Fe(NO₃)₃·9H₂O       | Jinan Xichuan Chemical Technology Co., Ltd       |
| 4            | Ammonium nitrate   | Analytically pure | NH₄NO₃             | Tianjin Damao Chemical Reagent Factory           |
| 5            | Ice ethanol        | Analytically pure | H₅C₆O₆             | Tianjin Damao Chemical Reagent Factory           |
| 6            | Acetylacetone      | Analytically pure | C₅H₈O₂              | Tianjin Damao Chemical Reagent Factory           |

Table 2. Performance indexes of SBR latex.

| Test project                        | Test requirements | Measurement results |
|--------------------------------------|-------------------|---------------------|
| Particle charge                      | Cationic (+)      | Cationic            |
| Solid content, no less than %        | 63                | 65                  |
| Ph value                             | 8–19              | 10                  |
| Appearance                           | Milky white liquid| Milky white liquid  |

Table 3. Main technical indexes of 4.75 mm ~9.5 mm crushed stone.

| Performance index                        | Unit     | Test result | Technical requirement | Test method |
|------------------------------------------|----------|-------------|-----------------------|-------------|
| Apparent specific gravity                | g/cm⁻³   | 2.710       | <2.60                 | T0304       |
| Water absorption                         | %        | 0.9         | <2.0                  | T0304       |
| Crushing value of stone                  | %        | 22.3        | <26                   | T0316       |
| Losangeles weared value                  | %        | 20.9        | <28                   | T0317       |
| Polished drum coating stone value        | BPN      | 51          | <42                   | T0321       |
| Ruggedness                              | %        | 9.7         | <12                   | T0314       |
| Needle-like content                     | %        | 11.6        | <15                   | T0312       |

Table 4. Main technical indexes of 2.36 mm ~4.75 mm gravel.

| Performance index                        | Unit     | Test result | Technical requirement | Test method |
|------------------------------------------|----------|-------------|-----------------------|-------------|
| Apparent specific gravity                | g/cm⁻³   | 2.775       | <2.60                 | T0304       |
| Water absorption                         | %        | 1.4         | <2.0                  | T0304       |
| Crushing value of stone                  | %        | 20.7        | <26                   | T0316       |
| Losangeles weared value                  | %        | 18.6        | <28                   | T0317       |
| Polished drum coating stone value        | BPN      | 56          | <42                   | T0321       |
| Ruggedness                              | %        | 9.2         | <12                   | T0314       |
| Needle-like content                     | %        | 10.8        | <15                   | T0312       |

Table 5. Main technical indicators of 1.18 mm ~2.36 mm aggregate.

| Item                             | Test result | Standard | Test method |
|----------------------------------|-------------|----------|-------------|
| Firmness is not greater than (%) | 8.8         | <12      | T0340       |
| Sand equivalent not less than (%)| 74          | <65      | T0334       |
| Apparent specific gravity        | 2.793       | <2.30    | T0328       |

Table 6. Main technical indicators of 0.6 mm ~1.18 mm aggregate.

| Item                             | Test result | Standard | Test method |
|----------------------------------|-------------|----------|-------------|
| Firmness is not greater than (%) | 8.6         | <12      | T0340       |
| Sand equivalent not less than (%)| 71          | <65      | T0334       |
| Apparent specific gravity        | 2.786       | <2.30    | T0328       |
Table 7. Main technical indicators of mineral powder.

| Fraction of partial size | Apparent density (t/m³) | Moisture content (%) | Appearance | Hydrophilic coefficient | Plasticity index (%) |
|--------------------------|-------------------------|----------------------|------------|-------------------------|---------------------|
| Test result              |                         |                      |            |                         |                     |
| 0.3 mm ~0.6 mm           | 2.742                   | 0.2                  | No         | 0.53                    | 2.4                 |
| 0.15 mm ~0.3 mm          | 2.765                   | 0.1                  | No         | 0.49                    | 1.8                 |
| 0.075 mm ~0.15 mm        | 2.793                   | 0.2                  | No         | 0.71                    | 2.6                 |
| <0.075 mm                | 2.722                   | 0.3                  | No         | 0.36                    | 2.2                 |
| Technology index         |                         |                      |            |                         |                     |
| 0.3 mm ~0.6 mm           | ≥2.50                   | ≤1                   | No agglomerates | <1                      | <4                  |
| 0.15 mm ~0.3 mm          |                         |                      |            |                         |                     |
| 0.075 mm ~0.15 mm        |                         |                      |            |                         |                     |
| <0.075 mm                |                         |                      |            |                         |                     |
2.2. Aggregate

The coarse aggregate is basalt rolled crushed stone, the fine aggregate is limestone rolled crushed stone, and the filler is limestone powder. After cleaning, drying and screening, they were divided into eight groups, which were \(< 0.075 \text{ mm}, 0.075 \text{ mm} \sim 0.15 \text{ mm}, 0.15 \text{ mm} \sim 0.30 \text{ mm}, 0.3 \text{ mm} \sim 0.6 \text{ mm}, 0.6 \text{ mm} \sim 1.18 \text{ mm}, 1.18 \text{ mm} \sim 2.36 \text{ mm}, 2.36 \text{ mm} \sim 4.75 \text{ mm} \text{ and } 4.75 \text{ mm} \sim 9.5 \text{ mm}, \text{ respectively. The performance of aggregates was tested according to the test method of specification. The main indexes of ore used were shown in table 3–7, which met the requirements of specification.}

2.3. Cement

In this study, composite silicate cement P. C32.5 is selected as demulsifier regulator, and cement accounting for 1%—3% of the mineral mass is added according to the actual ratio. The physical state of cement is gray, delicate, loose and without agglomeration. According to the ‘Highway engineering cement and cement concrete test procedures’ (JTG E30–2005)\(^{[18]}\) test, the indicators meet the requirements.

2.4. Aqueous

The water used for micro-surfacing mixing in this study is tap water from Zhengzhou City, which met the technical requirements of raw material properties for water.

3. Test method

3.1. Preparation of functional materials by sol-gel method

Among the three crystal types of anatase, slate and rutile of TiO\(_2\), anatase crystal lattice contains many defects and dislocations. Compared with other crystal types, it can generate more oxygen vacancies to capture photons, and achieve the best catalytic effect\(^{[19]}\). Titanium dioxide prepared by sol-gel method was calcined at 400 \(\sim\) 600 °C for 3 h\(^{[20]}\), and the crystal form was the best anatase type\(^{[21]}\). Therefore, the study of Fe-N-TiO\(_2\) composite functional materials is of great significance.

Referring to the research results of preparation conditions of nitrogen and iron co-doped titanium dioxide (Fe-N-TiO\(_2\)) by Wang Shanshan et al.\(^{[22]}\), combined with the influence of preparation conditions on the experimental results in the actual test process, Fe-N-TiO\(_2\) was designed and prepared. When Ti source is 10 ml butyl titanate, the dosage of main reagents used in group A1 \(\sim\) A9 is shown in table 8. Ti:N:Fe molecular weight was divided into A1 \(\sim\) A9 groups according to table 9.

The first step, set a constant temperature magnetic stirrer temperature of about 35 °C. 30 ml anhydrous ethanol was added to the beaker of constant temperature magnetic stirrer, and then 10 ml butyl titanate was slowly added to the dropper. After the butyl titanate and anhydrous ethanol were uniformly dissolved, a certain amount of iron nitrate (specific dosage reference table 3 related dosages) was added to continue stirring for about 30 min to ensure the full dissolution of iron nitrate powder. After the yellow transparent and homogeneous solution was presented in the beaker, it could be considered that the reaction between ferric nitrate and butyl

| Table 8. Dosage of main chemicals corresponding to 10 ml titanium tetrabutoxide. |
|---|---|---|---|---|---|---|---|---|
| Groups | A1 | A2 | A3 | A4 | A5 | A6 | A7 | A8 | A9 |
| Ferric nitrate (g) | 0.036 | 0.036 | 0.036 | 0.059 | 0.059 | 0.059 | 0.083 | 0.083 | 0.083 |
| Ammonium nitrate (g) | 0.353 | 0.588 | 0.824 | 0.353 | 0.588 | 0.824 | 0.353 | 0.588 | 0.824 |

| Table 9. Molecular weight ratio of Ti: N: Fe in each group. |
|---|---|---|---|---|---|---|---|
| Groups | A1 | A2 | A3 |
| Ti: N: Fe molecular weight ratio | 1:0.3:0.003 | 1:0.5:0.003 | 1:0.7:0.003 |
| Groups | A4 | A5 | A6 |
| Ti: N: Fe molecular weight ratio | 1:0.3:0.005 | 1:0.5:0.005 | 1:0.7:0.005 |
| Groups | A7 | A8 | A9 |
| Ti: N: Fe molecular weight ratio | 1:0.3:0.007 | 1:0.5:0.007 | 1:0.7:0.007 |

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titanate was sufficient. Using one-time drop tube slowly add about 0.5 ml acetyl acetone solution in the beaker, continue to stir about an hour can be fully reacted solution, denoted as a solution.

The second step, set the constant temperature magnetic stirrer temperature is about 35°C, first add 10 ml distilled water to the beaker on the constant temperature magnetic stirrer, and then add a certain amount of ammonium nitrate solid particles (specific dosage reference table 5 related dosage) diluted with 10 ml anhydrous ethanol and 2ml glacial acetic acid, stirring uniform to get ethyl solution.

The third step is to keep the temperature of the constant temperature magnetic stirrer about 35°C, and add the B solution to the A solution at the speed of 20s ml⁻¹. After adding ethyl solution, keep stirring until the formation of gel.

In the fourth step, the gel samples were aged for 12 h under the standard conditions of 25°C and 55% humidity, dried at 80°C in an electrothermal blast drying oven, and grinded into powder by mortar after 3 h. After calcination in a 500°C high-temperature box resistance furnace for 3 h, A_x powder was obtained, i.e., Fe-N-TiO_2 powder with different dosages. Finally, 9 groups of Fe-N-TiO_2 powder were loaded into the sealing bag, and the dosage label was affixed for standby.

### 3.2. Preparation of modified emulsified asphalt

Cationic styrene-butadiene rubber latex (SBR latex, hereinafter referred to as X, the same) and the mixture of environmentally friendly functional materials and SBR latex (hereinafter referred to as Y, the same) was used as modifiers to modify the slow-cracking and rapid-setting cationic emulsified asphalt. Through the comparative analysis of each performance index of the prepared modified emulsified asphalt, the modification process parameters and the optimal dosage of the modifier were determined, which provides a reference for the environmentally friendly composite modified emulsified asphalt functional materials.

#### 3.2.1. Preparation process optimization of X modified emulsified asphalt

X modified emulsified asphalt was prepared by high speed shearing machine. The preparation process of SBR latex content (percentage of emulsified asphalt content), mixing temperature, mixing time and mixing speed was optimized.

① SBR latex content

According to ‘Technical specification for highway asphalt pavement construction’ (JTG F40–2004) and related literature [23], SBR latex content was designed to be 0%, 1%, 3%, 5% and 7%, and its influence on the evaporation residue index and storage stability of modified emulsified asphalt was studied to determine the optimal value of SBR latex content.

② Mixing temperature

When cationic emulsified asphalt is modified by latex modifier, the shear modification process can be carried out without heating. The emulsion SBR modifier was used to modify the liquid emulsified asphalt. At present, the two can be melted at room temperature, so design mixing temperature was 25°C.

③ Mixing time

When emulsion SBR modifier was used to modify liquid emulsified asphalt, the mixing time was 1–5 min to achieve the purpose of full integration. Therefore, the mixing time was designed to be 5 min to ensure the full integration of emulsion and emulsified asphalt.

④ Mixing speed

Studies have shown that when the latex modifier modified asphalt, the shear rate increased slowly from 500r min⁻¹ to 5000r min⁻¹ [24]. When cationic emulsified asphalt is modified by latex modifier, it can be fully mixed at 500 r min⁻¹ shear rate [25]. Therefore, the mixing speed is 500r min⁻¹ to ensure the full fusion of latex and emulsified asphalt.

#### 3.2.2. Preparation of X modified emulsified asphalt

Firstly, the emulsified asphalt was poured into the iron container, and the temperature controller was not started to heat the asphalt. The shear speed of the high-speed shear was set to 500 r min⁻¹. Then, SBR latex modifiers (SBR latex content 0%, 1%, 3%, 5%, 7%) were added with droppers. Then set the shear time of high speed shear is 5 min. Finally, after the modification process is completed, the sheared modified emulsified asphalt is taken out and cooled to room temperature, namely, X modified emulsified asphalt.

#### 3.2.3. Preparation process optimization of Y modified emulsified asphalt

Based on the preparation of X modified emulsified asphalt, Y modified emulsified asphalt was prepared by high speed shearing machine. Considering Fe-N-TiO_2 as solid powder material, it cannot directly modify emulsified asphalt, so SBR latex and Fe-N-TiO_2 is used as composite modifiers for Y modified emulsified asphalt. Based on the above research, the mixing speed of the modification with high-speed shear was slowly increased from
500 r min$^{-1}$ to 5000 r min$^{-1}$, the mixing temperature was 25 °C, and the mixing time was 5 min. Considering the influence of preparation process on the modification effect in the test process, the particle size and dosage of Fe-N-TiO$_2$ powder material and main preparation process of mixing method were analyzed and optimized. The particle size of Fe-N-TiO$_2$ powder material was determined to be 100 meshes, and the dosage range was 1%—5%. SBR latex and Fe-N-TiO$_2$ were slowly added in the low-speed stirring process at 500 r min$^{-1}$. After complete addition, the shear speed of the high-speed shear was slowly increased to 5000 r min$^{-1}$ to improve the modification effect.

3.2.4. Preparation of Y modified emulsified asphalt

The modified emulsified asphalt was prepared by high-speed shearing method. The emulsified asphalt was poured into the iron container, and the temperature controller was not started to heat the asphalt. The shear rate of the high-speed shearing machine was set to 500 r min$^{-1}$, and then the SBR latex modifier with 5% dosage was added by the dropper. The Fe-N-TiO$_2$ powder material with set dosage and particle size was slowly added by the balance scale. After the SBR latex and Fe-N-TiO$_2$ powder material was all added, the shear rate of the high-speed shearing machine was slowly increased to 5000 r min$^{-1}$, and the shear time of the high-speed shearing machine was set to 5 min. After the modification process is completed, the shear modified emulsified asphalt is removed and cooled to room temperature, which is Y modified emulsified asphalt.

3.3. Preparation and mix proportion design of Protective environmental

The modified emulsified asphalt used to prepare protective environmental micro-surfacing is Y-modified emulsified asphalt prepared previously. Through comparison and analysis with the X-modified emulsified asphalt prepared previously, the influence of environmentally friendly functional material Fe-N-TiO$_2$ powder as a modifier on the pavement performance of micro-surfacing is analyzed.

3.3.1. Determination mineral aggregate gradation

After the micro-surfacing is paved to the road surface, the road surface is very rough in the initial use, but the apparent effect and anti-skid performance are better after a period of traffic. Therefore, MS-3 micro-surfacing mineral aggregate gradation is selected. According to the median of gradation range, the specific mineral aggregate gradation and the upper and lower limits are synthesized, as shown in table 10. The synthetic gradation curve is shown in figure 1, and the synthetic gradation curve conforms to the trend of S-shaped curve.
3.3.2. Determination method of optimal asphalt content
After determining the continuous gradation of aggregate, cement and water are added, and different ratios of oil to stone are changed. The cohesion test, wet wheel wear test after soaking for 1 h and sand adhesion test of load wheel are carried out respectively. The design technical indexes of the mixture meet the requirements of 'Technical specification for highway asphalt pavement construction' (JTG F40–2004) [26].

3.4. Road performance test method
In order to test the road performance of micro-surfacing with different contents of protective environmental functional materials, the high temperature stability of X and Y micro-surfacing mixture was studied by rutting test. According to the wet wheel wear test of 'Technical guideline for micro-surfacing and slurry seal', the water stability of X and Y micro-surfacing was evaluated. The pendulum tester test was used to evaluate the skid resistance of X and Y micro-surfacing mixtures.

3.5. Exhaust purification test method
Combined with the relevant research conclusions of the research group exhaust purification and PM2.5 adsorption effect detection device, and after adjustment according to the needs of experimental study, a new detection test method is determined, and the effect of the prepared functional materials on the purification of exhaust gas is evaluated.

Figure 2 shows the detection equipment. Figure 2(a) is the exhaust purification and PM2.5 adsorption effect detection device designed by our team. The facility from left to right includes: 1- delivery tube, 2- radiator, 3- delivery tube, 4- reaction chamber, 5- adsorption degradation material, 6- inlet port, 7- control valve, 8- PM2.5 detector, 9- automobile exhaust detector, 10- air outlet. Figure 2(b) shows the adjusted tail gas purification effect detection device. The device from left to right includes: delivery tube, radiator, delivery tube, control valve, inlet port, reaction chamber, adsorption degradation material, air outlet, 11- gas sampling pump, and 12 - automobile exhaust detector.

The specific explanations and relevant adjustments of each component are as follows:

(1) The guide pipe is connected with the exhaust port of the vehicle by connecting the variable diameter pipe. The two ends of the guide pipe are fixed and sealed by the American plastic handle throat hoop.

(2) Inlet control valve is installed on the lower side. The outlet is on the upper side and the outlet control valve is installed. When entering the air, the inlet and outlet are opened, and the motor vehicle is started to make the exhaust gas fill the reaction chamber and close the outlet and inlet.

(3) The material of the reaction chamber is organic glass, which can pass through more than 92% of the Sunlight and 73.5% of the ultraviolet light. It can minimize the loss of ultraviolet light and simulate the natural light environment more realistically.

(4) In figure (a), PM2.5 detector and automobile exhaust detector are placed in the reaction chamber, and the change of gas concentration in the reaction chamber is detected by regular observation. This method will
cause continuous alarm of detection equipment and affect the service life of the instrument. Exhaust gas is full of detection equipment, which may cause errors between the test results and the actual situation. In the actual detection process, when the light intensity of the Sun is high, it is easy to cause difficulty in reading the test results.

(5) (b) A small hole is opened on the side of the reaction chamber, and the gas is pumped out to the inlet hole of the automobile exhaust detector at regular intervals through the gas sampling pump, and the gas is detected by the way of detecting the gas composition in the reaction outdoor. The amount of gas extracted by gas sampling pump is lower than that in the reaction chamber, which will not affect the result detection. Outdoor detection helps to control the detection time, solve the detector power outage and other emergencies. The gas entering the detector can be controlled to determine the concentration of the adsorbed degradation material after degradation.

4. Experiment results and analysis

4.1. Performance analysis of functional materials

The functional materials prepared in this study mainly had four changes in properties during the preparation process, including sol stage, gel stage, drying stage and calcination stage. Figure 3 shows the four changes.

Figure 3 (a) take group A5 as an example, the change process of functional materials in the sol phase was completed. After the addition of B solution to A solution, the sol initially appeared in the transparent state in figure 1, and then the texture became dense. Subsequently, it can be observed that the bubbles in the beaker gradually disappeared, the sol structure was dense, and the gel began to form.

Figure 3 (b) A3, A6, A9 gel phase functional materials. When functional materials form gel state, the color becomes deeper than sol state. Since the ammonium nitrate solution was colorless and the iron nitrate solution was orange-yellow, it can be determined that the color change of functional materials was mainly related to the amount of iron element content in the raw material. Therefore, the color changes of A3, A6 and A9 groups can be compared and observed. Compared with the three groups of gel materials, it can be seen that with the increase of iron nitrate content, the color of the gel becomes deeper, which is consistent with the results predicted earlier.
Figure 3(c) is the drying period of A1—A9 groups. After aging and drying of the functional materials in the sol state, and grinding to powder, the properties of the nine groups of functional materials are compared. Intuitively, the higher the iron content, the deeper the color of the powder material after drying, the easier it is to form larger particles. It shows that high iron content may cause low utilization of raw materials.

In order to compare and study the performance changes of different iron content after calcination, this study took A3, A6 and A9 groups of functional materials as the representative groups for trait analysis, as shown in figure 3(d). The functional materials of group A3 showed light yellow and no metal luster. The functional materials of group A6 showed light yellow and no metal luster. The functional materials of group A3 showed black and metal luster. Three groups of functional materials were added to dilute hydrochloric acid solution, and it was observed that there was no significant change in group A3 and group A6, while group A9 functional materials reacted to produce bubbles, which proved that group A9 functional materials had iron precipitation after calcination.

It can be analyzed that when the Fe : Ti content ratio exceeds 0.005, excessive iron ions may not be able to effectively enter the TiO$_2$ lattice, and precipitate in the form of iron elemental at 500 °C. Due to the metal ions doped with titanium dioxide, it will be limited by the maximum doping amount. When the Fe : Ti content ratio is 0.007, it may exceed its maximum content, so it accumulates on the surface of titanium dioxide. The accumulation of iron will weaken the photocatalytic performance of the composite materials. By forming electron-hole composite centers on the band gap of the composite materials, the light quantum yield is reduced and the photocatalytic activity of the functional materials is reduced. Therefore, the Fe : Ti content ratio should be controlled below 0.005.

4.2. Technical index analysis of modified emulsified asphalt

4.2.1. Technical index analysis of X modified emulsified asphalt

The performance of X modified emulsified asphalt was analyzed to determine the optimum dosage of SBR latex modifier. Figure 4 is the broken line diagram of the residue on the screen (1.18 mm screen) with the change of SBR latex content.

Figure 4 shows that the residual amount on the sieve surface of emulsified asphalt decreases significantly after shearing. With the increase in the amount of modifier, the residual on the screen of X-modified emulsified asphalt showed a gradually accelerated upward trend. The reason is that the shear process promotes emulsified asphalt to reduce flocculation and agglomeration, and reduce the content of asphalt particles. At the same time, the increase of latex modifier can cause the occurrence of asphalt flocculation and coalescence to a certain extent, increase the size of asphalt particles, and cause the increase of residue on the screen. The residual amount on the screen of X-modified emulsified asphalt was 0.07%, which was still far less than the labeling requirement of 0.1%. Under the condition of test dosage, the residual amount on the screen met the technical index requirements.

Figure 5 shows the broken-line diagram of the evaporation residue content and three indicators of X-modified emulsified asphalt changing with SBR latex content.
It can be seen from figure 5 that the solid content of latex is slightly higher than the evaporation residue content of emulsified asphalt. Therefore, with the increase of SBR latex content, the evaporation residue content of X-modified emulsified asphalt will increase slightly. The penetration degree of evaporation residue of X modified emulsified asphalt (100g, 25 °C, 5s) decreased first and then increased slightly with SBR latex content. The penetration degree change of evaporation residue of SBR latex at 5% and 7% content was only 1.1, and the change was low. With the increase of 1% latex content, the penetration degree increases about 1.7, so the increase of SBR latex content can rapidly increase the high temperature performance of X modified emulsified asphalt. SBR latex modifier with 5% or less is recommended. The evaporation residue ductility (5 °C) of X modified emulsified asphalt increased significantly with the increase of SBR latex content. Without modifier, the ductility is only 10cm. When the content is 1%, the ductility meets the requirement of more than 20 cm. When the content reaches 5% and above, the ductility is greater than 150cm. This shows that the addition of SBR latex can significantly improve the low-temperature performance of X-modified emulsified asphalt. A certain amount of SBR latex is recommended to improve the low-temperature performance of emulsified asphalt. The softening point of the evaporation residue of X modified emulsified asphalt increases obviously with the increase of SBR latex content. When the content reaches 5% or more, the softening point reaches the requirement that the softening point of the evaporation residue of X modified emulsified asphalt is not less than 53 °C. This is because SBR latex can hinder the flow of asphalt in the form of filament winding structure in emulsified asphalt, thereby improving the high temperature performance of asphalt.

Figure 6 is the plot of storage stability (1d, 5d) of X modified emulsified asphalt versus SBR latex content. When the SBR latex content is 1%, the 5d storage stability of emulsified asphalt decreases, but the storage stability increases significantly with the increase of SBR latex content. This is because the modified emulsified asphalt and SBR latex form a double force unstable system, and the instability increases gradually. Because SBR latex has good storage stability, the instability of X modified emulsified asphalt has little improvement. The 5d storage stability of X modified emulsified asphalt with 7% SBR latex content exceeds 5% performance requirements, and its 1d storage stability is close to the limit of 1%.
In conclusion, with the increase of SBR latex content, the residue on the screen and evaporation residue content of emulsified asphalt are increased, and the storage stability of the emulsion is reduced. The three indexes are obviously improved and improved. Based on the analysis results, 5% SBR latex was used to modify emulsified asphalt.

4.2.2. Technical index analysis of Y modified emulsified asphalt

Taking the content of Fe-N-TiO2 powder material as the main technical index of modified emulsified asphalt as the research object, the influence of Y modified emulsified asphalt performance was tested, and the influence of different functional materials on the performance of modified emulsified asphalt was analyzed. The relevant test results are shown in figures 7–8. Figure 7 shows the broken line diagram of the residual amount on the screen (1.18 mm screen) with the change of Fe-N-TiO2 powder content.

With the increase of modifier dosage, the residual amount on the screen of Y modified emulsified asphalt showed a slow upward trend. The reason is that the increase of latex modifier can increase the residue on the screen to a certain extent, and the Nano-powder inorganic modifier can reduce the flocculation reaction of latex modifier.

Figure 8 shows the broken-line diagram of the evaporation residue content and the three major indexes of Y-modified emulsified asphalt changing with the content of Fe-N-TiO2 powder material.

It can be seen from figure 8 that the evaporation residue content of Y modified emulsified asphalt changes more with the increase of Fe-N-TiO2 powder material content, because Fe-N-TiO2 powder material does not evaporate due to the evaporation of water in modified emulsified asphalt, and the quality of evaporation residue...
will increase. The penetration degree of evaporation residue (100g, 25°C, 5s) of Y modified emulsified asphalt showed irregular changes with the increase of Fe-N-TiO₂ powder content, but the addition of Fe-N-TiO₂ powder material cannot greatly change the penetration degree value of Y modified emulsified asphalt. When the content of Fe-N-TiO₂ powder material is 3% and below, the ductility is greater than 150 cm, the low temperature performance of Y modified emulsified asphalt maintains the optimum, that is, 1% ~ 3% content of Fe-N-TiO₂ powder material does not affect the low temperature performance of Y modified emulsified asphalt. When the content of Fe-N-TiO₂ powder material is 3% ~ 5%, the ductility is less than 150 cm, the low temperature performance of Y modified emulsified asphalt is limited, but it still meets the performance requirements of more than 20 cm. The softening point of evaporation residue of Y modified emulsified asphalt increases first and then decreases with the increase of Fe-N-TiO₂ powder content, and the softening point reaches the highest when the content of Fe-N-TiO₂ powder material is 3%. This is because a certain content of Fe-N-TiO₂ powder materials may increase the toughness of modified emulsified asphalt, thereby improving the high temperature performance of asphalt.

Figure 9 shows that the storage stability (1d, 5d) of Y modified emulsified asphalt are affected by the content of Fe-N-TiO₂ powder.

It can be seen from figure 9 that with the increase of the content of Fe-N-TiO₂ powder material, the 1d and 5d storage stability values of Y modified emulsified asphalt are increasing. When the content of Fe-N-TiO₂ powder material is 4% and above, the 1d and 5d storage stability cannot meet the performance requirements of 1% and 5%.

It can be concluded that 1 ~ 3% content of Fe-N-TiO₂ powder materials can maintain the performance index of emulsified asphalt. At the same time, in order to provide sufficient amount of Fe-N-TiO₂ powder materials to ensure that micro-surface materials can have good photocatalytic effect, 3% content of Fe-N-TiO₂ powder materials and 5% content of SBR latex are used as composite modifiers of Y modified emulsified asphalt.
4.3. Micro-surfacing mixture proportion

4.3.1. Cohesion performance

The change of cohesive force of X and Y micro-surfacing mixture under different oil-stone ratios was studied by adjusting the oil-stone ratio with 6% external water consumption. The specific test results are shown in figure 10.

It can be seen from figure 10 that with the increase of the oil-stone ratio, the cohesion of the X and Y micro-surfacing materials at 30 min and 60 min increases gradually on the whole. When the oil-stone ratio is 7.5%, the cohesion of X, Y micro-surface materials reaches the maximum in 60 min. At the same oil-stone ratio, the cohesive force of X micro-surfacing material is greater than that of Y micro-surfacing material. It can be seen that high oil-stone ratio has high content of effective asphalt, which can improve the cohesive force of the mixture in a short time, but high oil-stone ratio will prolong the emulsification time, so the cohesive force reaches the maximum when the oil-stone ratio is 7.5%. The effective asphalt content of X micro-surfacing material is slightly higher than that of Y micro-surfacing material, so its cohesion is also slightly higher.

4.3.2. Wet wheel wear performance

X, Y modified emulsified asphalt 1d, 6d wet wheel wear curve as shown in figure 11.

It can be seen from figure 11 that with the increase of oil-stone ratio, the wet wheel wear value of X and Y micro-surface materials decreases rapidly, and the wear resistance is improved. X, Y micro-surface material in
the same immersion conditions wet wheel wear value with the change trend of oil-stone ratio is generally consistent, oil-stone ratio in 6.5%–8.5%, meet the requirements.

The analysis shows that for X and Y micro-surfacing materials, the content of evaporation residue is similar, and the effect on mineral aggregate gradation is not much different. The influence of solid modifier on the compatibility and water resistance of micro-surfacing mixture can be ignored. Therefore, under the same oil-stone ratio, the wet wheel wear values of the two are close. However, it can be seen that the oil-stone ratio has a significant impact on the compatibility and water resistance of micro-surfacing mixtures.

4.3.3. Adhesive sand amount
X, Y modified emulsified asphalt mortar volume change curve as shown in figure 12.

It can be seen from figure 12 that with the increase of oil-stone ratio, the amount of sand adhered to the materials at X and Y micro-surfacing increases in a positive proportion. Under the same conditions, the variation trend of the amount of sand adhered to the X and Y micro-surfacing materials with the oil-stone ratio were generally consistent. When the oil-stone ratio was 6.5%—8.0%, it met the specification requirements. When the oil-stone ratio was 8.5%, it was close to the limit value of the amount of sand adhered to the surface.

The analysis shows that for the X and Y micro-surfacing materials, the content of evaporation residue is similar, and the effect on mineral aggregate gradation is not much different. The influence of solid modifier on the hazards such as oil flooding of micro-surfacing mixture can be ignored. Therefore, under the same oil-stone ratio, the amount of sand adhesion between the two is close. The asphalt content should be less than 8.5% in order to effectively avoid the hazards such as oil flooding on the pavement at the micro-surfacing.
4.3.4. Optimal asphalt content
The wear value of wet wheel and the amount of adhesive sand of different X and Y micro-surface materials immersed in water for 1 h under different oil-stone ratios are plotted as the relationship curve of figure 13. It can be seen from figure 13 that the X and Y micro-surfacing materials are close to the optimal oil-stone ratio when the oil-stone ratio is about 7%, and the indicators such as cohesion are qualified when the oil-stone ratio is 7%. Therefore, the optimum oil-stone ratio is determined to be 7%.

4.4. Performance analysis of protective environmental micro-surfacing
4.4.1. Analysis of mixable time of protective environmental micro-surfacing
According to the mixing time test of "Technical guide for micro-surfacing and slurry seal", the 1 d and 6 d mixing time curves of X and Y modified emulsified asphalt are shown in figure 14.
It can be seen from figure 14 that with the increase of water addition, the mixing time of X and Y micro-surfacing mixture is prolonged. Under the same water consumption, the mixing time of X micro-surfacing mixture is longer than that of Y micro-surfacing mixture. When the external water consumption of the mixture at the X micro-surfacing is more than 7%, the mixture is too thin. When the external water consumption is less than 4%, the mixture is too dry and the mixing time does not meet the requirements. Under this oil-stone ratio, the external water consumption is appropriate at 5%-6%. When the external water consumption of the mixture...
at Y micro-surfacing is more than 8%, the mixture is too thin. When the external water consumption is less than 5%, the mixture is too dry and the mixing time does not meet the requirements. Under this oil-stone ratio, the external water consumption is 6%—7%.

The analysis shows that with the increase of applied water, emulsified asphalt is gradually diluted, and the probability of mutual aggregation between asphalt particles is reduced. At the same time, the diffusion layer expanded, the ζ potential increased the emulsification rate of emulsified asphalt decreased, and the mixing time prolonged. Because the evaporation residue content of X micro-surfacing mixture is lower than that of Y micro-surfacing mixture, the contact surface of asphalt particles is wider during mixing.

4.4.2. Road performance analysis of protective environmental micro-surfacing

The high temperature stability, water stability and skid resistance of X and Y micro-surfacing mixture were tested and analyzed to evaluate the road performance of the mixture.

(1) High temperature stability performance

Rutting test results are calculated as shown in table 11:

![Table 11: Rutting resistance deformation test results of different modified emulsified asphalt micro-surfacing mixture.](image)

| Targets of test | X modified emulsified asphalt | Y modified emulsified asphalt | Specification requirement |
|----------------|-------------------------------|-------------------------------|--------------------------|
| PLD (%)        | 4.5                           | 4.2                           | ≤5                       |

(2) Moisture stability

The results of 6 d wet wheel wear test of X and Y micro-surfacing mixtures are shown in table 12.

![Table 12: Experimental results on water stability of different modified emulsified asphalt micro-surfacing mixtures.](image)

| Targets of test | X modified emulsified asphalt | Y modified emulsified asphalt | Specification requirement |
|----------------|-------------------------------|-------------------------------|--------------------------|
| Wet wheel wear value 6d (g m⁻²) | 552.5 | 562.1 | ≤800 |

(3) Skid resistance

It can be seen from table 13 that the pendulum values of the two kinds of micro-surfacing mixtures are similar, and their pendulum values are much larger than the specification requirements, which can basically
meet the requirements of highway traffic safety for pavement skid resistance. It can be concluded that 7% powder modifier has little effect on skid resistance. This may be because the fineness of powder modifier is sufficient, and the formed asphalt mortar has relatively weak influence on pavement structure.

4.4.3. Performance study of protective environmental micro-surface exhaust purification

With Ti : N : Fe = 1 : 0.7 : 0.005, 1 : 0.5 : 0.005, 1 : 0.7 : 0.003, 1 : 0.3 : 0.005 as the representative dosage of powder modifier and Y micro-surfacing mixture as the representative ratio, the effect of protective environmental functional materials on micro-surfacing exhaust purification was analyzed. The prepared four groups of micro-surfacing mixtures were spread over the wheel track specimen of asphalt surface layer, denoted as Y-1, Y-2, Y-3 and Y-4, and the test was carried out after cooling for 24 h. Purification efficiency index as the evaluation standard, the formula (1):

$$\eta = \frac{C_0 - C_t}{C_0} \times 100\%$$  \hspace{1cm} (1)

Formula: $\eta$-purification efficiency, $C_0$ - Initial exhaust concentration, $C_t$-t time exhaust concentration.

The experiment was carried out under four outdoor temperature conditions of $15 \pm 2^\circ\text{C}$ and bright sunshine. The initial detection concentration of exhaust gas was about 10 min and HC concentration was 80 ppm. The starting time was unified by 10:00, and the actual starting time was about 5 min. In the icon, 0 ~10 min represented 10:00 ~15:00. The detection results of exhaust gas concentration were marked, as shown in figure 15–17:

(a) Process diagram of HC concentration change efficiency at t time
(b) Trend map of HC purification

Figure 15. HC detection results of tail gas purification.

(a) Process diagram of CO concentration change efficiency at t time
(b) Trend map of CO purification

Figure 16. CO detection results of tail gas purification.
Figure 15 is the HC test results of tail gas purification. With the increase of illumination time, the concentration of hydrocarbons decreases gradually. Within 4 h, the concentration of hydrocarbons decreased by about 10 ppm after the action of four micro-surfacing materials, indicating that the prepared micro-surfacing mixture has excellent catalytic purification function of hydrocarbons. After 5h catalytic purification reaction, Y-1 catalyzes 12% hydrocarbons and Y-4 catalyzes 10.5% hydrocarbons. Comparing the catalytic effect of four groups of mixture, it can be seen that the degradation effect of Y-1 is the best, followed by Y-2 and Y-3, and Y-4 is the worst. It shows that in the process of catalytic purification, the catalytic reactions in the visible and ultraviolet regions are carried out, and the catalytic purification function in the visible region plays a leading role. At the time of 9(14:30), the concentration change of hydrocarbons in the four groups of experiments was close to 0, and the catalytic effect could last about 4 h or more, with excellent purification function.

Figure 16 shows the CO detection results of tail gas purification. With the increase of illumination time, the concentration change of carbon and oxygen compounds showed a trend of acceleration first and then decrease. At 5 time (12:30), the photocatalytic effect of the four groups of micro-surface materials reached the optimal. At 10:00 (15:00), the photocatalytic activity of Y-1 was 4% higher than that of Y-4, and the degradation effect was the best among the four groups of photocatalytic micro-surfacing materials. The catalyst proportion of Ti : N : Fe = 1 : 0.7 : 0.005 could achieve relatively excellent catalytic effect, and its catalytic efficiency was relatively constant, which was a relatively good proportion. The catalytic effect of Y-4 is obviously lower than that of Y-1 ~ Y-3, which is not an excellent photocatalytic purification of carbon oxides. In summary, when purifying CO pollutants, the Y-1 micro-surfacing mixture was the best.

Figure 17 is the NOX detection results of tail gas purification. With the change of time, the nitrogen oxides at the four groups of micro-surfaces show a catalytic trend of first acceleration and then deceleration. At 7 (13:30), the concentration of NOX in the Y-2 ~ Y-4 group began to change close to 0, but the concentration of NOX in the Y-1 group was still decreasing at 10 (15:00), and the micro-surface materials showed high photocatalytic performance. Four groups of micro-surface materials photocatalytic purification of NOX reached 30% purification effect. This indicates that in the process of catalytic purification of tail gas, the environmentally friendly micro-surfacing pavement has a good effect on the catalytic purification of low-concentration nitrogen oxides, and the catalytic aging of Y-1 group may be more durable.

To sum up, a relatively warm environment with relatively weak UV light, the experimental study on tail gas purification shows that the visible light has a strong effect and the UV light intensity is insufficient. Therefore, the catalysis in the visible light section plays a leading role, which is in line with the purpose of improving the degradation effect of environmental protection pavement materials on the visible light condition, and is a good attempt.

5. Conclusion

(1) Fe-N-TiO2 materials with different molecular weight ratio of Ti : N : Fe was prepared. A preparation method of Fe-N-TiO2 materials suitable for simple test conditions was determined. It needs 12 h aging. The dosage of Ti : N : Fe is 1 : 0.3 : 0.003 ~1 : 0.7 : 0.007. Fe : Ti content ratio should be controlled below 0.005, and the optimum amount of functional materials is Ti : N : Fe = 1 : 0.7 : 0.005.
(2) The optimum preparation process of styrene butadiene latex modified emulsified asphalt (X modified emulsified asphalt) and styrene butadiene latex and functional material composite modified emulsified asphalt (Y modified emulsified asphalt) was determined. Under the optimum preparation conditions, the residue on the modified emulsified asphalt sieve was 0.06%, the evaporation residue ductility was $\geq 150$ cm, and the softening point was 58.1 °C, and the performance index was the best.

(3) 7% oil-stone ratio of $X$, $Y$ micro-surfacing mixture, adding water in 6%–7% can get better performance indicators, cohesion close to the optimal value, 1d and 6d wet wheel wear value was 225g m$^{-2}$ and 507g m$^{-2}$, load wheel sand value was 288g m$^{-2}$. The 3% content of protective environmental functional materials can be used as modifier of emulsified asphalt for micro-surfacing.

(4) When the powder modifier content was 3%, the rutting deformation performance index of $Y$ micro-surfacing mixture was 0.3% lower than that of $X$ micro-surfacing mixture, and the anti-rutting performance was reduced; 6 d wet wheel wear value was 2% higher than $X$ micro surface mixture, water stability had a certain reduction; the pendulum value was close to the mixture at $X$ micro-surfacing.

(5) The HC purification rate of Y-1 micro-surfacing material was 12%, the CO purification rate was 15%, and the NOx purification rate was 30%. The Y-1 micro-surfacing material had the best purification effect, which could be used as an environmental micro-surfacing material for road vehicle exhaust purification.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

CRediT authorship contribution statement

Z. Li: Conceptualization, Investigation, Project administration, Supervision, Writing-Review & Editing. T. Guo: Conceptualization, Formal analysis, Methodology, Visualization. Y. Chen: Supervision, Project administration, Data curation, Formal analysis. M. Yang: Conceptualization, Writing-Original Draft, Supervision, Investigation. N. Wang: Conceptualization, Supervision, Writing-Original Draft. J. Wang: Conceptualization, Project administration, Supervision, Investigation. L. Jin: Funding acquisition, Investigation.

Declaration of competing interest

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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