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The Impact of Heterogeneity of Aggregates Coated with Asphalt Mortar on Their FTIR Spectra and Spectral Reproducibility

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Abstract: Since FTIR is a sensitive micro-region measurement method, research on the impact of the heterogeneity of both aggregates and asphalt mortar is meaningful and comprehensive for accurate measurement with FTIR spectroscopy. In this paper, the impact of the heterogeneity of aggregates coated with asphalt mortar on their FTIR spectra and spectral reproducibility was creatively studied. The comparative analysis of the respective absorption peaks indicated that the characteristic absorption peaks of the aggregate coated with asphalt mortar were the superposition of the respective absorption peaks of its components. And research on the spectra of the coated aggregates obtained from the same batch of asphalt mixture fabricated at the same time showed that significantly different peak intensities could be affected by minor variations in their components due to the heterogeneity. Furthermore, statistical analysis suggested that the original spectral reproducibility of the coated aggregates was greatly affected by their heterogeneity, with a high coefficient of variation values. In conclusion, the heterogeneity of the coated aggregates could affect peak intensities and spectral reproducibility in micro-regions.

Keywords: aggregates coated with asphalt mortar; heterogeneity; spectral reproducibility; statistical analysis; ATR-FTIR

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1. Introduction

Asphalt pavement is extensively used in road construction because of its excellent performance on the road, including attributes such as excellent resistance, comfortable driving, and convenient maintenance [1,2]. The conditions of asphalt pavement are important for driving safety, speed, and comfort [3]. Therefore, the rapid and accurate measurement of an asphalt mixture is meaningful for real-time intelligent monitoring of pavement conditions.

Fourier-transform infrared spectroscopy (FTIR) is a rapid testing method to characterize the molecular properties of organic and inorganic materials in heterogeneous and complex mixtures [4], such as geological samples [5], archeological materials [6], and painting materials [7]. In the past few decades, FTIR, including attenuated total reflection Fourier-transform infrared (ATR-FTIR) spectroscopy, has also been widely used in measuring asphalt materials [8–10]. These studies have achieved a lot of results, such as the analysis of oil sources [11,12], asphalt aging mechanism [13–15], modifier types and dosage of asphalt performance and structure of the degree of influence [16–19], and recycled asphalt [20,21], among others. However, it seems that in the past, FTIR spectroscopy was mainly used for measuring asphalt binders, but not directly for asphalt mixtures.

Asphalt mixture is a heterogeneous material [22,23] due to its various components with different properties and characteristics, such as aggregates, asphalt mortar, and air voids, which are combined to form a complex three-dimensional structure with diverse interactions [24]. The gradation and content of aggregates, the content of asphalt, and the three-dimensional geometry features of aggregates all contribute to the heterogeneity of asphalt mixtures [25], which affects the mechanical strength, adhesion properties, and resistance to environmental factors such as water and temperature. The above studies show that the heterogeneity of asphalt mixture has become an important research field in road engineering.

Asphalt mortar is also highly heterogeneous as an important component of the asphalt mixture [26], which directly affects the performance of asphalt mixtures [27]. In recent years, some researchers also have paid attention to the research of asphalt mortar (asphalt + filler) with FTIR spectroscopy. Zhang et al. [28] found that the infrared characteristic absorption peaks of asphalt mortar are the superposition of absorption peaks of mineral powder and asphalt. Jing et al. [29] observed that asphalt mortar had all functional groups of its component materials. Du et al. [30] used FTIR to characterize the functional groups and differentiate the interaction between asphalt and limestone mineral filler. Specifically, Zhang et al. [31] used the obtained spectral images of functional compounds to study the heterogeneous mortar micro-regions using ATR-FTIR. However, it seems that past studies on the heterogeneity of the asphalt mixture using FTIR spectroscopy mainly focused on asphalt mortar, ignoring aggregates. As aggregates have an indispensable contribution to the heterogeneity of asphalt mixture, research on aggregates coated with asphalt mortar with FTIR spectroscopy is comprehensive and crucial for future rigorous studies. Therefore, research on the effect of the heterogeneity of aggregates coated with asphalt mortar on their spectra is meaningful for accurate measurement with FTIR spectroscopy.

This study mainly focused on the impact of the heterogeneity of aggregates coated with asphalt mortar on its FTIR spectra and spectral reproducibility. Firstly, spectra of fine aggregates, coarse aggregates, and mineral powders were collected by FTIR spectroscopy in transmission mode via a KBr squashing method, followed by analysis of the characteristic peaks. Secondly, spectra of coated aggregates obtained from the same batch of asphalt mixture fabricated at the same time were measured by ATR-FTIR spectroscopy, followed by the spectral comparative analysis between asphalt and coated aggregates. Finally, a statistical analysis of the spectral reproducibility of aggregates coated with asphalt mortar with ATR-FTIR spectroscopy was carried out.

2. Principle of FTIR Technology

There are two main methods commonly used for infrared (IR) spectroscopic detection: transmission mode and reflection mode, which are determined by whether or not an infrared radiation beam passes through the sample [32].

2.1. Transmission Mode [13]

Transmission mode was the earliest used technology since the introduction of FTIR spectroscopy to the detection of asphalt materials. Due to the demand of the IR beam to penetrate the sample, a solid or powdery test sample should be made into a thin film or plate, which is suitable for mineral powder and aggregates, but not for asphalt mixture.

2.2. Reflection Mode

The ATR-FTIR technology invented by Fahrenfort and Harrick [33] is a technique that enables direct sample measurement using the IR beam's total reflection phenomenon, which has several advantages over the transmission mode [13]. Compared with the transmission method, the ATR-FTIR technology primarily used to examine the surface of the sample only requires the sample to be placed in tight contact with the surface of the ATR crystal, requiring less effort and time for sample preparation [34]. For these advantages, ATR-FTIR has become a common way mainly for analyzing asphalt materials [35–37]. In this study,

the ATR-FTIR technique is creatively used for measurement of the surface of the aggregates coated with asphalt mortar.

3. Materials and Methods

3.1. Materials

- Asphalt. JL-70 base asphalt was used and its physical properties are shown in Table 1;
- Aggregates. Granite was used as the mineral powder and aggregate, and limestone was used as the coarse aggregate. Mass percentage of coarse aggregate, fine aggregate, and mineral powder was 67%, 28%, and 5%, respectively. The aggregate gradation is described in Table 2;
- Mixture. AC13-graded asphalt mixture gradation was adopted for fabricating the mixture samples. The optimum asphalt content of mixture is 4.8%. Aggregates coated with asphalt mortar for ATR-FTIR measurement were randomly obtained from the same batch of asphalt mixture fabricated at the same time.

Table 1. Physical properties.

Sample	Penetration (0.1 mm)	Ductility (cm)	Softening Point (°C)
JL-70 base asphalt	64.4	>100	46.7

Table 2. AC-13 aggregate gradation.

Sieve Size (mm)	16	13.2	9.5	4.75	2.36	1.18	0.6	0.3	0.15	0.075
Passing (%)	100	95.9	74.9	46.3	27.7	17.4	12.1	9.3	7.6	5.7

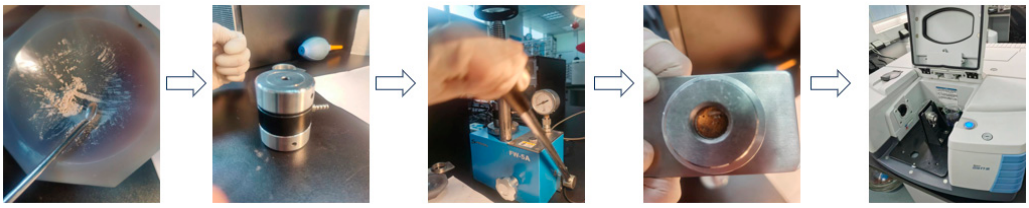
3.2. FTIR Measurement of Aggregates and Mineral Powders Using Transmission Mode

3.2.1. Samples Preparation

To investigate the crucial functional groups of aggregates and mineral powder, the FTIR spectra of fine aggregate, coarse aggregate, and mineral powder were collected by the transmission mode via a KBr squashing method [38]. After the samples were dried and ground to a particle size of less than about 2.5 um, the thin KBr plate was pressed by a manual tablet press at 8–10 Mpa for 30 s.

3.2.2. Spectra Collection

The prepared KBr plate was measured against a blank KBr plate by a Nicolet iS50 FTIR spectrometer in the range of 4000~500 cm^{-1} with a resolution of 4 cm^{-1} and a total of 32 scans. The steps shown in Figure 1 were repeated until the FTIR spectra of fine aggregates, coarse aggregates, and mineral powders were collected.



(**a**) (**b**) (**c**) (**d**) (**e**)

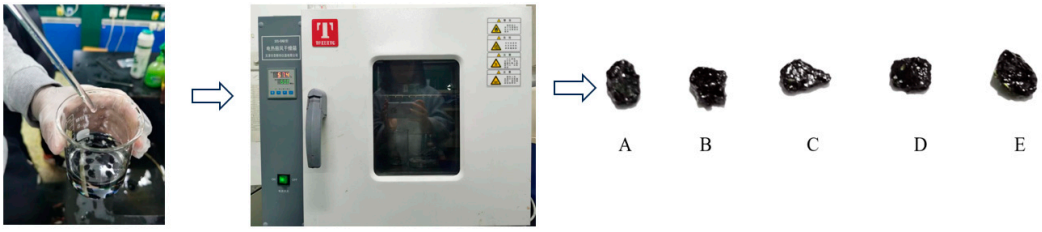
Figure 1. Processing: (**a**) grinding 0.5 mg sample and 150 mg KBr to particle size less than 2.5 um; (**b**) fabricating the thin KBr plate; (**c**) pressing the thin KBr plate by a manual tablet press at 10 Mpa for 30 s; (**d**) checking whether the thin KBr plate is qualified; (**e**) FTIR measurement in transmission mode.

3.3. ATR-FTIR Measurement of Asphalt and Aggregates Coated with Asphalt Mortar

3.3.1. Samples Preparation

To compare the difference between the FTIR spectra of aggregates coated with asphalt mortar and asphalt, the spectra of JL-70 neat asphalt were also recorded referring to the common ATR-FTIR testing process of asphalt [35].

To remove the dust, aggregates coated with asphalt mortar were cleaned with pure water. Then, the coated aggregates should be dried before measurement to prevent the influence of water. The drying process was conducted by putting samples into an electric thermostatic drying oven at 57.4 °C for 30 min. After cleaning and drying, 5 samples from the same batch of asphalt mixture fabricated at the same time with relatively flat surfaces were measured as shown in Figure 2, which were named MixA, MixB, MixC, MixD, and MixE.



(**a**) (**b**) (**c**)

Figure 2. Samples preparation: (**a**) cleaning; (**b**) drying; (**c**) aggregates coated with asphalt mortar.

3.3.2. Spectra Collection

The prepared samples of asphalt and aggregates coated with asphalt mortar were also measured against a blank background by a Nicolet iS50 FTIR spectrometer with a diamond crystal as shown in Figure 3. The FTIR spectra were obtained in the range of 4000~500 cm^{-1} with a resolution of 4 cm^{-1} and a total of 32 scans. During the measurement of coated aggregates with ATR-FTIR spectroscopy, it is worth noting that different measurement points from flat micro-regions of coated aggregates were used to ensure the sample was placed in tight contact with the surface of the ATR crystal.

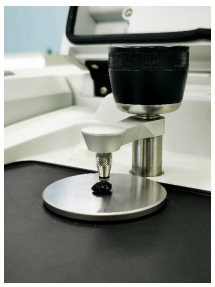


Figure 3. ATR-FTIR measurement of aggregate coated with asphalt mortar.

4. Results and Discussion

4.1. Analysis of Characteristic Peaks of Aggregates and Mineral Powders

Before spectral analysis, transmittance was converted into absorbance by OMNIC data analysis software 9.2. The original absorbance spectra of coarse aggregates, fine aggregates, and mineral powders were drawn by Matlab software R2019b after peak searching and peak marking, as shown in Figure 4. Obviously, characteristic peaks of aggregates and mineral powder are in the range of 2000~500 cm^{-1}. Moreover, coarse limestone aggregates display eight absorption peaks, 2513 cm^{-1}, 1797 cm^{-1}, 1462 cm^{-1}, 1443 cm^{-1}, 1428 cm^{-1}, 1414 cm^{-1}, 876 cm^{-1}, and 712 cm^{-1}, which are typical peaks of carbonate compounds according to previous research on limestone [38,39]. And granite

powders display four absorption peaks, Si-O stretching vibration at 1009 cm^{-1}, Si-Si stretching vibration at 776 cm^{-1}, Si-Al stretching vibration at 727 cm^{-1}, O-Si-O stretching vibration at 647 cm^{-1}, according to previous research on granite [38–40]. Granite powders and granite fine aggregates display similar absorption peaks with a maximum absorption peak at 1000 cm^{-1}.

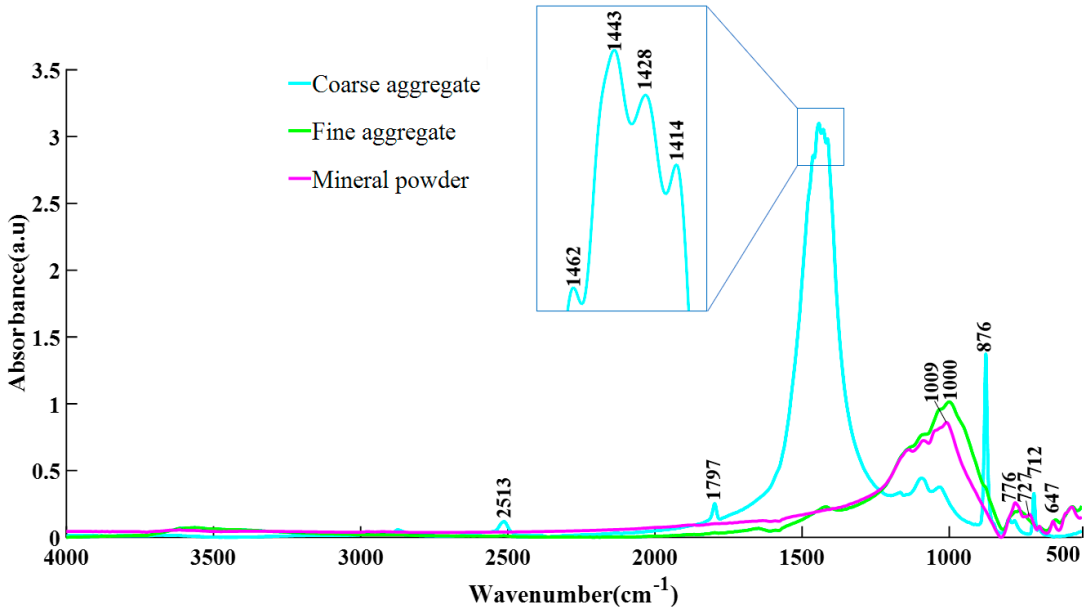


Figure 4. Original spectra of aggregates and mineral powders.

4.2. Study on the Heterogeneity of Aggregates Coated with Asphalt Mortar Based on ATR-FTIR Spectroscopy

4.2.1. Spectral Comparative Analysis of Asphalt and Aggregates Coated with Asphalt Mortar

To better compare the spectra of asphalt and aggregates coated with asphalt mortar, as shown in Figure 5, the intensity ratio of the spectra needs to be adjusted to the same level. Therefore, spectral normalization pretreatment is required before spectral analysis. The spectrum preprocessing was carried out by Matlab software R2019b using the normalized "mapmaxmin" function. The normalized spectra of asphalt and aggregates coated with asphalt mortar after peak searching and peak marking are shown in Figure 6.

In Figure 6, aggregates coated with asphalt mortar and asphalt exhibit similar spectral responses in the range of 3000~2500 cm^{-1} but different spectral responses in the range of 2000~500 cm^{-1} with obvious overlapping peaks at 1419 cm^{-1} and 1018 cm^{-1}. The ATR-FTIR spectra of aggregates coated with asphalt mortar showed that the samples exhibited absorption peaks at around 2952 cm^{-1}, 2920 cm^{-1}, 2851 cm^{-1}, 1797 cm^{-1}, 1723 cm^{-1}, 1453 cm^{-1}, 1419 cm^{-1}, 1377 cm^{-1}, 1268 cm^{-1}, 1018 cm^{-1}, 873 cm^{-1}, 780 cm^{-1}, 729 cm^{-1}, and 712 cm^{-1}. According to the analysis in Section 4.1, the absorption peaks at 1797 cm^{-1}, 1419 cm^{-1}, 873 cm^{-1}, and 712 cm^{-1} could be attributed to typical peaks of coarse limestone aggregate, while the absorption peaks at 1018 cm^{-1} and 780 cm^{-1} could be attributed to typical peaks of granite powder. And the absorption peaks at 2952 cm^{-1}, 2920 cm^{-1}, 2851 cm^{-1}, 1453 cm^{-1}, 1377 cm^{-1}, 729 cm^{-1} could be attributed to typical peaks of asphalt. Furthermore, absorption peaks at 1723 cm^{-1} (stretching vibration of C=O) and 1268 cm^{-1} (ether) could be attributed to residual asphalt cleaner, which could be dismissed.

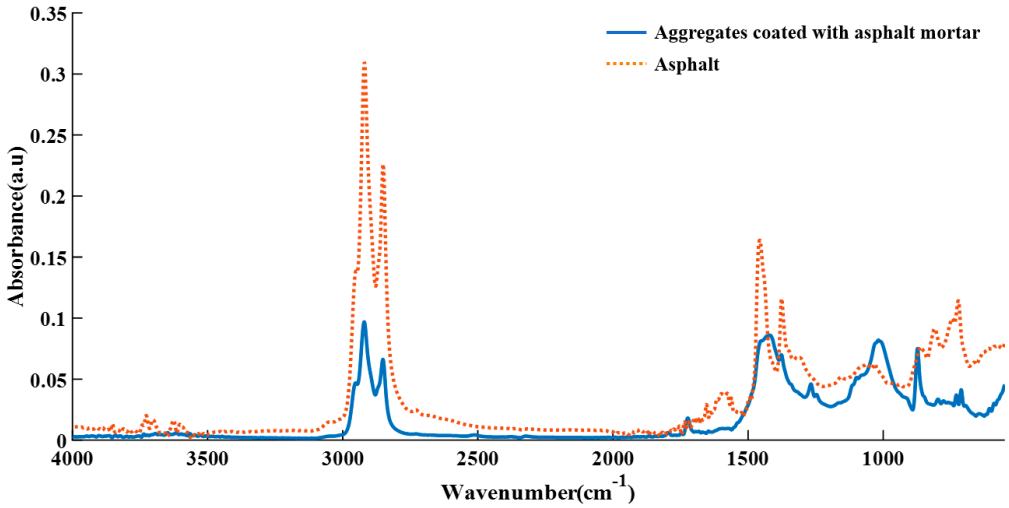


Figure 5. Original spectra of asphalt and aggregates coated with asphalt mortar.

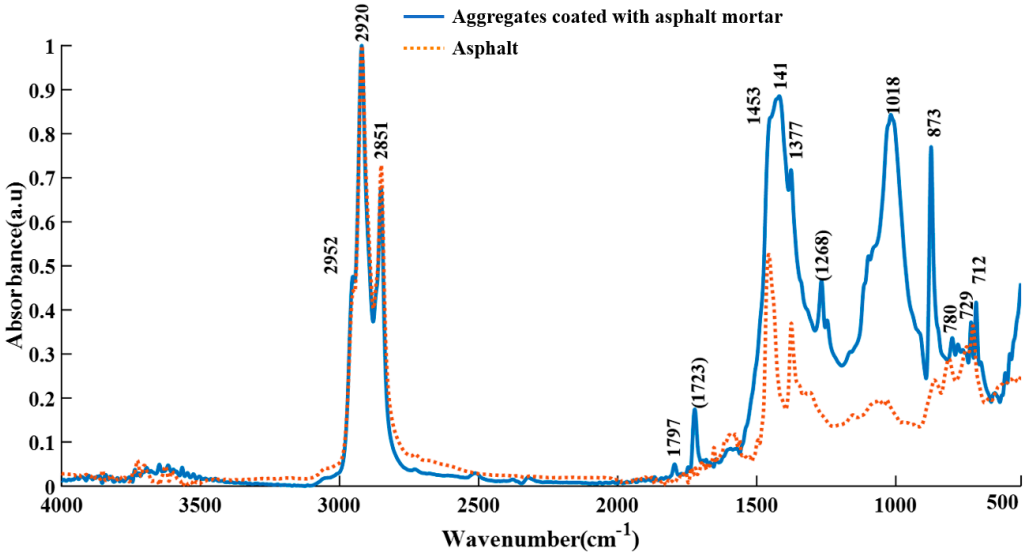


Figure 6. Normalized spectra of asphalt and aggregates coated with asphalt mortar.

The vibration frequency is called group frequency and can be used to identify the presence of a specific group. By analyzing the position of the absorption peak in the infrared spectrum, we could know the types of groups that might be contained in the sample molecule. To further investigate the differences between asphalt and aggregates coated with asphalt mortar, spectral comparative analysis was conducted by analyzing the distribution of the characteristic peaks. The descriptions of peak positions are listed in Table 3, according to previous research on graded asphalt and aggregates [13,24,35,38–40]. There are a total of 12 major characteristic peaks of the aggregates coated with asphalt mortar, with five concealed characteristic peaks of the asphalt, including the significant absorption peak at 1030 cm^{-1}.

From the spectral analysis above, the characteristic absorption peaks of the aggregates coated with asphalt mortar are the superposition of the respective absorption peaks of the aggregates, mineral powders, and asphalt binders, which can all contribute to the spectrum of the coated aggregates.

Table 3. Peak positions of asphalt and aggregates coated with asphalt mortar.

Peak Position (cm^{-1})		Functional Groups/Composition	Details
Aggregates Coated with Asphalt Mortar	Asphalt		
712	-	Carbonate compounds	In-plane bending vibration
-	721	$-CH_2$	Cooperative vibration;
729	-	Si-Al	stretching vibration
780	-	Si-Si	Stretching
873	-	Carbonate compounds	Out-of-plane bending vibration
1018	-	Si-O	Stretching
-	1030	S=O	Stretching
-	1153	C-O	Ester; stretching
-	1307	C-O	Carbonyl acid; stretching
1377	1375	CH_3	Aliphatic; plan deformation
1419	-	carbonate compounds	Anti-symmetric stretching vibration
1453	1456	CH_3&CH_2	Aliphatic; deformation
-	1598	C=C	Conjugated ring vibration
1797	-	carbonate compounds	Weak symmetrical stretching vibration; in-plane bending vibration
2851	2850	=C-H	Aldehyde; stretching
2920	2920	C-H	Aliphatic hydrogen; CH_3 and CH_2; asymmetric stretching
2952	2951	C-H	Aliphatic hydrogen; CH_3

4.2.2. Study on the Effect of the Heterogeneity of Aggregates Coated with Asphalt Mortar on Its Spectrum

To further study the effect of the heterogeneity of aggregates coated with asphalt mortar on its spectrum, 15 spectra of the five samples were measured by ATR-FTIR technology as shown in Figure 7, which is drawn by Matlab software. It can be seen from Figure 7 that the spectral shapes and peak positions of the five samples obtained from the same batch of asphalt mixture fabricated at the same time across the entire mid-infrared region are very similar. However, the peak intensities from the five samples are significantly different, among which the difference in Mix D is the smallest and that in Mix B is the largest.

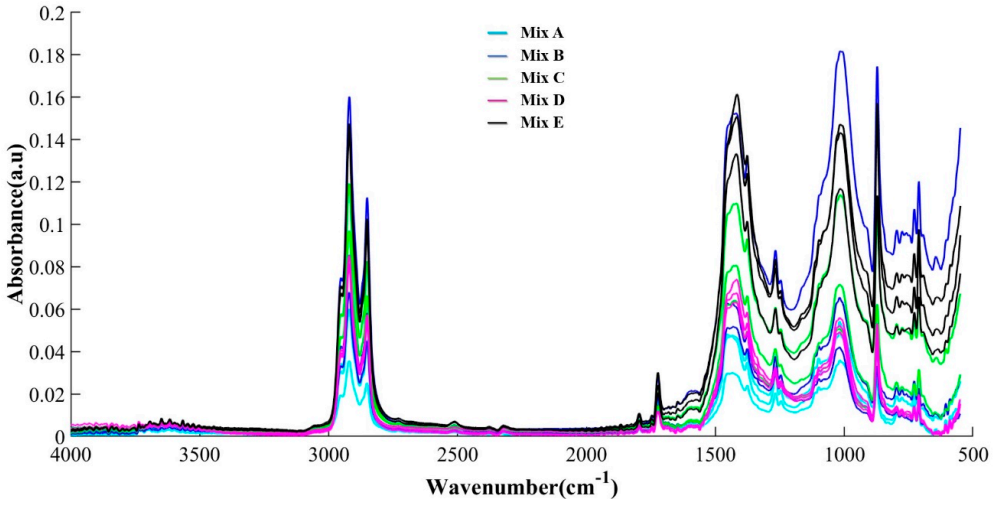


Figure 7. Original spectra of the five samples with ATR-FTIR.

Therefore, it can be concluded that samples at different measurement points had the same composition but different amounts and that the spectra after the superposition

of the absorption peaks are affected by minor variations in the components due to the heterogeneity of aggregates coated with asphalt mortar.

To improve the data quality of the spectra and the accuracy of the subsequent analysis, spectrum preprocessing was carried out in the Matlab software by using the normalized "normalize(data, 'norm')" function, as shown in Figure 8.

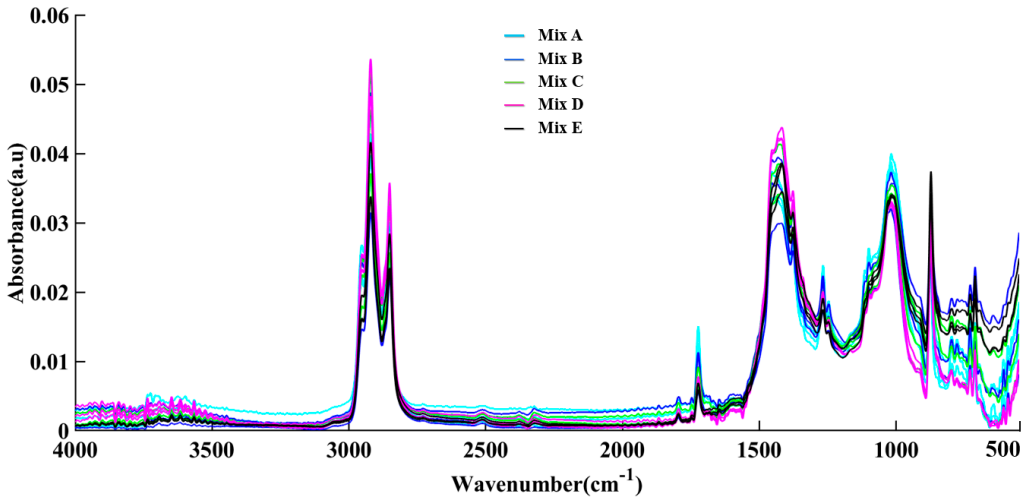


Figure 8. Normalized spectra of the aggregates coated with asphalt mortar with ATR-FTIR.

4.3. Statistical Analysis on the Spectral Reproducibility of the Aggregates Coated with Asphalt Mortar

Reproducibility is the closeness of the agreement between the measuring results of the same measurand [41], which is widely known as an important factor for the accuracy of measurement. Mean value (MV), standard deviation (SD), and coefficient of variation (CV) (CV = 100 × SD/MV, %) are usually calculated to investigate the reproducibility [42,43], as shown in Figure 9, Tables 4 and 5.

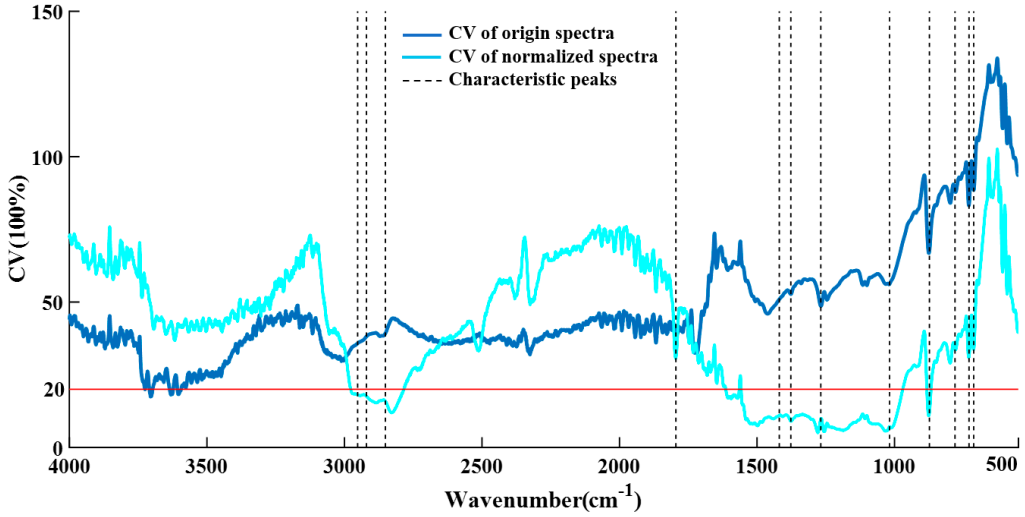


Figure 9. CV values at different wavenumbers.

In Figure 9, it can be seen that the CV values of the original spectra across the entire fingerprint region are higher, almost above 50%, than those of the other region. Though the CV values of original spectra in the range of 3400~3700 cm^{-1} are lower than 20%, this is meaningless because characteristic absorption peaks do not exist in this region. Furthermore, the CV values at the 12 characteristic peaks of original spectra in Table 4 all show a high level higher than 30%, and the CV value at 712 cm^{-1} is as high as 88.5%.

Therefore, it can be concluded that the original spectral reproducibility of aggregates coated with asphalt mortar with ATR-FTIR spectroscopy is greatly affected by the heterogeneity of the coated aggregates.

Table 4. MV, SD, and CV values at 12 characteristic peaks of original spectra.

Wavenumber (cm^{-1})	712	729	780	873	1018	1377	1419	1453	1797	2851	2920	2952
MV	0.0410	0.0369	0.0316	0.0749	0.0819	0.0698	0.0859	0.0811	0.0060	0.0661	0.0969	0.0468
SD	0.0364	0.0308	0.0278	0.0507	0.0460	0.0369	0.0437	0.0379	0.0025	0.0260	0.0370	0.0168
CV (100%)	88.5	83.2	88.1	67.6	56.1	52.7	50.9	46.7	40.9	39.2	38.2	35.8

Table 5. MV, SD, and CV values at 12 characteristic peaks of normalized spectra.

Wavenumber (cm^{-1})	712	729	780	873	1018	1377	1419	1453	1797	2851	2920	2952
MV	0.0149	0.0141	0.0116	0.0302	0.0350	0.0300	0.0372	0.0358	0.0028	0.0302	0.0444	0.0217
SD	0.0050	0.0044	0.0039	0.0036	0.0025	0.0027	0.0041	0.0037	0.0009	0.0048	0.0078	0.0039
CV (100%)	33.7	31.1	33.7	12.0	7.2	9.0	11.0	10.4	31.5	15.9	17.6	18.1

However, the CV values at the 12 characteristic peaks of normalized spectra in Table 5 all show a relatively low level, mostly lower than 20%. And compared to the original spectra in Figure 9, the CV values of normalized spectra in the range of 1500~1000 cm^{-1} decrease to less than 20%, indicating that norm normalization could improve data quality. Hence, the absorption peaks at 2952 cm^{-1}, 2920 cm^{-1}, 2851 cm^{-1}, 1453 cm^{-1}, 1419 cm^{-1}, 1377 cm^{-1}, 1018 cm^{-1}, and 873 cm^{-1} in Table 3 might be considered useful peaks due to their low CV values.

5. Conclusions

In this paper, the impact of the heterogeneity of aggregates coated with asphalt mortar on their spectra based on ATR-FTIR spectroscopy was mainly carried out. The following conclusions can be drawn based on the research results of this paper:

- As the characteristic absorption peaks of the aggregates coated with asphalt mortar are the superposition of the respective absorption peaks of the aggregates, mineral powders, and asphalt binders, these components can all contribute to the spectrum of the coated aggregates;
- The spectral shapes and peak positions of aggregates coated with asphalt mortar obtained from the same batch of asphalt mixture fabricated at the same time are very similar across the entire mid-infrared region, and the peak intensities are significantly different, showing that intensities are affected by minor variations in the components due to the heterogeneity of the coated aggregates;
- CV values are high across the entire mid-infrared region, indicating that the original spectral reproducibility of aggregates coated with asphalt mortar with ATR-FTIR spectroscopy is greatly affected by the heterogeneity of the coated aggregates;
- Spectra pretreatment of norm normalization could reduce the CV values for better data quality.

As spectral reproducibility is greatly affected by the heterogeneity of the aggregates coated with asphalt mortar, future research will focus on spectra preprocessing, including abnormal spectrum rejection, noise reduction, signal enhancement, and feature selection, to improve the accuracy of qualitative and quantitative analysis.

Author Contributions: Conceptualization, J.Y. and R.J.; methodology, J.Y., X.Z. (Xinglin Zhou) and M.R.; software, J.Y.; validation, J.Y., X.Z. (Xinxing Zhou) and L.L.; formal analysis, J.Y. and P.Z.; investigation, J.Y.; resources, M.R. and X.Z. (Xinxing Zhou); data curation, J.Y.; writing—original

draft preparation, J.Y., P.Z. and L.L.; writing—review and editing, J.Y., M.R. and X.Z. (Xinxing Zhou); visualization, L.L. and P.Z.; supervision, P.Z. and R.J.; project administration, R.J., M.R. and X.Z. (Xinglin Zhou); funding acquisition, X.Z. (Xinglin Zhou) and M.R. All authors have read and agreed to the published version of the manuscript.

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

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