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Microscopic investigation on blending of montmorillonite modified bitumen and reclaimed asphalt binder

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Abstract

The performance of asphalt mixtures containing reclaimed asphalt pavement (RAP) are strongly influenced by the degree of blending between RAP and virgin bitumen. In the aim of revealing the blending interface, the present work reports an experimental investigation on the blending degree between modified bitumen and RAP (or aged bitumen). A montmorillonite-rhodamine composite modifier was prepared as a tracer, which has exclusive element and fluorescence. Using microscopic test technique, the blending process of and the crack process of RAP bitumen was observed, and the blending image of new reclaimed asphalt mixture was obtained. The interface was investigated through image processing and machine learning. Based on the results, the montmorillonite modifier slightly affected on the softening point and penetration of virgin bitumen after addition. The blending interface was engagement like gears. The optimal heating temperature range is 110 °C to 115 °C for forming a better adhesive interface between RAP and modified bitumen. Moreover, the interface transition zone thickness between RAP and modified bitumen was measured to be 4 to 8 μm.

1. Introduction

Using high amounts of reclaimed asphalt pavement (RAP) has become inevitable and important for the road industry globally. From an environmental and economic perspective, the use of RAP in new asphalt mixture is a beneficial and thus attractive development. For example, in the Netherlands, the dosage of RAP has been at least 90% in producing new asphalt mixture for base layers [1]. Further, in order to soften the RAP bitumen, new soft bitumen and/or rejuvenator are usually added during the mixing process to produce new asphalt mixture, because RAP bitumen is already aged and hardened during the pavement service life. The blending of new and aged bitumen lead to cohesion and internal friction of new asphalt mixture. The interface between new and aged bitumen is closely related to the interface strength and performance of recycled asphalt mixture. However, the interface formation between new and aged is a complex blending process, and the interface shows a multifarious characteristic, including three blending interfaces like new-ageing new-new and ageing-ageing [2–4]. Hence, the characteristics of blending interface fusion are very important and need to be revealed urgently.

Related topics have attracted the attention of researchers all over the world, especially, the blending of RAP bitumen and new bitumen. The initial studies focused on the relationship between macroscopic performance and microstructure of new asphalt mixture. The results illustrated that the blending degree of RAP bitumen and new bitumen depends on the mixing temperature, mechanical and chemical properties of two bitumen [5–8]. In addition, an assumption of ‘black aggregate’ was proposed, which supposes that the RAP bitumen does not blend with new bitumen but the RAP material just acts as aggregates [9].

Afterwards, more and more microscopic results on blending degree of two bitumen have been obtained through different methods, such as Fourier Transform Infrared Spectroscopy (FTIR) [10, 11], Scanning Electron Microscope (SEM) coupled with an Energy Dispersion Spectrum (EDS) [12], Fluorescent Microscope (FM) [13],...
2. Materials and methods

2.1. Materials

2.1.1. Bitumen

PJ-90 bitumen was obtained from Liaohai Oilfield, Panjin, China. The conventional properties of bitumen are as follows: softening point was 46.7 °C, penetration was 86.9 dmm (25 °C) and ductility was 121.7 cm (15 °C). Aiming to simulate the RAP bitumen, an aged PJ-90 bitumen was prepared. The thermal oxidation ageing of bitumen was simulated in laboratory by thin film oven test (TFOT). The conditioning temperature was 163 °C and ageing time was 5 h. All methods were according to the Chinese standard (JTG E20-2011) [25]. After ageing, the conventional properties of aged bitumen are as follows: softening point was 52.9 °C, penetration was 45.6 dmm (25 °C) and ductility was 47.6 cm (15 °C).

2.1.2. Aggregates

RAP was obtained from Ning-Hu Highway of China. The size range was 2.36–26.5 mm. RAP aggregates were prepared as two types samples. One was cut into slices that are smooth on both sides for measuring the thickness of bituminous films on RAP. The film thickness of RAP is 9.1 μm to 11.6 μm when the slices were observed under microscope. The other is that one side of the RAP aggregate was cut smoothly and the other side was kept as it is, which was used to observe the crack behavior of the surface bitumen. Furthermore, limestone with particle size smaller than 2.36 mm was supplied from a local source in Shenyang, China and its powder was used as the mineral filler.

2.1.3. Montmorillonite

Montmorillonite (Mt) was obtained from the Clay Mineral Repositories of Purdue University (West Lafayette, IN) and was used without further purification. The basic information of the Mt was described with more details in our previous work [26]. For Mt tracer preparation, rhodamine 6 G was purchased from Shanghai Aladdin as fluorescent material and it was reagent grade with a purity of 95%.

2.2. Preparation methods

2.2.1. Preparation of montmorillonite tracer (MtT) and MtT modified bitumen

50 mg rhodamine and 200 ml distilled water were weighed and mixed in a beaker. The suspension was transferred to a conical flask, and then the conical flask was placed on a magnetic stirrer for 10 min at the maximum speed (1500 r min⁻¹). Afterwards, 2 g montmorillonite was added into the solution after the
Rhodamine had completely dissolved in distilled water and the solution had become scarlet. This new solution with montmorillonite was again stirred for another 2 h. Further, the prepared solution was transferred into a centrifuge of speed $5000 \text{ r min}^{-1}$. The supernatant of the centrifuge tube was poured off, and the sediment at the bottom was transferred into a glass dish and placed in a vacuum drying oven for drying. The drying temperature was $60 \degree C$. The dried solid was milled until the size less than $75 \mu m$. The prepared powder was the MtT, which can be used as a modifier or a tracer.

To prepare the MtT modified bitumen, the MtT powder and PJ-90 bitumen were mixed. The dosage of MtT was 5% by weight of bitumen. The PJ-90 bitumen was heated to $110 \degree C$, and then the MtT was added into bitumen for stirring 1 h with a speed of $3000 \text{ r min}^{-1}$.

### 2.2.2. Preparation of RAP asphalt mixture and slice samples
Gradation of AC-20 was selected for asphalt mixture in this paper, and 40 samples were prepared for optimal binder percentage determination according to Marshall’s design method. RAP was selected as the coarse aggregate and limestone was selected as the fine aggregate and filler. The passing rate of AC-20 gradation for each sieve is given in Table 1. Depending on the results optimal virgin binder percentage was 4.0%. It should be noted that the RAP was heated to $110 \degree C$ based on the results of this study and the limestone was heated to $170 \degree C$. In addition, the prepared Marshall specimens were sequentially placed in a high-speed cutting machine and cut into slices with a size of $10 \times 10 \times 1.2 \text{ mm}$.

### 2.3. Experimental methods

#### 2.3.1. Scanning electron microscopy measurement (SEM)
Microstructural images of the RAP slice samples were obtained using a Hitachi S-4800 Scanning Electron Microscope coupled with an energy dispersion spectrum (EDS) at an accelerating voltage of 5 kV.

#### 2.3.2. Fluorescent microscope (FM)
Fluorescent images of the RAP slice samples were obtained using an Olympus BX53 Fluorescence Microscope. A blue light was employed for exciting the fluorescence of samples due to that the excitation wavelength of Rhodamine 6 G is 520 nm.

#### 2.3.3. Ultrafine field microscope (UFM)
Detailed images of the RAP slice samples were obtained using a Keyence VH-Z100R Ultrafine Field Microscope. Its advantage is that it can more accurately reflect the difference of characteristics on the surface of the material, which is beneficial to the further pixel analysis.

#### 2.3.4. Fourier transform infrared spectroscopy (FTIR)
The FTIR test was conducted on the Mt and MtT using Thermo Scientific Nicolet IS5 FTIR Spectrometer in transmittance mode with 120 scans per spectrum. Samples were prepared by weighing 2 mg of each ground sample with 200 mg of dry KBr.

#### 2.3.5. X-ray diffraction (XRD)
The XRD test was performed using a Shimadzu 6000 Diffractometer with Cu-Kα radiation generated at $30 \text{ mA}$ and $40 \text{ kV}$. Mt and MtT were step-scanned as random powder mounts from $3\degree$ to $50\degree$, continuously with the $2.0\degree/\text{min}$ scanning speeds. The change in intensity of $d_{001}$ of Mt and MtT were measured in order to estimate the intercalation. In addition, the $d$ value was calculated according to Bragg equation.

#### 2.3.6. Slump and blending of two bitumen
The slump test and blending test were carried out under the UFM with a heater plate as shown in figure 1. In the slump test, the MtT bitumen and aged PJ-90 bitumen were weighed 2 mg and placed on the glass slide, respectively. Afterwards, above two bitumen were heated from ambient temperature to $180 \degree C$. The diameter was measured by the UFM’s in-built software over the whole process. In the blending test, a blue line was marked

| Mesh size [mm] | 26.5 | 19  | 16  | 13.2 | 9.5 | 4.75 | 2.36 | 1.18 | 0.6  | 0.3  | 0.15 | 0.075 |
|---------------|------|-----|-----|------|-----|------|------|------|------|------|------|-------|
| Passing [%]   | 100  | 95  | 85  | 71   | 60  | 41   | 30   | 22.5 | 16   | 11   | 8.5  | 5     |

Table 1. Aggregate gradation for AC-20.
on the glass slide in order to evaluate the fluidity of the two bitumen. Afterwards, 2 mg above bitumen samples were placed on both sides. Then, these samples were heated from ambient temperature to 180 °C. The blending process was observed under UFM.

2.4. Image processing methods

2.4.1. Gray scale arithmetic

Gray scale is the color depth of pixel point in the monochrome image, which generally ranges from 0 to 255 (the white is 255 and the black is 0). It is also called gray scale image, which has a wide range of applications in the field of image recognition. The grayscale image is obtained by the transition of pure black and pure white. The gray is obtained by adding white to black, and the different gray values are obtained by mixing pure black and pure white according to different proportion. Hence, a 2D image with bright degree information (dark and bright, black and white) become gray scale images.

However, if it is a color image, its grayscale value needs to be mapped by function. Any color is made up of red (R), green (G) and blue (B). The gray scale of color image is the pixel value of the converted monochrome image. A floating-point arithmetic was employed for color conversion. If the original color of a point is RGB (R, G, B), then, the gray scale was calculated by equation (1). Afterwards, the RGB (R, G, B) of point was substituted by RGB (Gray, Gray, Gray) of the calculated value. In this way, the color image was converted to the monochrome image.

\[
\text{Gray scale} = R \times 0.3 + G \times 0.59 + B \times 0.11
\]  

(1)

In the present work, a RAP was cut into two parts in order to keep the smooth of bottom. Afterwards, the RAP was heated from ambient temperature to 120 °C. The crack process was recorded under UFM by its in-built software. Last, a 300 × 300 pixel area of the image center was computed and analyzed by Gray Scale Arithmetic at different temperatures.

2.4.2. K-means clustering

K-means clustering is a classical algorithm in machine learning. Cluster algorithm seeks data segmentation, in which data objects in the same clusters are homogenous, while data objects in different groups are well separated. Pixel values in the same region of the image are considerably similar, and the pixels can be treated as a cluster. Such homogeneity and separation are evaluated through criterion functions. Sum-of-squared-error criterion is one of the most widely used criterion function in clustering practice. Suppose that a set of data objects \(x_j (j = 1, \ldots, N)\) are organized into K clusters \(C = \{c_1, \ldots, c_k\}\). The sum-of-squared-error criterion is defined as equation (2)

\[
J(U, M) = \sum_{k=1}^{K} \sum_{j=1}^{N} u_{kj} \|x_j - m_k\|^2 = \sum_{k=1}^{K} \sum_{j=1}^{N} u_{kj}(x_j - m_k)^2(x_j - m_k)
\]  

(2)
where $U = \{ u_{kj} \}$ is a partition matrix (equation (3)),

$$u_{kj} = \begin{cases} 1, & \text{if } x_j \in \text{cluster } k \\ 0, & \text{otherwise} \end{cases}$$  \quad (3)$$

$M = \{ m_1, \ldots, m_k \}$ is the cluster center (means) matrix (equation (4)),

$$m_k = \frac{1}{N_k} \sum_{j=1}^{N} u_{kj}x_j$$  \quad (4)$$

The partition that minimizes the sum-of-squared-error criterion is regarded as optimal. For the microscopic image, $||x_j - m_k||^2$ is the color Euclidean distance between each pixel and the corresponding cluster center. In the RGB color space, equation (2) can be rewritten to equation (5).

$$J(U, M) = \sum_{k=1}^{K} \sum_{j=1}^{N} u_{kj} \sqrt{(R_j - R_k)^2 + (G_j - G_k)^2 + (B_j - B_k)^2}$$  \quad (5)$$

3. Results and discussion

3.1. Characteristics of MtT

Figure 2 shows the XRD pattern of rhodamine intercalated montmorillonite with different concentrations. It can be seen clearly that the interlayer spacing ($d$) increased, which means that the rhodamine solution was inserted into the Mt layer successfully after stirring with the Mt. The interlayer spacing of virginal Mt was 12.88 Å. After the intercalation process, the interlayer spacing of montmorillonite increased linearly up to 20.45 Å with increasing rhodamine concentrations.

Furthermore, when the concentration was lower than 2.1 mmol L$^{-1}$, the intercalated effect was not significant and the interlayer spacing changed slightly. At 6.3 mmol L$^{-1}$ concentration, the interlayer spacing achieved the maximum. Because 6.3 mmol L$^{-1}$ was the maximum solution concentration for current intercalation process, the concentration of rhodamine was selected as 6.3 mmol L$^{-1}$ for this study, which ensured the fluorescence of the Mt.

Figure 3 shows the FTIR spectra of virginal Mt and MtT wherein the characteristic peaks observed in the FTIR spectra are summarized in table 2. For virginal Mt, the peaks at 3625 cm$^{-1}$ and 3441 cm$^{-1}$ were related to the asymmetrical stretching of Al–OH and stretching vibration of the interlayer water, respectively. The peak at 1638 cm$^{-1}$ was correspondent to the bending vibration of crystal water. Moreover, the relatively intense vibrational peaks of aluminosilicates assigned to the vibrations of Si-O-T bond ($T = Si$ or Al) were noticeable at 1088 cm$^{-1}$, 520 cm$^{-1}$ and 470 cm$^{-1}$.

Furthermore, comparing with the virginal Mt, the characteristic peaks of MtT changed slightly. It indicated that the microstructure of Mt has a good stability and not damage easily. The peak at 1640 was correspondent to
the stretching of C=C bond in benzoic ring. The peaks at 1650 and 1307 cm\(^{-1}\) were related to the stretching of C–Cl bond and C–O bond in benzoic ring. As shown in figure 3 (zoomed view), the absorption band (about 1550 cm\(^{-1}\) to 1700 cm\(^{-1}\)) of Mt appeared a ‘blue shift’ due to rhodamine intercalation. Because chlorine atoms in rhodamine molecules have an electrophilic effect for functional group enhancement. In addition, the appearance of C–O bond in benzoic ring also approved that rhodamine could be intercalated into Mt successfully.

Figure 4 shows the morphology of MtT through SEM. The morphology of the Mt was clearly observed from a 90 magnification SEM. The particle size of Mt ranged from 20 \(\mu\)m to 75 \(\mu\)m. Although the size of MtT was varied, the shape was similar. But the particle size of MtT was uncertain under microscopic view due to the samples were slices through cutting. To be sure, the particle size of MtT should be smaller than 75 \(\mu\)m, because it was milled and cut.

Figure 5 shows the morphology of MtT in the PJ-90 bitumen through SEM-EDS. The MtT was observed clearly like a rectangle plate through 3000 magnification SEM. The particle size of the MtT particle was 23 \(\mu\)m, and the surface was evenly distributed with thin strips of wrinkles. In addition, a part of the MtT particle in figure 5 was inserted into and bonded with bitumen. It was technically difficult to find the actual MtT accurately by EDS, because the MtT was coated by bitumen, creating a greater risk of inaccurate results. As shown in figure 5 (EDS to the right-hand side), although sodium was not detected by EDS, iron and zinc were detected. Particularly, zinc is a special element that can distinguish the MtT tracer from bitumen and RAP in the prepared asphalt mixture slice samples. Related results will be discussed later.

Furthermore, the morphology of MtT in the bitumen cannot reflected the ‘reality’ of blending interface directly after the mixing. Because the particle size and location of MtT was only analyzed the preliminary blending interface zone. The fluorescent effect of rhodamine and Zinc elements of MtT would be more helpful to observe the real blending interface.

In addition, the softening point, penetration and ductility results of the MtT modified bitumen samples were 49.1 °C, 84.2 dmm (25 °C) and 102.3 cm (15 °C) respectively. The changes of these bitumen properties were markedly limited; hence, these bitumen properties were mostly maintained even after the addition of MtT.

### Table 2. Characteristic peaks of virginal Mt and MtT.

| Materials | Chemical bond | Wavenumber [cm\(^{-1}\)] | Interpretation |
|-----------|---------------|--------------------------|----------------|
| Mt        | Al–OH         | 3625                     | Stretching     |
|           | –OH           | 3441                     | Stretching     |
|           | –OH           | 1638                     | Bending        |
| Si–O–T(T = Si or Al) | 1088             | Stretching     |
|           |               | 460                      | Bending        |
| MtT       | C=O           | 1640                     | Stretching     |
|           | C=O           | 1650                     | Stretching     |
|           | C=O           | 1307                     | Stretching     |
3.2. Blending of MtT bitumen and aged bitumen

Slump is a measured index of the workability or consistency of cement. In other words, it measures how easy the cement concrete is to deliver and mold. The higher the slump, the more workable the concrete. Accordingly, the workability measurement of bitumen always employs viscometer or rheometer. But viscosity and rheology cannot reflect the blending workability between two bitumen. Hence, slump was employed for blending evaluation between the MtT modified bitumen and laboratory-aged bitumen.

Figure 6 shows the slump process of the MtT bitumen with temperature increasing. It should be noted that the surface of the MtT modified bitumen was attached to some dust (grey points in the images). From 70 °C to 80 °C, the slump of MtT bitumen changed slightly. But the slump changed quickly after 90 °C. Furthermore, the bitumen was spread out on the glass slide, and gradually flowed out of the field of microscopic view.

Figure 7 shows the slump process of the aged bitumen with temperature increasing. Obviously, comparing with MtT bitumen, the surface of aged bitumen was rough and had a shallow crack at 70 °C and 80 °C. Because a temperature gradient was formed from top to bottom when a heater was employed and heated at bottom of specimens. The bottom bitumen flowed, but the top bitumen could not flow, afterwards, a crack appeared due to the ageing and force of bottom bitumen. Moreover, the slump changed slowly from 70 °C to 90 °C and quickly after 100 °C. It indicated that the aged bitumen had poor fluidity. With the help of this dust the crack phenomenon of aged bitumen can be clearly observed. This phenomenon was not observed in MtT bitumen slump test.

Figure 8 shows the diameter change of MtT and aged bitumen with temperature increasing from 1000 μm to 2000 μm. Obviously, the diameter variation of two bitumen can be divided into 3 phases. In the initial phase,
Figure 6. Slump process of the MtT modified bitumen with temperature increasing under UFM.

Figure 7. Slump process of the MtT bitumen with temperature increasing under UFM.

Figure 8. Diameter change of MtT bitumen and aged bitumen with temperature increasing.
from 50 °C to 60 °C, the temperature just exceeded the softening point (52.9 °C). The fluidity of two bitumen was poor, resulting the diameter changed slightly and remains at the initial 1000 μm. In the phase 2, form 60 °C to 110 °C, the diameter of two bitumen increased continuously and the increased trend was like a portion of a downward parabola. The slope increased significantly first (before 90 °C), indicating that the inside and outside of bitumen had been heated evenly and the fluidity of two bitumen was excellent. At 80 °C, the difference in diameter was 21% between two bitumen. After 90 °C, the slope of the diameter curve tended to be constant. In the last phase, higher than 110 °C, the bitumen drops stopped slumping due to the invariant bitumen weight and the diameter kept almost unchanged.

Furthermore, the diameter of MtT bitumen was larger than that of aged bitumen during the phase 2, as seen in figures 6 and 7. This is because the content of components with good fluidity (possibly saturated and aromatic fractions) of MtT bitumen was higher than that of aged bitumen. These components have a significant impact on the fluidity of bitumen, however, within a limited temperature range. With temperature increasing, the fluidity of two bitumen was similar again.

Figure 9 shows the blending interface between MtT bitumen and aged bitumen at different temperatures. The two binders started to flow at 70 °C and contact each other at 80 °C. From 85 °C until 100 °C, the two binders were blended together. MtT bitumen was surrounded by aged bitumen due to the fluidity difference at lower temperature. Please noted that cracks appeared on the surface of aged bitumen again, but they could not be observed on the surface of MtT bitumen. At 110 °C, MtT bitumen flowed to the aged bitumen along the cracks, leading to the engagement phenomenon between the two binders.

Furthermore, the area of final blend (whole circular area in the microscopic view) was 4.686 mm² which was measured by ultrafine field microscope after blending. According to the diameter measurement of two bitumen in figure 8, the area of MtT bitumen and aged bitumen was 3.167 and 3.078 mm², respectively. In order to use a parameter for evaluating the blending, the area ratio was calculated based on above three area. After blending, the area ratio was 0.75. Obviously, the last area of two bitumen mixture was less than additive area of individual bitumen together. It illustrated that the flow of MtT bitumen was restricted when it flowed to the aged bitumen along the cracks. There was an ‘engagement’ between MtT bitumen and aged bitumen.

3.3. Cracking process of RAP bitumen

According to cracking observation of aged bitumen, if the cracks appear on the RAP bitumen, the engagement would be proved and interface transition zone would be determined. Obviously, the crack of RAP bitumen also appeared on the surface from 90 °C to 120 °C as shown in figure 10. Before blending, the cracking process of RAP bitumen was computed by Gray Scale Arithmetic with temperature increasing.

According to gray scale arithmetic, the distribution of pixel gray scale values of the RAP bitumen at different temperatures were obtained as shown in figure 10. Obviously, a watershed appeared in the figure, indicating that there were two types of peaks. One was that the gray scale values of peaks were between 20 and 30, which represented the dark part (i.e. bitumen in the cracks). The other was that the gray scale values of peaks were between 100 and 110, which represented the bright part (i.e. dust and bitumen on the surface). In general, the
gray scale values of the peak shifted from large to small with the temperature increasing. The dust and surface bitumen integrated in the cracks, and the gray scale got smaller and smaller.

At lower temperature, from 50 °C to 80 °C, there was only one peak, and the shape and height of peaks changed slightly, but the gray scale value of peaks shifted to the direction of lower values. It illustrated that the microcracks appeared on the surface when the temperature exceeded the softening point. With the temperature increasing from 90 °C to 105 °C, the gray scale value of peaks shifted back to the initial position. It revealed that the surface bitumen began to flow, and then, flowed to the microcracks. At this point, the rate of cracking development was smaller than the flow velocity of RAP bitumen.

After 100 °C, the peak of dark part raised gradually, because the gray scale was changed due to the black increase. It demonstrated that the cracks on the surface increased, and more dust-free internal bitumen was observed. Further, at 112 °C, the peak of bright part decreased, and the peak of dark part raised. The shape and height of two peaks were similar. The occupied area of cracks and surface bitumen in the image achieved a balance, that is, the surface bitumen would sink in the crack nearby. The RAP bitumen in this condition was most suitable for forming an engagement structure with new bitumen. Furthermore, at 115 °C, the peak of bright part continued decline and the peak of dark part raised significantly. It illustrated that the flow velocity of RAP bitumen was accelerated, and the boundary between bitumen and crack was fuzzy. More internal bitumen of RAP bitumen was observed in the microscopic view.

At higher temperature, from 117 °C to 120 °C, the peak of bright part became weakness and the peak of dark part raised significantly with temperature increasing. In the microscopic view, the RAP bitumen shown a well flow status, and some dust floated on the surface.

Figure 10. Pixel curve (up) and images (down) of cracks process on the RAP bitumen surface.
In general, the transition region of temperature was 110 to 115 °C. In this temperature region, the cracks on the surface of RAP bitumen increased and cracked area achieved the maximum. It is beneficial to form the engagement interface between RAP bitumen and new bitumen.

3.4. Blending of MtT bitumen and RAP

In order to make a more comprehensive observation of the interface, the tracer based on montmorillonite called MtT was employed. It comes with two advantages, namely fluorescent effect and exclusive Zinc element.

Figure 11 shows the fluorescent effect of MtT in RAP slice. A gap between two coarse aggregates was selected for observing, as shown in left image. In the gap, the whole morphology included bitumen, fine aggregates, and fillers when the fluorescence excitation was turned off. The MtT cannot be observed directly, just like fillers. However, the MtT were observed directly when the fluorescence excitation was turned on. In the microscopic view, the morphology of MtT changed from particle to point for two reasons. First, the MtT particles were chopped up when the slice sample was prepared. Second, MtT particles immersed in the bitumen.

Furthermore, MtT was spread in the bitumen including structural bitumen and free bitumen. The weeny MtT was observed around the aggregate. According to the measured film thickness of RAP bitumen, some of them entered into the engagement transition area of MtT bitumen and RAP bitumen. However, it hard to distinguish the boundary between MtT bitumen and RAP bitumen only with the help of MtT.

After a few error points were deleted which appeared on the aggregate surface, figure 12 shows the distribution of Zinc (green points) in MtT. A gap between two coarse aggregates was also selected for observing under SEM with ×30 magnification. Similar to figure 11, the Zinc was mainly distributed in the bitumen.
including structural bitumen (aggregates periphery) and free bitumen (dark zone). Compared to figure 11, few element points distributed in the free bitumen. Most element points surrounded the aggregate surface closely. The distance between element points and aggregate surface was less than 10 μm. The MtT had flowed into RAP bitumen. It directly validated the nucleation of MtT again and confirmed that the blending status was well.

However, the method of elements mapping cannot be used to detect the interface between MtT bitumen and RAP bitumen. This is because the MtT bitumen and RAP bitumen are the same kind of materials which have the same conductivity. Hence, the distinction of two binders was slight.

In general, the phenomenon of engagement was verified, and the blending status of two binders was well. But the interface between two binders was still difficult to observe, and the thickness of the blending transition zone could not be determined. Meanwhile, machine learning is not a new field, but it is quickly growing and provides a possibility to read detailed information from images. An algorithm called K-means clustering was employed to distinguish the interface between two binders. K-means clustering belongs to unsupervised learning, that is, the collected data cannot be classified, but the characteristic of collected data can be learnt by machine. Afterwards, the similar data are classified into the same dataset.

A gap between two coarse aggregates was also selected for observing under UFM and machine learning. With the help of machine learning, figure 13 shows clustering results of RAP slice by K-means. The collected pixel data in the whole image was divided into 4 categories. Yellow symbolized aggregates, red symbolized RAP bitumen on the aggregate surface, light blue symbolized interfacial transition zone and dark blue symbolized MtT bitumen.

In figure 13, the coarse aggregates on two sides, the fine aggregates and fillers in the gap were clearly analyzed by machine learning. Furthermore, the RAP bitumen on the aggregate surface, the interface transition zone and the MtT bitumen were also distinguished. According to the scale under the microscope, the thickness of the RAP bitumen (red) on the aggregate surface and the interface transition zone (light blue) could be accurately measured in the image. The initial average thickness of the RAP bitumen was 10 μm approximately, but the thickness after blending was 3 to 8 μm, which was significantly less than the initial thickness. Moreover, a blending transition zone was produced by RAP bitumen and MtT bitumen, and the thickness of transition zone was 4 to 8 μm.

4. Conclusions

This study investigated the blending interface of MtT bitumen and RAP bitumen with the help of microscope and machine learning with the aim of revealing the blending mechanism of RAP. On the basis of the above described results and discussion, the following conclusions can be drawn:

The MtT modified bitumen has fluorescent effect and exclusive Zinc which could be observed under FM and SEM. The overall impacts on the bitumen performance (i.e. softening point, penetration, ductility) were limited. In addition, the fluidity of MtT bitumen was better than that of aged bitumen. Furthermore, the blending interface between two different bitumen was engagement like gears. According to gray scale arithmetic, the cracks on the surface of RAP bitumen increased and cracked area achieved the maximum at the temperature region of 110 °C to 115 °C. It is a suitable heating temperature and beneficial to form the engagement interface between RAP bitumen and new bitumen. Moreover, the interface transition zone between two different bitumen could be detected through fluorescent effect, but the thickness of the blending transition zone could not
be determined directly from microscopic images. With the help of machine learning, the thickness of interfacial transition zone was determined as 4 to 8 μm.

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Data availability statement

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

Conflicts of interest

The authors declared that they have no conflicts of interest to this work.

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