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The Performance of Aged Asphalt Materials Rejuvenated with Waste Engine Oil

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Abstract: The ability to recycle large amounts of asphalt pavement hinges on the capability of restoring the properties of the aged asphalt binder contained within the old pavement to that of virgin binder. Common practice in asphalt pavement recycling is to blend reclaimed asphalt pavements (RAP) with a recycling agent to chemically restore the aged asphalt binder. Waste engine oil from automobiles has been shown to improve asphalt binder when applied in small quantities, with the added advantage of being a waste product itself. Using waste engine oil as a chemical additive to restore the properties of RAP uses one waste material to increase the recyclability of another, which is environmentally and socially desirable.

In this study, a PG 58-28 neat, virgin binder was blended with reclaimed asphalt binder (RAB) and waste engine oil. The blends were then tested to study the interactions between RAB and waste engine oil. Using Fourier-Transform Infrared Spectroscopy (FT-IR), the differences in the samples were compared using the structural indices associated with asphalt binder aging. This testing revealed a decrease in the two aging indices of the blended asphalt binder, indicating that waste engine oil has the ability to chemically restore aged asphalt binder.

Asphalt mixture testing was then performed with mixtures of virgin asphalt, virgin binder, RAP and waste engine oil, in quantities similar to the binder testing, to see if the rejuvenation shown in FT-IR led to an improvement in the performance of the pavement specimens. After specimens were created, testing for freeze thaw durability, and rutting susceptibility was conducted. The results of the mixture testing failed to show an improvement of the freeze thaw durability or rutting susceptibility of specimens created with RAP and waste engine oil when compared to mixtures containing only new materials.

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***Key words**: Asphalt binder; Binder rejuvenator, Fourier-Transform Infrared Spectroscopy (FT-IR); Mixture testing, Waste engine oil.*

Introduction

Reusing asphalt pavements decreases the consumption of virgin materials. However, recycling asphalt pavement is not as simple as grinding up the existing road material and using it to repave. Several barriers exist to simply repaving existing asphalt roads without modification; principally the fact that the asphalt binder contained in the road undergoes aging while in service and cannot be used as a direct substitute for virgin binder. This aging makes the binder stiffer and more brittle than virgin binder, which makes roads paved with unmodified reclaimed asphalt pavement (RAP) more susceptible to cracking. One way to manage the problem of binder ageing is to add a chemical recycling agent into new asphalt pavements containing RAP to restore the properties of the binder being recycled.

Recycling Agents

There exists a large body of literature on the use and consequences of using asphalt recycling agents. The purpose of recycling agents is to restore asphalt consistency and chemistry. An ideal recycling agent should improve the asphalt binder contained in the RAP and return the mixture to a suitable paving material. In general, there are two types of chemical additives that can be added to pavements containing RAP: rejuvenating agents and softening agents. The primary difference between a softening agent and a rejuvenating agent is that a rejuvenating agent will restore the chemical structure of aged asphalt, while a softening agent reduces the overall viscosity of the binder [1]. In either case, recycling agents are usually made out of a petroleum product comprised of either highly polar or aromatic oils, and must diffuse the asphaltenes contained inside the asphalt binder [2, 3].

Previous literature has reported that using more than ten percent recycling agent, by weight of binder, proves detrimental to the asphalt binder [4, 5]. Despite having potentially negative effects on the asphalt binder, recycling agents are recommended for mixtures containing either high amounts of RAP or heavily oxidized RAP. Reclaimed asphalt binder (RAB), which has been extracted and recovered from RAP, containing recycling agents have been shown to improve asphalt binder properties and performance measures, such as viscosity and fatigue resistance, when compared to RAB without recycling agents [2, 6]. There are varied opinions on the feasibility of lubricating oil as an additive in asphalt binder, as most research has focused on trying to improve the low temperature properties of unaged asphalt [4, 7, 8]. Although it has been shown that the addition of engine oil can improve the low temperature properties of asphalt, little research has been done on waste engine oil as a recycling agent for asphalt pavements containing RAP [9, 10].

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With regards to Superpave performance classifications, it was reported that reclaimed asphalts that are sufficiently blended with aromatic modifiers will usually meet the specification for virgin binder [11]. However, Superpave specifications target the engineering properties of the samples and do not focus on the chemical composition of asphalt binder. Even though the engineering properties of the asphalt binder are what will control the performance of the pavement, it is important to acknowledge the need for understanding the interactions between asphalt binder and the additives being used.

Asphalt Binder Chemistry

At the molecular level, asphalt binder is comprised of asphaltenes and maltenes. The asphaltenes are large polar compounds which are responsible for the binders' viscosity and adhesion. Maltenes are a colloidal combination of oils and resins in which the asphaltenes are dispersed [5, 12]. The role of the maltenes is to provide stability to the asphaltenes; additives in the binder typically modify the maltene structure [5]. Using advanced chemical analysis, which is beyond the scope of this paper, the maltenes can be further broken down into saturates, and polar and non-polar aromatics [3, 13].

During mixing and construction, there is a loss of aromatics in the asphalt binder due to volatilization. Furthermore, as the asphalt ages in service, oxidation will occur on the exposed asphalt binder [14]. These two processes cause the conversion of maltenes into asphaltenes [15], leading to an overall loss of maltenes as a pavement ages. The loss of maltenes translates to a stiffer pavement that is more brittle due to a lack of cohesion inside the binder. In order to rejuvenate aged binder, the chosen rejuvenator must provide maltenes to the binder to restore the binders' stability [4]. One advantage to using RAP is that recycled asphalt binder that has been rejuvenated tends to age more slowly than a purely virgin pavement [11].

Experimental / Materials and Methods

Materials

Several types of binder and rejuvenator were chosen for FT-IR testing. The base binder was a PG 58-28 binder from Hancock, Michigan. The engine oil was provided by the Michigan Tech motor pool. The RAP was sourced from a stockpile in Hancock, Michigan. The reclaimed asphalt binder (RAB) was extracted and recovered according to ASTM D 2172 and ASTM D 1856 [16, 17]. For the blends of virgin asphalt binder and RAB, the chosen concentrations were 75% and 25% respectively. The virgin asphalt binder was combined with the RAB by continuously stirring with a glass rod on a hotplate until homogeneity was achieved. For the samples containing waste engine oil, the oil was added to the virgin binder/RAB mixture as a percentage of total weight. The oil was combined with the binder by stirring on a hotplate with a glass rod until the oil had been visually incorporated into the mixture.

Even though the FT-IR testing comprised of RAB and unaged virgin binder, some blends of binder underwent short term ageing simulation in the rolling thin-film oven (RTFO) to check for mass loss. The mass loss obtained from the RTFO is shown in Table 1.

Table 1. Mass Loss of Selected Samples.

Sample	Mass Loss (%)
Waste Engine (W.E.) Oil (Rep 1)	2
Waste Engine Oil (Rep 2)	2.57
Virgin Binder (Rep 1)	0.86
Virgin Binder (Rep 2)	1.42
25% RAB, 8% W.E. Oil (Rep 1)	0.86
25% RAB, 8% W.E. Oil (Rep 2)	0.29

Table 2. FT-IR Samples.

Sample Identification	Description	Comments
Virgin Binder	PG 58-28	Neat Binder
RAB	Recovered Binder	ASTM D 2172-05 and ASTM 1856-95a
E. Oil	Unused Engine Oil	Chevron Delo 400 LE SAE 15 w40
W.E. Oil	Pure Waste Engine Oil	Used E. Oil. with 144 Engine Hours
25% RAB	Virgin Binder with 25% RAB	Combination of Above Samples
25% RAB, 4% W.E. Oil	Virgin Binder with 25% RAB and 4% Waste Engine Oil	Combination of Above Samples
25% RAB, 8% W.E. Oil	Virgin Binder with 25% RAB and 8% Waste Engine Oil	Combination of above Samples

Table 3. Asphalt Mixture Mix Formula.

P 3/4"	100
P 1/2"	94
P 3/8"	86.3
P No. 4	68.2
P No. 8	49.2
P No. 16	38.4
P No. 30	27.8
P No. 50	15
P No. 100	6.7
P No. 200	4.5
% RAP	24
% Air Voids	4

Complete results of asphalt binder testing, for the combinations used can be found in previous research [6]. Table 2 shows the complete factorial of samples tested. Waste engine oil was tested to determine the aging indices of the used oil. Also, unused engine oil was tested for comparison with the amount of aging that had taken place in the waste engine oil.

Using a blend of six different aggregate stockpiles, a mix-design conforming to a Michigan 4E1 pavement classification was achieved. This type of pavement has a ½ inch nominal aggregate size and is rated to handle a load of one million equivalent single axle loads (ESALS) at the design thickness over its lifetime [18]. Mixture information is given in Table 3.

Table 4 shows a comprehensive breakdown of the compositions of the samples tested. As shown in the table, "Control" refers to a

Table 4. Specified Asphalt Mixture Compositions.

Sample Name	Virgin Aggregates (g)	Virgin Binder (g)	RAP (g)	Binder from RAP (g)	Waste Engine Oil(g)	Total Weight (g)
Control	3500	207.6	0	0	0	3707.6
0% Oil	2660	173.4	840	32.34	0	3673.4
4% Oil	2660	164.8	840	32.34	8.6	3673.4
8% Oil	2660	155.5	840	32.34	17.9	3673.4

standard asphalt mixture with no RAP and no waste engine oil, "0% oil" is the standard mixture blended with 24% RAP, "4% oil" and "8% oil" are the standard mixture blended with 24% RAP and 4% or 8% waste engine oil respectively. The waste engine oil was added by total weight of binder, so the 4% oil and 8% oil mixes had less virgin binder to compensate for the added oil.

Based on volumetric testing, the optimal binder content was found to be 5.56% asphalt binder, which was the binder content used for all subsequent tests. Extraction and recovery testing revealed the asphalt binder content of the RAP to be 3.85%, which was used to calculate the binder contribution to the new mixture from the RAP. For the samples requiring waste engine oil, the waste engine oil was counted towards the total binder content in the mixture. This means the total binder content included the binder contained in the RAP, the waste engine oil and the virgin binder added together. Mixing and compaction followed standard practices, with the exceptions of the RAP was preheated to 100℃ prior to mixing, and the waste engine oil was added with the virgin asphalt binder [19]. Table 5 shows the averaged values obtained from gyratory compaction of the three replicates made for each mixture.

Table 5. Averaged Gyratory Compaction Values.

Sample Name	Final Height (mm)	Gyrations
Control	86.2	76
0% Oil	86.2	119
4% Oil	86.2	80.8
8% Oil	86.2	113

Table 6. FT-IR Compounds and Functional Groups.

Compound Name	Functional Groups	Spectrum Range (cm^{-1})
Alkanes	C-H	650-910
Butadiene	HC=CH	965
Sulfoxide	S=O	1030
Aromatic Hydrocarbons	C-H, CH_2 and CH_3	1375-1530
Aromatics	C=C	1600
Carbonyl	C=O	1700
Saturated Hydrocarbons	C-H	2850-3000

Methods

FT-IR Testing

In order to quantify the effects of waste engine oil on RAB, Fourier Transform Infrared Spectroscopy (FT-IR) was employed to analyze the different compounds contained within the asphalt binder blends. FT-IR uses infrared light to identify the different organic chemical compounds found in various organic substances. FT-IR was chosen for this study because it can provide the user with both qualitative and quantitative results. This means that FT-IR will not only identify the different compounds, or functional groups, present in a sample, but it will also give some measure of the percentage of each group contained within the sample.

When asphalt binder is analyzed using FT-IR testing, a number of functional groups can be observed within the binder. Table 6 contains a comprehensive list of the common functional groups within asphalt binder [20-22]. Since both asphalt binder and engine oil are both hydrocarbons, most of the samples are comprised mainly of aromatic and saturated hydrocarbons. When dealing with hydrocarbon aging, the two compounds of most interest are the sulfoxides and carbonyls whose wavelengths peak at 1030 cm^{-1} and 1700 cm^{-1} respectively. These two compounds are commonly used to indicate the amount of aging asphalt binder has undergone. The increase in either of these two compounds corresponds to an increase in polar compounds of higher molecular size [23]. Previous research has shown that when the amount of sulfoxides or the amount of carbonyls increases, asphalt binder has undergone aging [20, 24, 25].

To quantify how much of the sample is representative of each peak, Lamontagne proposed a method of numerically integrating the bands around the peaks of interest then normalizing the area over the entire area of the spectral bands between 600 cm^{-1} and 2000 cm^{-1} [26]. This method can be represented by Eqs. (1) and (2), where $I_{C=O}$ is the structural index of the carbonyl compound and $I_{S=O}$ is the structural index of the sulfoxide compound [24, 27]. Used individually, the structural index number is not very useful, since no standardized reference values have been accepted to compare different asphalt binders. However, if FT-IR testing is conducted on a series of binders, the indices of each binder can be compared against each other to give a relative level of aging that has taken place.

$$I_{C=O} = \frac{\text{Area of carbonyl band centered around 1700 cm}^{-1}}{\sum \text{Area of the spectral bands between 2000 cm}^{-1} \text{ and 600 cm}^{-1}} \quad (1)$$

$$I_{S=O} = \frac{\text{Area of sulfoxide band centered around 1030 cm}^{-1}}{\sum \text{Area of the spectral bands between 2000 cm}^{-1} \text{ and 600 cm}^{-1}} \quad (2)$$

Using the changes in structural indices, the FT-IR results can provide insight as to the relative levels of maltenes contained in the asphalt samples. If the FT-IR shows an increase in either the carbonyl or sulfoxide index, it is indicative of an increase in the amount of large polar molecules contained inside the binder [23, 28]. Since Asphaltenes are much larger and more polar than their maltene counterpart, the increase in either index means more asphaltenes. Using these results, the FT-IR test can be used to infer a change of the molecular structure of the asphalt binder. Further evidence of this conclusion can be inferred from Table 6; the only compounds bonded with oxygen are the carbonyls and sulfoxides. If the asphalt binder were to oxidize, it would show as in increase in the carbonyl or sulfoxide indices.

To conduct the FT-IR testing, all of the samples were placed on a silicon substrate, since asphalt binders are unable to maintain a thin rigid shape. The binder was heated on a hotplate, and then a small amount was dripped onto the substrate. The substrate was placed on the hotplate to facilitate the thinning of the sample through heating. Additionally, a glass rod was used to spread the binder to the thickness of a thin film over the substrate, which was achieved when the binder appeared translucent. The exact thickness of the films was not measured.

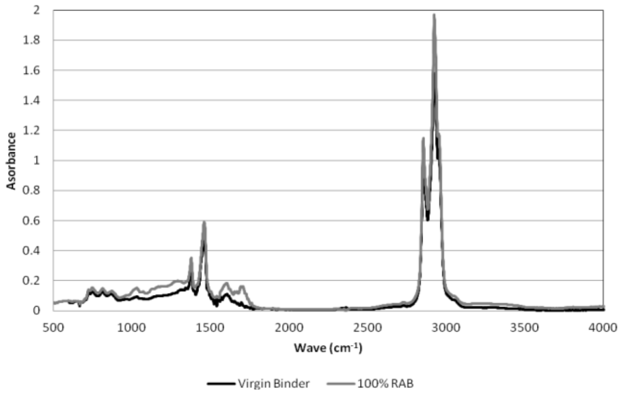
Once the substrates with the binder had cooled, the specimens could be tested. The specimens were tested in by a machine utilizing attenuated total reflectance (ATR). First, a blank silicon substrate was loaded into the FT-IR machine and analyzed to create a background reading of absorbency. This background reading was subtracted from all subsequent tests, leaving only the absorbency of the compounds present in the sample. One at a time, samples were tested and absorbance of every frequency between 500 cm^{-1} to 4000 cm^{-1} was collected.

Mixture Testing

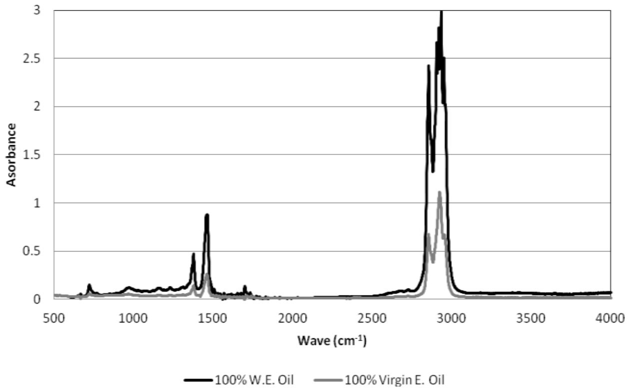
Two different tests were performed on the asphalt mixture samples: the rutting test and the tensile strength ratio (TSR) test. The propensity for rutting was tested using a rutting machine, which simulates traffic loads by passing a 100 pound wheel load over samples at a the pavement's design high temperature, 58°C [29]. For this research, the Asphalt Pavement Analyzer (APA) rutting machine was used to conduct the rutting test. Samples are loaded into the APA rutting machine and allowed to reach thermal equilibrium before testing began. The test runs for 7500 load cycles over the samples, and records the depth of the rut over the duration of the test.

In total, three replicate samples were tested in rutting for each type of sample, Control, 0% Oil, 4% Oil, and 8% Oil. The samples were loaded into the APA rutting machine and given three hours to reach thermal equilibrium at 58℃. The APA machine was run for 7500 cycles and the cumulative rut depth was recorded at each cycle.

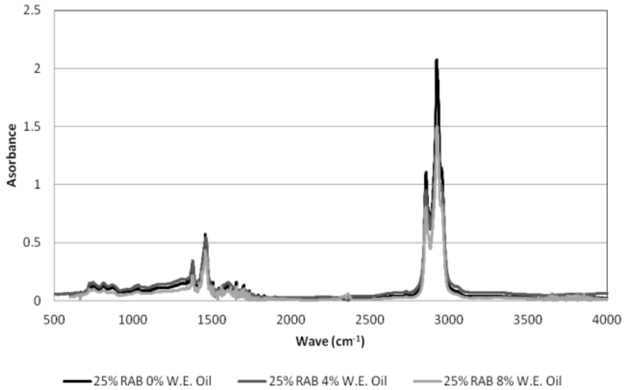
To determine the moisture susceptibility of asphalt samples, the tensile strength ratio (TSR) test was performed. The TSR test was chosen as a way to quantify the effect that waste engine oil would have on the amount of damage a pavement would experience at low temperatures. The TSR testing procedure followed AASHTO T 283 and it compares the indirect tensile strength of one group of unconditioned control samples to another set that has been vacuum-saturated with water, and then frozen [30]. After freezing, the conditioned samples are thermally shocked in a hot water bath to further induce damage. Both the conditioned and unconditioned samples are then crushed in indirect tension and the peak indirect tensile strengths are recorded. Dividing the conditioned indirect tensile strength by the unconditioned indirect tensile strength gives the TSR for each sample.



(a) FT-IR Results for pure binders



(b) FT-IR Results for pure engine oil



(c) FT-IR Results for blends of binder and waste engine oil

Fig. 1. FT-IR Results for (a) Pure Binders (b) Pure Engine Oil and (c) Blends of Binder and Waste Engine Oil.

Results and Discussion

FT-IR Results

The absorbance bands from 500 cm^{-1} to 4000 cm^{-1} were collected from the FT-IR testing of the samples. From these tests, a plot of the wavelengths spanning that range was generated as shown in Fig. 1. A majority of the absorbance seen in the figure are between 1375-1530 cm^{-1} and 2850-3000 cm^{-1}. Recall from earlier, these ranges correspond to aromatic hydrocarbons and saturated hydrocarbons, and for the sake of comparing hydrocarbons to each other, these ranges are not very useful since asphalt binder and waste engine oil are comprised almost entirely of hydrocarbons.

What is meaningful are the changes in the functional groups within the binder, since they can help characterize the aging present in the samples.

Since this research is aimed at analyzing the aging taking place between the samples, only the peak areas around spectral bands at 1030 cm^{-1} and 1700 cm^{-1} were analyzed. Those two peaks, corresponding to sulfoxide (S=O) and carbonyl (C=O) bands, have been identified as relating to aging in asphalt binder [23, 28]. To better illustrate these peaks, Fig. 2 shows an enlarged portion of the graphs of only pure RAB and pure virgin binder. The sulfoxide peak around 1030 cm-1 is much wider and larger for the RAB sample compared to the virgin binder sample. Also, the C=O peak at 1700 is much more pronounced for the RAB sample compared to the virgin binder.

Fig. 3 shows waste engine oil compared to unused engine oil. In the figure, the unused engine oil sample had less sulfoxides and carbonyls than the waste engine oil. The C=O peak for unused engine oil shows considerably less carbonyls than the aged oil. It is possible that a phase stretching phenomenon could have occurred in this sample, meaning the 1700 cm^{-1} could have spread or shifted to incorporate a larger area. For comparison analysis, the unused engine oil peak was measured at 1710 cm^{-1}.

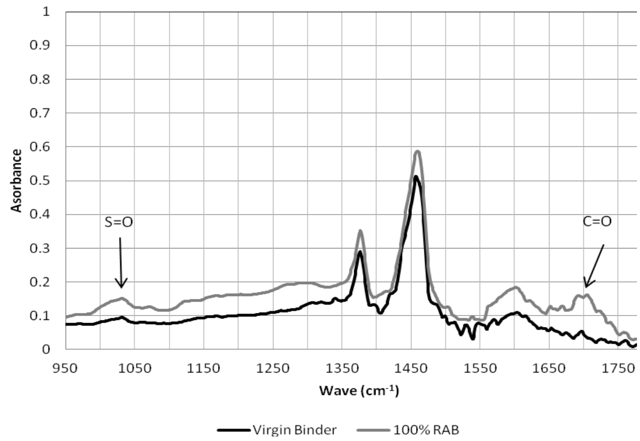


Fig. 2. FT-IR Results: Virgin Binder vs. Pure RAB.

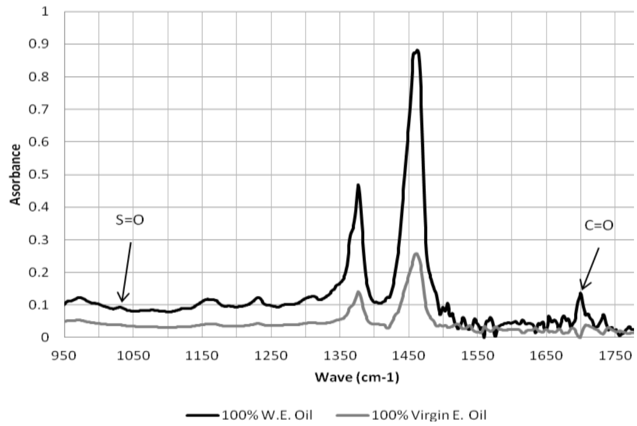


Fig. 3. FT-IR Results: Engine Oil vs. Waste Engine Oil.

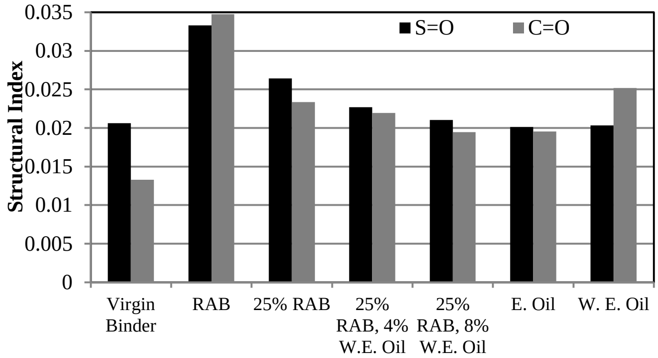


Fig. 4. FT-IR Structural Indices.

FT-IR Discussion

To compute the structural index of each of the samples tested, the peak wavelength of interest is numerically integrating using a valley to valley approach, to obtain the area under the peak. Then the calculated area is divided by the entire area contained between 600 cm^{-1} to 2000 cm^{-1} [26]. The resultant ratio is referred to as the structural index for the compound of interest.

Using the computed structural indexes, a graph of the S=O and C=O structural indices is given in Fig. 4. The FT-IR results of the blended virgin binder, RAB and waste engine oil shows a dispersion of asphaltenes, in that there was a reduction in the percentage of sulfoxides and carbonyls contained within the rejuvenated samples. The reduction of the structural indices of the blended asphalt binder means the ratio of asphaltenes to maltenes has decreased and more maltenes are now present inside the binder.

The RAB indices are shown to be twice that of the virgin binders, indicating pure RAB was more aged than virgin binder. When the virgin binder was blended with RAB there was an increase in the structural indices when compared to only virgin binder, which was expected because of the inclusion of aged material. The unused engine oil had lower indices than the waste engine oil, which was also as expected. The last take away from the figure is the waste engine oil's ability to reduce the indices of the asphalt binder blended with RAB. The reduction in structural indices continued to increase as the percentage of wasted engine oil was increased.

It looks deceptive that the addition of waste engine oil to the 25% RAB sample leads to a reduction in the C=O structural index, since both the waste engine oil and 25% RAB samples have a higher C=O index than their combination. The mixture of waste engine oil and RAB is not as simple as a weighted combination of each component. Since FT-IR testing can only identify the compounds present, not the presence of any reactions that have taken place, the reduction in both structural indices means the sulfoxides and carbonyls could have been converted to other chemical compounds. The combination of 25% RAB and waste engine oil produced a binder with a carbonyl index lower than either of the two inputs, suggesting a chemical reaction has taken place. Research conducted by Carpenter and Wolosick, who tested RAB with recycling agents, concluded there must be a blending of the components taking place instead of a mixture of new and old material [31]. Their findings support the findings of this study, in which a blending of the asphalt binder and waste engine oil was shown. If a composite of new and old had occurred, it would be impossible to mix two high carbonyl indices and produce a lower index. This finding lends support to the

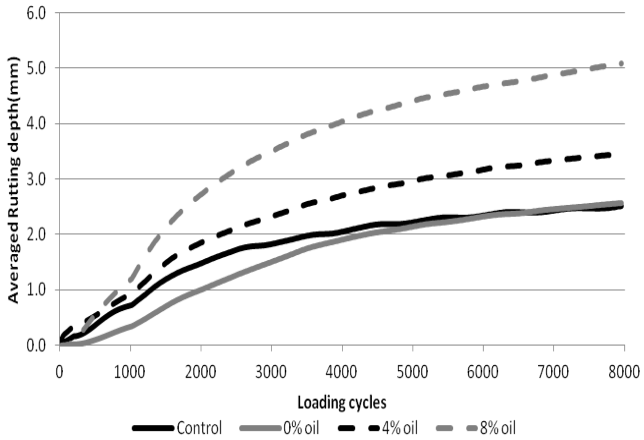


Fig. 5. APA Rutting Results.

Table 7. TSR Results.

	Conditioned Strength (kN)	Unconditioned Strength (kN)	TSR (Conditioned/ Unconditioned)
Control	22.97	26.04	0.88
0% oil	20.96	23.88	0.88
4% oil	14.22	15.37	0.93
8% oil	11.72	14.65	0.80

ability of waste engine oil to chemically rejuvenate aged asphalt binder.

Mixture Results

Plots of the average rut depth versus load cycle can be found in Fig. 5. Because of variability in the data collection from the machine, a running average over 200 cycles is reported. These results show similar resistance to rutting for the control sample and the 0% oil sample; however, the 0% oil sample did have a slightly better initial resistance to rutting. The 4% oil samples had an increased amount of rutting compared to the control, while the 8% oil samples exhibited the greatest amount of rutting.

The TSR values for all of the samples, shown in Table 7, were between 0.80 and 0.93, which is within the acceptable range for TSR values. There was also a loss in indirect tensile strength compared to the control for both the 0% oil sample and the 4% oil and 8% oil samples. This loss in indirect tensile strength became greater with increasing levels of waste engine oil.

Mixture Discussion

Asphalt pavements constructed with RAP is known to produce an overall stiffer pavement [32, 33]. Stiffer pavements are less likely to rut, therefore one would expect to see less rutting as a pavements' stiffness increases. However, the laboratory testing did not show an increase in rutting resistance, which one could expect with a stiffer pavement. The rutting test did show that with the addition of 4% engine oil let to an increase in rutting, with an even greater increase realized with the addition of 8% oil. This excessive rutting makes sense, given that Katamine showed that higher percentages of oil lead to increased deformations [5].

One possible explanation for the lack of increased rutting resistance for the 0% oil sample could be explained by the practice of allowing low amounts of RAP without modification into new mixtures. Another explanation for the lack of stiffening from the RAP could be attributed to the specimens being constructed of 24% RAP by weight of the total mixture; however the RAP binder content was only 3.85% aged binder. The remaining binder required to achieve optimal binder content was from virgin binder which resulted in a mixture whose percentage of binder from the RAP was less than 24%.

The waste engine oil did not adversely affect the TSR of the pavement; however the overall reduction in indirect tensile strength could be detrimental to pavements. The TSR results obtained are similar to previous research, where recycling agents were shown to reduce tensile strength [2, 4]. This reduction in tensile strength means the asphalt pavement may not be able to resist normal stresses in the pavement and could crack prematurely.

Conclusions

FT-IR testing was performed on asphalt binder samples consisting of virgin binder, virgin binder blended with RAB, and two blends of virgin binder, RAB and waste engine oil. FT-IR testing gave relative quantities of the different compounds within the samples of asphalt binder. Those results were used to calculate structural indices of the sulfoxide and carbonyl compounds, which measure aging within asphalt binder, for comparison between samples.

APA rutting and TSR testing were conducted on asphalt mixture with similar percentages of RAP and waste engine oil as those used in the FT-IR testing. The addition of RAP did not increase the indirect tensile strength of the samples or increase their resistance to rutting. With increasing additions of waste engine oil to the pavement specimens, an increase in rutting and reduction in indirect tensile strength was realized.

FT-IR testing showed reductions in carbonyl and sulfoxide structural indices as more waste engine oil is added to a mixture. That reduction translates to an increase in the relative amount of maltenes. Pavements with excessive amounts of maltenes in them are known to suffer from rutting and moisture sensitivity issues, the effects of which can be seen in the results of the mixture testing presented in this paper. While results from FT-IR testing show potential for waste engine oil as a rejuvenator, the fact that it was shown to reduce pavement performance presents a challenge. Further research will be necessary to find a balance between adding enough rejuvenator to restore the binder contained in the RAP, but not too much as to where the rejuvenator becomes detrimental on the performance of the pavement.

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