



# TECHSUMMARY September 2022

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## Field Implementation of Handheld FTIR Spectrometer for Polymer Content Determination and for Quality Control of RAP Mixtures

### INTRODUCTION

Fourier Transform Infrared Spectroscopy (FT-IRS) has attained its acknowledgement in chemical analysis of asphalt binder considering its simplicity in sampling process and data interpretation proficiency [1]. Recent studies show that FT-IR spectroscopy has been effectively applied in recognizing an array of modifiers used in asphalt binders such as SBS, polyolefin, crumb rubber, WMA (warm mix asphalt) additives, and polyphosphoric acid (PPA) [2]. Application of FT-IRS in quantification of aging in asphalt binder is also well studied. The principle of FT-IR spectroscopy is to detect the molecular structures of a sample based on their vibrational and rotational frequency at a specific wavenumber in the range of 600  $\text{cm}^{-1}$  to 4000  $\text{cm}^{-1}$  when an infrared beam passes through the sample and is absorbed by it. This absorbance is displayed in the form of an absorbance intensity versus wavenumber. According to Beer's law, this absorbance intensity is proportional to the concentration of the molecule. From this relationship, presence of a functional group in any material can be identified, and the quantity can be determined. So, advantages of the application of FT-IRS in asphalt can be taken by quantifying the polymers contents in the binder, and aging of asphalt binder as well as mixes. The research is divided into six subsections for better representation of the findings: (1) quantification of SBS content, (2) degradation of SBS due to aging, (3) selection of asphalt binder aging parameter, (4) effect of laboratory binder and mix aging, (5) RAP content determination, and (6) identification and quantification of rejuvenators.



Figure 1. Sample collection technique for FT-IR spectroscopy

### OBJECTIVE

The main objectives of this study were to: (1) develop a universal curve that can be used to successfully predict SBS content (%) in a modified-asphalt binder in the field and (2) implement the handheld FT-IR spectrometer in the field as a tool for quality control of plant mix containing RAP.

### SCOPE

PG 52-34, PG 58-28, and PG 64-22 binders were collected from LA, MS, TX, and NC. Polymer-modified binders PG 64-28, PG 70-22, PG 70-28, and PG 76-22 were collected from LA, MS, TX, AR, and NV. Both asphalt binders and mixtures were used to achieve the goals of this study. The FT-IR spectrometer was used for the data collection of different type of original binders; modified binders (with polymer, rejuvenator, and RAP); and aged binders. Aging of binders was performed by RTFO, PAV, UV, and forced draft oven. The DSR test was performed to perceive the rheological properties. DCM solvent was used to extract the binder from the mixture and RAP. Two different plants were visited to implement the SBS content determination technique. RAP and mixture were collected from 10 different sources and five plants were visited twice to implement the RAP content determination technique.

### METHODOLOGY

A 4300 handheld FT-IR spectrometer was used to obtain spectra considering its flexibility, robustness, carrying capability for field data collection, and easy sampling procedure. The diamond ATR sensor was considered since collected spectra remained unaffected by the sample amount placed on the sensor [12].

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Each spectrum collected by the FT-IR spectrometer was converted to the absorbance spectrum by the default Microlab PC software provided with the instrument. A total of 24 scans for each sample was conducted in 650-4000  $\text{cm}^{-1}$  region and reported in the form of 4  $\text{cm}^{-1}$  resolution. Before each scan, background spectra were collected to check if the sensor was properly clean. Figure 1 shows the sample collection technique and Figure 2 shows the instrumentation with a 4300 handheld FT-IR spectrometer [62].

## CONCLUSIONS

The key findings from this study are stated below:

1. Prediction result of SBS (%) in field measurement justified the specified SBS (%) range stated by the manufacturer. Also, the percentage error of the predicted SBS concentration (%) was 5% when the actual amount of SBS (%) was known. The total process for sample collection and data processing for SBS (%) prediction in each field demonstration required 15 minutes by the handheld FT-IRS.
2. After RTFO and PAV aging, SBS content (%) decreased, which indicated SBS polymer degraded after aging. After RTFO aging, 7.5-14.5% SBS content (%) was decreased, and after PAV aging, 16-30% SBS content (%) was decreased.
3. Both the carbonyl and sulfoxide functional groups in asphalt binder are affected by the aging process. The change in concentration of sulfoxide with the extent of asphalt binder or mixture aging was not consistent. Moreover, in case of mixture aging, the index values were influenced by the fines present in the mixture. Therefore, sulfoxide index should not be considered as a reliable indicator to quantify the aging rate of an asphalt binder or mixture.
4. Carbonyl index calculated by peak height ratio at wavenumber 1696  $\text{cm}^{-1}$  to 2920  $\text{cm}^{-1}$  provide accurate result to quantify laboratory binder and mix aging.
5. For quantification of RAP content in the mix, the area ratio at 1696  $\text{cm}^{-1}$  to 1456  $\text{cm}^{-1}$  yields more consistent results than the other indices.
6. Carbonyl index can successfully quantify binder and mix aging. For all types of laboratory aging methods, carbonyl index gradually increases with duration of aging. Rate of increase of ICO is not linear; it rather slows down while the aging progresses.
7. FT-IRS can precisely detect the presence of aged binder in the mix within a very short time and using a small amount of mix. So, the handheld FT-IRS has the potential to be used as a quality control tool in the field.
8. The carbonyl index of a mix increases linearly with increase in the RAP content. This linear relationship can be utilized to determine the RAP content in the mix.
9. FT-IRS can successfully determine the RAP content in the mix within  $\pm 5\%$  range of the design RAP content.
10. From the qualitative analysis of the FT-IR spectra, it was apparent that bio rejuvenator added two functional groups (at 1744  $\text{cm}^{-1}$  and at 1162  $\text{cm}^{-1}$ ), which can be used to identify bio rejuvenator in asphalt samples.
11. Unlike bio rejuvenator, aromatic rejuvenator did not add any significant functional group in the asphalt binder.

## RECOMMENDATIONS

Ten plant RAP mixes were utilized in this study that justifies the viability of using a handheld FT-IR spectrometer for quality control of SBS-modified asphalt binder and RAP mixes. A handheld FT-IR spectrometer can determine the percentage of SBS/SB in asphalt binder and the percentage of RAP content in RAP mixes in the field precisely, accurately, and quickly. The outcome of this study is immediately implementable. The authors would like to recommend the use of FT-IR spectrometer following the procedure used in this study.

The FT-IRS method has the potential to be used for pavement maintenance purposes. Thresholds can be developed using aging indices that will trigger maintenance actions. Also, investigations need to be performed on the potential use of the FT-IR spectrometer for identifying cracking susceptible, extremely aged surfaces. Forensic analyses can be performed using the FT-IR method to find the possible causes of premature failure. In forensic analyses, the FT-IR method can detect segregation of polymer, degradation of polymer, excessive usage of RAP, extremely oxidized RAP usage, presence of rejuvenators, etc.

Based on the findings of this study, the authors recommend immediate implementation of the FT-IR spectrometer as well as studies for viability of extensive usage of a handheld FT-IR spectrometer in various potential other applications as mentioned before.



Figure 2. Placing sample on diamond ATR sensor of FT-IR