Advancing Analytical Techniques For REOB Detection In Asphalt Binders

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Introduction

Asphalt is the highest boiling, non-distillable component of crude oil, created during petroleum refining. (Baumgardner, et al., 2023). It is a critical material used in paving roads and is produced on a large scale to meet the demands of infrastructure development. A typical 1-mile, four-lane highway with a 4-inch lift can weigh about 6,300 tons, of which approximately 300 tons are asphalt binder. (Arnold, 2023). This large-scale production has made quality control a critical aspect of asphalt manufacturing, as even minor changes in composition can affect long-term performance. One challenge is the use of Re-refined Engine Oil Bottoms (REOB) as an additive. REOB, also known as Vacuum Tower Asphalt Extender (VTAE), are byproducts of waste engine oil recycling that have been used as fluxing agents in asphalt binders to improve low-temperature performance. REOB has also been linked to increased pavement brittleness and premature cracking, particularly in aging asphalt mixtures. Detecting and quantifying REOB is challenging as its chemical similarity to asphalt makes it difficult to regulate and prevent unwanted incorporation (Baumgardner et al., 2023). Wielinski et al. (2015) attempted to use Thermogravimetric Analysis (TGA) to predict REOB content by testing samples in a nitrogen environment, followed by the introduction of air to observe combustion behavior. The study analyzed an asphalt binder both in its unmodified form and with 9% REOB, but did not attempt to measure the binder with other percentages of REOB. (Wielinski et al., 2015). Also of importance, Re-refined Engine Oil Bottoms (REOB) have been found to contain residual metal contaminants, including copper and iron, which originate from normal engine wear during the original use of the oil. These metals, along with other non-distillable components, remain in REOB after the re-refining process. (Baumgardner et al., 2023).

Fig. 1: Sample of Asphalt Binder

Fig. 2: REOB Sampling From Pale

Unlike the study by Wielinski et al. (2015), which utilized a nitrogen environment followed by the introduction of air, this study employs Thermogravimetric Analysis (TGA) exclusively in an air environment to prevent the combustion of these metal residues. This method ensures that any residual metals from the REOB remain during the TGA cycle, allowing for a more direct correlation between residue mass and REOB concentration in the binder. Additionally, both REOB and SHRP binders exhibit distinct TGA decomposition curves, meaning that blending them at varying concentrations should produce predictable thermal degradation patterns. Figure 4 below illustrates the TGA curves of only REOB and a SHRP binder, highlighting their distinct decomposition behaviors. It is hypothesized that by analyzing these differences, a quantifiable relationship of REOB content in modified asphalt binders can be established.

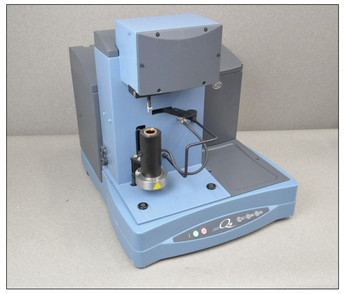

Fig. 3: Q50 Thermogravimetric analyzer

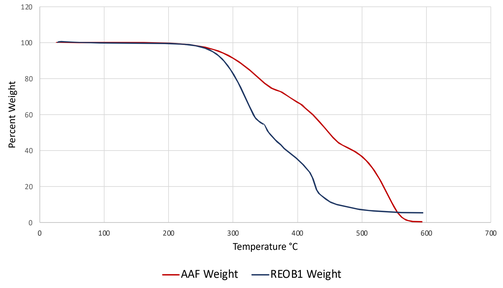

Fig. 4: TGA Comparison of AAF Binder and REOB

Materials and Methods

Table 1: Material Details

Binders	AAF, AAC, AAK
REOBs	REOB 1, REOB 2
REOB Concentrations	0%, 2%, 5%, 8%, 12%

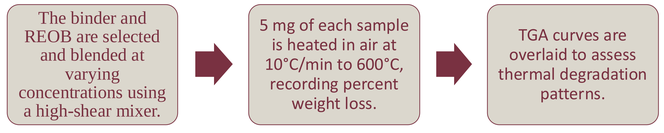

Fig. 5: Procedure Flowchart

To confirm that the two REOB types were chemically distinct, their levels of absorption in the fingerprint region were verified using Fourier Transform Infrared Spectroscopy (FTIR). This confirmed that both REOBs possessed varying levels residue and are worth testing individually to account for the chemical variance of REOB in the field.

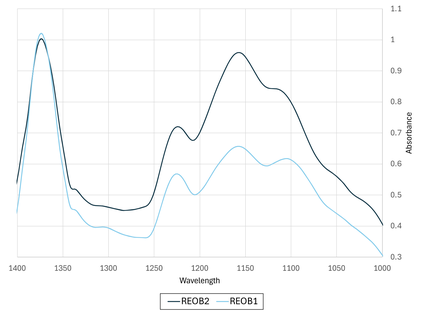

Fig. 6: FTIR Comparison of REOBs

Objective

This research focuses on Thermogravimetric Analysis (TGA) as a method for detecting and quantifying REOB in asphalt binders. By analyzing weight loss patterns under controlled heating conditions, this study aims to expand knowledge on REOB detection and quantification, contributing to more effective assessment techniques for asphalt quality control.

Current Results

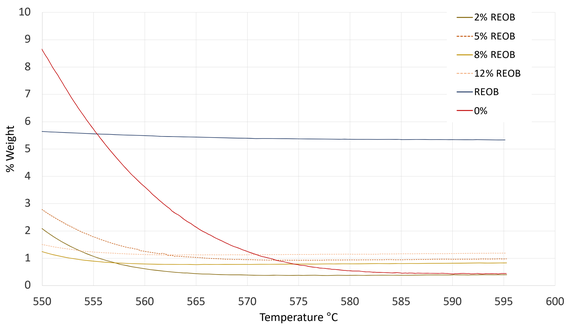

Fig. 7: Residual Weights Comparison

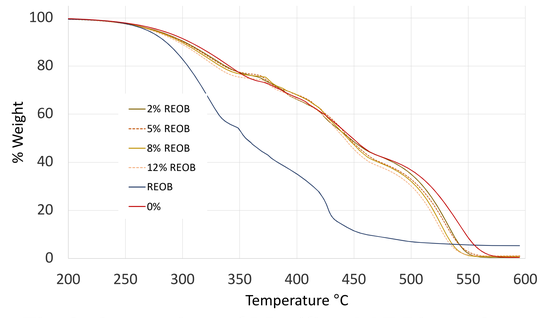

Fig. 8: Comparison of Modified AAF Binder Samples Degradation Behavior

Expected Results

Further testing with AAK and AAC binders is expected to show similar thermal decomposition trends, where higher REOB concentrations resulting in greater residual mass due to metal contaminants. However, differences in binder composition may cause slight variations in decomposition behavior. Pure REOB should retain the highest residue, followed by binders with increasing REOB content.

Future Work

Further testing is required to evaluate the second REOB type and additional binder formulations to determine whether the observed decomposition trends remain consistent. Additionally, TGA trials conducted solely in a nitrogen environment for AAF and AAC binders did not yield clear correlations, but similar tests will be performed for the remaining samples and compared with air trials to assess potential patterns. Dynamic Thermogravimetric Analysis (D-TGA), the method employed by Wielinski et al. (2015), will also be explored as a potential tool for improved REOB quantification, with a focus on optimizing the temperature at which air is introduced in the TGA cycle. Beyond these tests, future research could examine how aging processes, such as Rolling Thin Film Oven Test (RTFOT) and Pressure Aging Vessel (PAV) conditioning, influence thermal decomposition and residual mass trends in REOB-modified binders. Expanding the study to include additional REOB sources and binder types will further validate whether the identified patterns are broadly applicable across asphalt formulations.

References

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- Baumgardner, G., Hand, A. J., Hajj, E. Y., & Aschenbrener, T. B. (2023). *Responsible use of Re-Refined Engine Oil Bottoms (REOB) in asphalt binders.* Washington, D.C.: U.S. Department of Transportation, Federal Highway Administration.
- Wielinski, J., Kriech, A., Huber, G., Horton, A., & Osborn, L. (2015). *Analysis of Vacuum Tower Asphalt Extender and Effect on Bitumen and Asphalt Properties.* Road Materials and Pavement Design, 16, 90-110.

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