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Aging Characteristics of Plant Hot-Mix Recycled Asphalt and Its Induced Aging on New Asphalt

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Abstract: In order to explore the aging process and mechanism of new and old asphalt in plant hot-mix recycled asphalt mixture during the mixing and use process, the liquid viscosity test and low-temperature bending creep test are carried out to test the dynamic viscosity, stiffness modulus, creep rate, and low-temperature flexibility of the matrix asphalt, recycled asphalt, and old and new asphalt after rolling thin film oven test (RTFOT) aging and pressurized aging vessel (PAV) aging. The macroscopic performance attenuation law of new and old asphalt during the aging process in thermal regeneration is compared and analyzed. After that, the aging process and mechanism of new and old asphalt are explored by infrared spectroscopy and differential calorimetric analysis scanning. The results show that RTFOT aging and PAV aging make the viscosity of recycled asphalt rise significantly and the low temperature performance decline rapidly. After RTFOT aging and PAV aging of new and old asphalt, the dynamic viscosity and low-temperature performance change range is much higher than that of the matrix asphalt. Some technical indicators are even closer to the recycled asphalt after aging, which proves that its aging speed is faster than that of the matrix asphalt. Meanwhile, the results of infrared spectroscopy and differential scanning calorimetry analysis show that in addition to the independent aging of new asphalt and recycled asphalt, there is also a chemical effect between them–that is, some active groups in recycled asphalt have a more obvious promotion effect on the aging process of new asphalt, here called "induced aging". This induced aging changes the aging mechanism of the matrix asphalt by changing the aging process of it, which greatly limits the popularization and application of thermal regeneration technology.

Keywords: aging mechanism; hot recycling; induced aging; matrix asphalt; performance degradation; recycled asphalt

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Citation: Chen, H.; Tang, G.; Wang, X. Aging Characteristics of Plant Hot-Mix Recycled Asphalt and Its Induced Aging on New Asphalt. *Processes* **2022**, *10*, 908. https://doi.org/10.3390/pr10050908

Academic Editor: Chi-Min Shu

Received: 16 March 2022
Accepted: 26 April 2022
Published: 5 May 2022

1. Introduction

After more than 20 years of rapid development of highways in China, both mileage and ordinary roads have come first [1]. However, with the continuous increase of road mileage and service life dominated by asphalt pavements, the recycling of old asphalt pavements has become increasingly serious and urgent. Waste asphalt concrete will bring double pressure of environmental pollution and resource waste to society, and effective recycling of it is also a social issue related to the national economy and maintenance of sustainable social development. Although the recycling methods of old asphalt mixture include hot recycling, cold recycling, and warm recycling that has emerged in recent years, the plant hot-mix recycling is still the main method for current waste asphalt mixture recycling due to its relatively strict process and reliable construction quality [2–4]. Many researches on the plant hot-mixed recycling technology have been carried out. Generally, the results are mainly related to two aspects: first, to achieve the purpose of improving the pavement performance of hot recycling mixture by adding additives (such as rejuvenators, new asphalt, etc.) in order to soften the aged asphalt and optimize the production and construction technology of plant hot-mix recycling technology [5–7]. This kind of research on RAP recycling mainly focuses on the volatilization and loss of light components in the aging process of asphalt from the physical perspective, and the main role of the rejuvenators is to supplement the lost light components and increase the aging resistance of the asphalt [8,9]; second, to study the recycling aging mechanism, the fusion mechanism of new and old asphalt, or the interface adhesion characteristics of recycled asphalt and new and old aggregates through microscopic methods [10,11]. However, there are few relevant reports on the aging of recycled asphalt in the production, construction, and long-term use of hot recycled mixture and its impact on the aging process and mechanism of new asphalt [12–14]. The road performance data of hot recycled asphalt mixture measured in the indoor test is often not bad, and it at least meets the specification requirements. However, there is still a big gap in the durability of hot recycling asphalt pavement compared with new asphalt pavement. The inherent reason is probably that during the production and use of hot recycling asphalt mixture, some active groups in recycled asphalt have a strong induction and promotion effect on the aging of matrix asphalt. Thus, it accelerates the aging of new asphalt, and finally leads to poor service durability of reclaimed asphalt pavement. On this basis, this article first analyzes and tests the dynamic viscosity and low-temperature performance of recycled asphalt and new asphalt mixture after being subjected to rolling thin-film oven test (RTFOT) aging and pressure aging vessel (PAV) aging. In addition, taking recycled asphalt and new asphalt as a reference, the performance change law of new and old asphalt mixture before and after aging is analyzed. Then, through the analysis of microscopic test technology, it is demonstrated that the recycled asphalt in new and old asphalt has an obvious inducing aging effect on new asphalt. This induced aging promotes the rapid aging of new asphalt in plant hot-mix recycled mixture, resulting in the rapid attenuation of its road performance. This is also a problem that has been neglected in the current research of hot recycling technology. The results can provide a basis for subsequent research about deactivation treatment of the active groups in recycled asphalt, and can also arouse the attention of road builders, designers, and researchers on the recycling of reclaimed asphalt pavement, which is expected to improve the service life of recycled asphalt pavement.

2. Experiment

2.1. Raw Material

The raw materials of this paper mainly include the following three kinds:

(1) Recycled asphalt: the waste mixture of Chongqing to Sui-ning expressway (open to traffic by end 2007) was prepared by centrifugal extraction;
(2) New asphalt: CNOOC $70^{\#}$ matrix asphalt;
(3) Old and new asphalt mixture: recycled asphalt and new asphalt were evenly mixed according to the mass ratio of 3:7 by stirring 5 min at 140 ± 5 °C and 500 ± 50 r/min. (The mass ratio of 3:7 is determined according to the requirements of *Technical Specifications for Highway Asphalt Pavement Recycling JTG/T5521-2019*).

Relevant technical indexes are listed in Table 1.

Table 1. Technical indexes of the matrix asphalt and recycled asphalt.

Asphalt Type	25 °C Penetration/(0.1 mm)	15 °C Ductility/cm	Softening Point/°C
New asphalt	63.3	135.3	48.9
Recycled asphalt	35.3	17.4	63.1
Old and new asphalt	51.6	97.2	54.3

2.2. Performance Test and Characterization

The relevant test parameters of rolling thin-film oven test (RTFOT) aging are as follows: test temperature, 163 ± 0.5 °C; mass of each sample, 35 ± 0.5 g, each group of five; hot air velocity, 4000 ± 200 mL/min; sample rotation rate: 15 ± 0.2 r/min; aging time, 85 min; the same below. The relevant test parameters of pressure aging vessel (PAV) are as follows:

pressure, 2.1 ± 0.1 MPa; test temperature, 100 ± 0.5 °C; mass of each sample, 50 ± 0.5 g, each group of five; thickness of asphalt film, 3.2 mm; aging time, 20 h ± 10 min; the same below.

Dynamic viscosity test: the dynamic viscosity of the three asphalts at 120 °C before and after aging is tested by Brookfield Viscometer according to the determination standard of liquid viscosity in GBT22235-2008;

Low temperature bending creep (BBR) test: test the stiffness modulus S and creep rate m at −12 °C and −18 °C before and after test aging of the three asphalts;

Low temperature flexibility test: according to the test method of building waterproof coatings GB/T16777-2008, the low-temperature flexibility of the three asphalts before and after aging is tested respectively;

Infrared Radiation spectroscopy (IR) test: vertex 70 infrared analyzer produced by Bruker company was used to analyze the infrared spectra of the three asphalts before and after aging test, with 30–8000 cm^{-1} of spectral scanning range, 4 cm^{-1} of resolution and 16 scans.

Differential scanning calorimetry (DSC) test: the glass transition temperature, nitrogen atmosphere and heating rate of the three asphalts before and after aging were measured by TA Q20 differential scanning calorimeter in a nitrogen atmosphere, and the heating rate was 10 °C/min.

2.3. Statistical Analysis

The dynamic viscosity of the three asphalts at 120 °C was tested five times before and after aging. The stiffness modulus S and creep rate m at −12 °C and −18 °C of the three asphalts before and after aging were tested three times. One-way ANOVAs with LSD multiple comparison analysis were performed using IBM SPSS Statistics 26 and $p < 0.05$ as the significance level.

3. Results and Discussion

3.1. Dynamic Viscosity Test

The aging degree of asphalt is closely related to its dynamic viscosity, that is, the dynamic viscosity of asphalt can describe its aging degree more intuitively. With the deepening of asphalt aging, its dynamic viscosity will also increase. The results of dynamic viscosity at 120 °C of new asphalt, recycled asphalt, and their mixture were tested, respectively. They are shown in Figure 1.

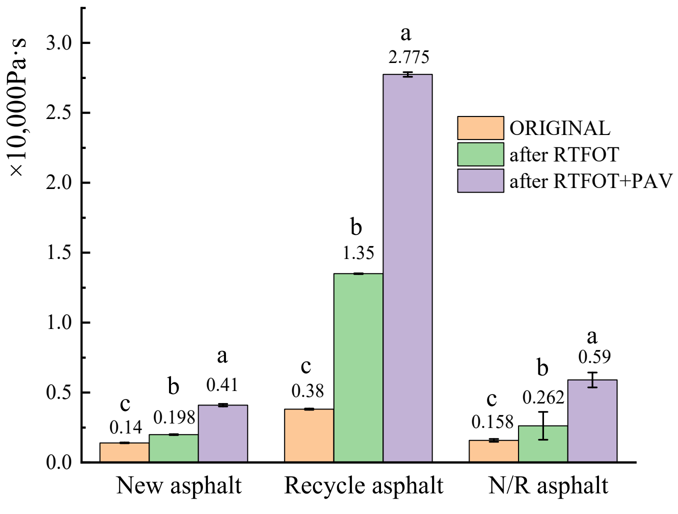

Figure 1. Dynamic viscosity of the three asphalts at 120 °C before and after aging. a, b, c symbols on each column represent significant difference at 0.05 level.

It can be seen from Figure 1 that after RTFOT aging and PAV aging, the dynamic viscosity of the three asphalts at 120 °C has increased to varying degrees ($p < 0.05$). In particular, long-term aging has an obvious effect on the improvement of asphalt viscosity. After RTFOT aging and PAV aging successively, it is found that the dynamic viscosity of recycled asphalt increases significantly, and the aging rate is alarming. It indicates that the anti-aging ability of recycled asphalt is very low and thus it is not suitable for direct use of a binder for hot recycling. The dynamic viscosity of new asphalt is close to that of old and new asphalt. However, after RTFOT aging and PAV aging, the dynamic viscosity of new asphalt increased by 42.1% and 192.8%, respectively, compared with no aging, and the dynamic viscosity of new and old asphalt increased by 65.8% and 273.4%, respectively. The growth rate of new and old asphalt was significantly higher than that of new asphalt. Further analysis of the viscosity changes of new asphalt and old and new asphalt during aging, it is also found that there is little difference in the initial viscosity between new and old asphalt, and the difference is only 188 Pa·s. After RTFOT aging, the difference reaches 632 Pa·s. After further PAV aging, the difference reaches 1800 Pa·s. This demonstrates that under the same aging, the aging rate of old and new asphalt is significantly higher than that of new asphalt. The reason for this needs to be further studied.

It can be inferred from the dynamic viscosity of the three asphalts after RTFOT aging and PAV aging that the recycled asphalt not only ages rapidly, but also accelerates the aging process of new asphalt through a certain chemical action. This fact is extremely unfavorable to the recycling and utilization of waste plant hot-mix asphalt mixture, and also a key problem that has to be solved by the plant hot-mix recycling technology.

3.2. BBR Low Temperature Bending Performance

The creep rate and stiffness modulus measured by the BBR low-temperature flexural creep test are used to evaluate the low-temperature performance of the asphalt. The creep rate reflects the stress relaxation ability of asphalt, and is positively related to the low-temperature crack resistance of it. The greater the stiffness modulus is, the worse the low temperature performance of asphalt will be [15]. In this paper, the residues of new asphalt, recycled asphalt, and new and old asphalt after RTFOT aging and PAV aging have been subjected to the test. The results are shown in Table 2.

Table 2. BBR test results of the three asphalts.

Aging Type	Asphalt Type	−12 °C		−18 °C	
		Stiffness Modulus S/MPa	m	Stiffness Modulus S/MPa	m
ORIGINAL	New asphalt	116	0.316	131	0.282
	N/R asphalt	142	0.291	208	0.258
	Recycled asphalt	163	0.257	279	0.223
RTFOT	New asphalt	135	0.299	228	0.268
	N/R asphalt	171	0.271	334	0.225
	Recycled asphalt	182	0.231	347	0.212
RTFOT + PAV	New asphalt	159	0.278	276	0.243
	N/R asphalt	202	0.244	405	0.217
	Recycled asphalt	211	0.209	429	0.188

From Table 2, the stiffness modulus and creep rate m of the three asphalts change significantly ($p < 0.05$) before and after aging. After RTFOT aging, the stiffness modulus S of the three asphalts at −12 °C and −18 °C all increase significantly. After further aging of PAV, the stiffness modulus continues to increase. It shows that the stress required for the deformation of asphalt increases, and the asphalt brittleness also increases. However, after RTFOT aging and PAV aging, the creep rate m of the three asphalts at −12 °C and −18 °C shows a change law opposite to the stiffness modulus. This indicates that with the deepening of aging, the stress relaxation ability of the three asphalts decreases to varying degrees, and the low-temperature crack resistance deteriorates obviously. It should be noted that although the stiffness modulus and creep rate of the three asphalts have changed significantly after aging, the following laws can be found: first, regardless of whether it is RTFOT aging or PAV aging, the change magnitude order of stiffness modulus and creep rate at −12 °C and −18 °C is always new asphalt > recycled asphalt > matrix asphalt; second, after RTFOT aging and PAV aging, the stiffness modulus and creep rate of old and new asphalt are close to those of recycled asphalt. The former is significantly higher than that of matrix asphalt, while the latter is significantly lower than that of matrix asphalt.

Further analysis also finds that the addition of recycled asphalt will cause a sudden change in the stiffness modulus and creep rate of the matrix asphalt during RTFOT aging and PAV aging. Thus, it is speculated that in the RTFOT aging and PAV aging of old and new asphalt, in addition to the respective aging of the matrix asphalt and recycled asphalt, there may also be a special chemical interaction between them. This chemical action accelerates the aging process of the matrix asphalt to a great extent and changes the aging mechanism of it. This special chemical action is called "induced aging" in this article.

3.3. Low Temperature Flexibility Analysis

The low temperature flexibility of polymer materials is linearly and negatively correlated with the degree of aging [16]. The test results of new asphalt, new and old asphalt, and recycled asphalt are shown in Table 3.

Table 3. Cracks of three asphalt samples before and after aging.

Asphalt Type	Original Sample	RTFOT Aging	RTFOT + PAV Aging
New asphalt	None	Little and thin	More and thin
New and old asphalt	None	More and thick	More and thick
Recycled asphalt	More	More and thick	Many and thick

Comparative observation revealed that the new asphalt and the new and old asphalt did not show any cracks in their original form, while the recycled asphalt cracked more obviously. This indicates that the low-temperature flexibility of new asphalt and new and old asphalt is better, while that of recycled asphalt is poor. The cracks after RTFOT aging show that compared with new asphalt, the low temperature flexibility of new and old asphalt deteriorates rapidly, and the further aging of recycled asphalt leads to a significant decrease in its low temperature flexibility. After further PAV aging, the new asphalt has more obvious cracks with finer and evenly distribution. The low-temperature cracking of new and old asphalt and the recycled asphalt is further enhanced. The cracks are numerous and thick, indicating that its low-temperature flexibility decreases seriously and has remarkable brittleness characteristics. After RTFOT and PAV aging, the low-temperature flexibility deterioration rate of new and old asphalt is significantly faster than that of new asphalt, and finally becomes close to that of recycled asphalt after further aging by RTFOT and PAV. The low-temperature flexibility test results show that recycled asphalt not only has poor low-temperature flexibility and weak anti-aging ability, but also that a small amount of recycled asphalt may accelerate the aging process of new asphalt. This is not conducive to the durability of plant hot-mix recycled asphalt pavement.

3.4. Infrared Spectroscopy Analysis of Induced Aging

As one of the most commonly used methods to analyze the chemical structure of materials, infrared spectroscopy can be used to qualitatively and quantitatively analyze the types and structures of chemical functional groups of materials. In this paper, the chemical functional groups of the matrix asphalt, new and old asphalt, and recycled asphalt before and after aging are tested, and the types of functional groups and the changes in the absorption peak strength are qualitatively analyzed. The change in the area of the absorption peak of the functional group is quantitatively calculated to explore its micro-aging mechanism.

3.4.1. Qualitative Analysis of Infrared Spectroscopy

The infrared spectra of the matrix asphalt, new and old asphalt, and recycled asphalt are shown in Figures 2–4. The vicinity of 1700 cm^{-1} is the carbonyl C=O vibration absorption peak. The vicinity of 1600 cm^{-1} is the stretching vibration absorption peak of the benzene ring skeleton and its unsaturated double bond. The absorption peak near 1450 cm^{-1} is the superposition of the bending vibration of methylene-CH_2- and the asymmetric bending vibration of methyl-CH_3. The absorption peak near 1030 cm^{-1} is attributed to the sulfoxide S=O stretching vibration [17].

As shown in Figure 2, the absorption peak intensities near 1030 cm^{-1} and 1700 cm^{-1} are recycled asphalt > new and old asphalt > matrix asphalt. This indicates that the original recovered asphalt contains the most carbonyl C=O and sulfoxide groups S=O, and there are basically no such two groups in the matrix asphalt. It proves that the aging degree of asphalt has a greater correlation with the content of carbonyl and sulfoxide groups. This is because the sulfur and carbon in the asphalt are oxidized into sulfoxide group S=O and carbonyl group C=O, respectively, during the aging process. Relative to Figure 2, in Figure 3, the absorption peaks of recycled asphalt and new and old asphalt near 1030 cm^{-1} and 1700 cm^{-1} both show significant enhancement, while the absorption peak intensity of matrix asphalt does not change significantly. It shows that the aging effect of RTFOT on the matrix asphalt is not significant, but quite significant on the new and old asphalt and recycled asphalt. Upon further analysis of Figures 3 and 4, it is also found that PAV aging strengthens the absorption peaks of the three asphalts near 1030 cm^{-1} and 1700 cm^{-1}. However, in terms of the reinforcement, it is obvious that the matrix asphalt is the smallest, and the old and new asphalt is close to the recycled asphalt. The reason for this is that the mass ratio of recycled asphalt in the original sample of old and new asphalt is only 30%. The aging speed during the RTFOT aging and PAV aging process is quite close to that of pure recycled asphalt, but far from that of the matrix asphalt. Thus, in the aging process of new and old asphalt, instead of pure RTFOT aging and PAV aging of recycled asphalt and the matrix asphalt, there may be a chemical effect that accelerates the aging process of new asphalt due to their interaction, that is, the active components in recycled asphalt (such as active free radicals) induce the accelerated aging of the matrix asphalt.

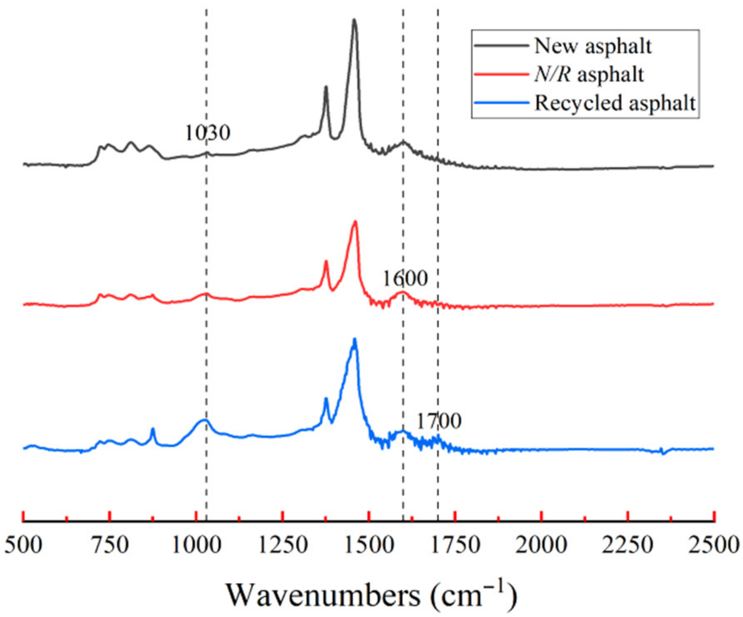

Figure 2. Infrared spectra of three original asphalts.

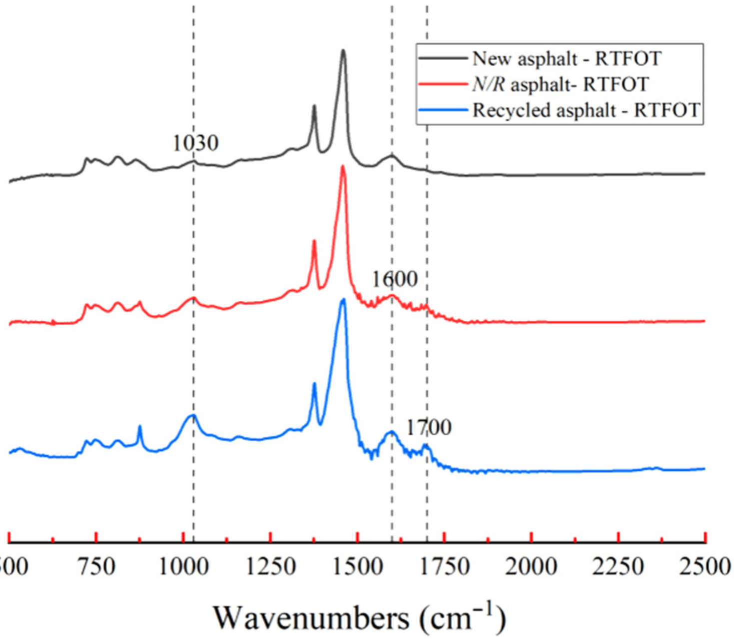

Figure 3. Infrared spectra of three asphalts after RTFOT aging.

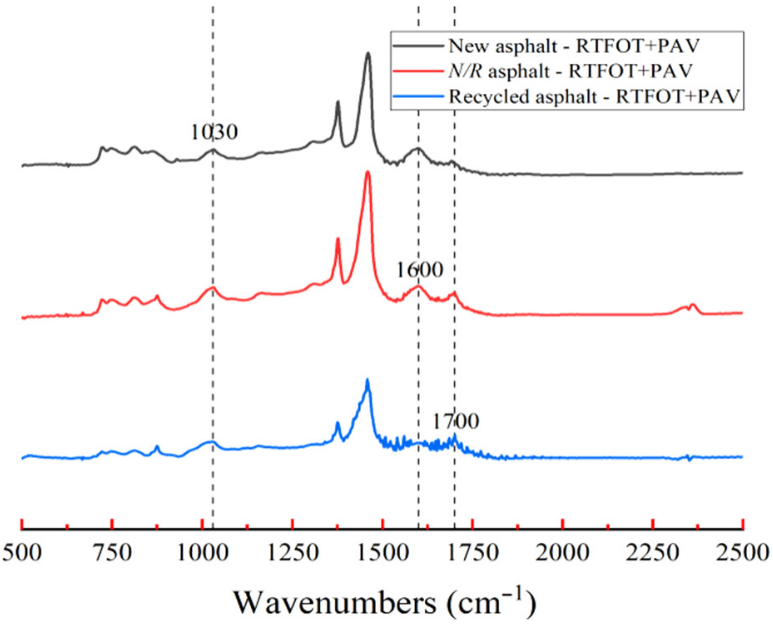

Figure 4. Infrared spectra of three asphalts after RTFOT and PAV aging.

3.4.2. Quantitative Analysis of Infrared Spectroscopy

In order to further confirm the existence of induced aging in old and new asphalt, and considering that the characteristic functional groups with significant changes in the aging process are mainly carbonyl and sulfoxide, this paper characterizes their aging process and mechanism by calculating the peak area changes of these two functional groups before and after aging. The peak area changes of the two functional groups before and after aging characterize their aging process and mechanism. In order to eliminate the error caused by the difference of each sample in the preparation, the concept of the functional group index is introduced. The functional group index refers to the ratio of the area of the absorption peak of a functional group to the area of all functional groups in a certain wave number range [18,19]. The functional group index mentioned in this article is the ratio of the area of the absorption peak of carbonyl C=O and sulfoxide group S=O to the sum of the area of the peak region in 2000–600 cm^{-1}. The area of the absorption peak is obtained by integration through Origin 8.0 software. The functional group index is calculated as follows:

$$I_{X=O} = \frac{A_{X=O}}{\sum A_{2000 \sim 600\ \mathrm{cm}^{-1}}} \tag{1}$$

where, $X = C = O, S = O$.

Firstly, the carbonyl group, sulfoxide group, and benzene ring in Figures 2–4 are integrated in the peak region of 2000–600 cm^{-1} to calculate the three functional group indices. The results are shown in Table 4, Figures 5 and 6.

From Figures 5 and 6 and Table 4, it can be seen that after RTFOT aging and PAV aging, the sulfoxide group index of the matrix asphalt continues to increase. Thus, the sulfur element in the matrix asphalt is oxidized to the sulfoxide group in succession during aging. During the RTFOT aging and PAV aging process, the change law of the sulfoxide group of recycled asphalt is just opposite to that of the matrix asphalt. This is because the sulfur element in recycled asphalt mainly exists in the form of sulfoxide group. During the aging process, some sulfoxide groups generate hydrogen sulfide, resulting in the decrease of the sulfoxide group index. The sulfur element in the matrix asphalt mainly exists in the form of elemental or other chemical states, and is oxidized to the sulfoxide group, which leads to the increase of its sulfoxide group index. However, in the aging process of old and new asphalt, both the sulfoxide group of recycled asphalt decreases and the sulfoxide group of the matrix asphalt increases. Therefore, the sulfoxide group index is relatively constant in RTFOT aging, but it still shows an upward trend after PAV aging.

Table 4. Characteristic functional group indexes of three asphalts before and after aging.

Characteristic Functional Group Coefficient I		Carbonyl Index $I_{C=O}$	Sulfoxide Index $I_{S=O}$
The matrix asphalt	Original sample	0.0113	0.0281
	RTFOT	0.0432	0.0393
	RTFOT + PAV	0.0530	0.0506
New and old asphalt	Original sample	0.0370	0.0504
	RTFOT	0.0798	0.0490
	RTFOT + PAV	0.0954	0.0646
Recycled asphalt	Original sample	0.0559	0.0813
	RTFOT	0.0856	0.0728
	RTFOT + PAV	0.0978	0.0585

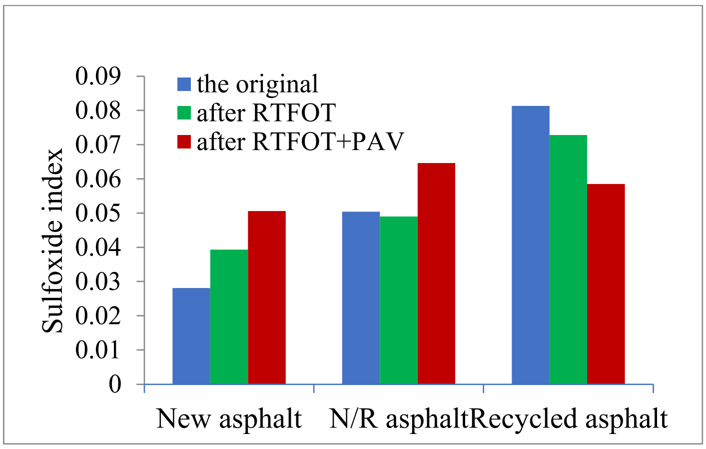

Figure 5. Sulfoxide index of the three asphalts before and after aging.

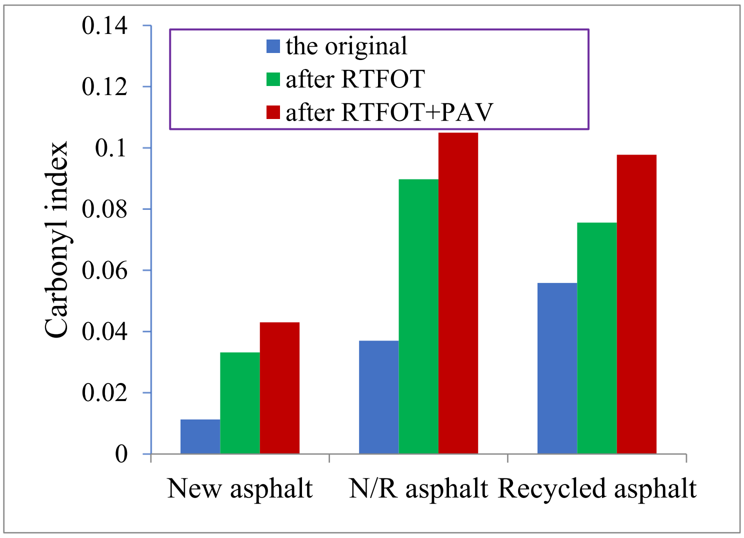

Figure 6. Carbonyl index of the three asphalts before and after aging.

Next, we compare and analyze the changes of carbonyl index after RTFOT aging and PAV aging. The relationship between carbonyl indexes in three original asphalts is recycled asphalt > new and old asphalt > matrix asphalt. After RTFOT aging, the carbonyl index of the three asphalts increases significantly, with an increase of 0.0297, 0.0428 and 0.0319, respectively. Obviously, the carbonyl index of new and old asphalt increases largely. The difference between the carbonyl indexes of recycled asphalt is greatly reduced, and that of the matrix asphalt is increased. Therefore, compared with the matrix asphalt, the anti-aging ability of new and old asphalt declines rapidly in the short-term aging process. It can be inferred that the aging process of new and old asphalt is complicated, not just the aging of the matrix asphalt and recycled asphalt. There may be an induced aging that can significantly promote the aging process of the matrix asphalt, resulting in a significant acceleration of the aging of the matrix asphalt in the new and old asphalt.

3.5. Differential Scanning Calorimetry (DSC)

The aging degree is evaluated by studying the glass transition temperature T_g of new asphalt, new and old asphalt before and after short-term and long-term aging, respectively, and the change of temperature transition range around TG [20]. Then, the induced aging effect of old asphalt on new asphalt in the aging process of old and new asphalt mixture is demonstrated. The glass transition temperature T_g and its temperature transition range of the matrix asphalt, old and new asphalt, and old asphalt before and after aging are measured by differential calorimetry scanning method. The results are shown in Table 5.

Table 5. Glass transition range and glass transition temperature of three asphalts before and after aging.

	The Matrix Asphalt		New and Old Asphalt		Recycled Asphalt	
	Glass Transition Range/°C	T_g/°C	Glass Transition Range/°C	T_g/°C	Glass Transition Range/°C	T_g/°C
Original sample	8.01–44.11	8.69	8.85–45.21	9.68	11.82–45.88	16.90
After RTOFT	9.68–46.15	10.13	10.91–45.49	14.75	12.66–46.82	17.22
After RTOFT and PAV	11.23–46.86	13.18	12.48–46.92	17.61	14.85–47.24	19.33

The results in Table 5 show that with the deepening of aging, the glass transition range of the three asphalts has shifted significantly to a high temperature. The glass transition temperature also increased in varying degrees. There may be two reasons: one is that RTFOT aging and PAV aging lead to partial crosslinking of the molecular structure of asphalt, which restricts the activity of the molecular segments of the main chain and reduces the chain length between adjacent crosslinking points; the other is that the side chain carbonyl is introduced into the molecular main chain of asphalt, which increases the rigidity of asphalt main chain molecules [21]. Further comparing the glass transition temperature of the three asphalts, it is also found that the glass transition temperature of old and new asphalt is significantly higher than that of the matrix asphalt. After long-term aging, its glass transition temperature (17.61 °C) is much higher than that of matrix asphalt after long-term aging (13.18 °C). It is also close to the glass transition temperature of recycled asphalt after short-term aging, indicating that the aging rate of old and new asphalt is significantly higher than that of matrix asphalt.

4. Conclusions

(1) After RTFOT aging and PAV aging, the dynamic viscosity and low temperature properties of new and old asphalt are significantly higher than those of the matrix asphalt. The viscosity of recycled asphalt with high viscosity increases exponentially after RTFOT aging and PAV aging, indicating that recycled asphalt should not be directly used as the binder of hot recycled mixture.

(2) The qualitative analysis and quantitative calculation results of functional groups of infrared spectrum show that the carbonyl index and sulfoxide index of old and new asphalt will increase significantly during RTFOT aging and PAV aging, and the increase range is much larger than that of matrix asphalt. In addition to the independent aging of the matrix asphalt and recycled asphalt, there is also an "induced aging" to accelerate the aging of new asphalt. This induced aging significantly accelerated the aging process of new asphalt and changed its aging mechanism.

(3) The glass transition temperature of old and new asphalt increases significantly higher than that of matrix asphalt, which is close to the glass transition temperature of recycled asphalt after short-term aging. It is also proved that the aging rate of old and new asphalt is significantly higher than that of new asphalt.

Author Contributions: Conceptualization, H.C.; methodology, H.C.; software, G.T.; validation, H.C., G.T. and X.W.; analysis of date, H.C. and G.T.; language organization, H.C.; writing—original draft preparation, H.C.; PAV test, G.T. and X.W. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: The data presented in this study is available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

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