***Original Research Article***

# Optimizing Percentages of Asphalt Content Extracted from Mixes Containing RAP and/or RAS

.


## ABSTRACT

Extracting asphalt binders from mixes containing reclaimed asphalt pavement (RAP) and/or recycled asphalt shingles needs more investigation. Centrifuge extraction is the most popular method utilized to extract asphalt binders from mixes. Two mineral matter determination methods (MMDMs)—ashing and centrifuge—were used to quantify the fine materials (dust) that are extracted with the effluent. MMDM could underestimate the extracted asphalt content (AC) percentage by overestimating dust amounts. Thus, the extracted AC percentages using ashing and centrifuge MMDMs were compared with the actual AC percentages. The extracted AC percentages using the centrifuge MMDM showed more accurate values when compared to the extracted AC percentages using the ashing MMDM. The fabrication methods used in field, plant, and lab mixes and the additives used in these mixes altered the interaction processes between RAP binders and virgin asphalt binders (VABs). More interactions occurred in the plant mixes due to reheating these mixes in the lab before the compaction. Thus, the extracted AC percentages from the plant mixes were higher than the extracted AC percentages from the same mixes collected from the field. Evoflex increased the interactions between RAP binder and VAB, which increased the extracted AC percentages.



*Keywords: Extraction, centrifuge extractor, asphalt content, ashing, filterless centrifuge, interactions.*


**Comment [AA1]:** e

## 1. INTRODUCTION

Recycling of asphalt pavements began during the 1970s with the oil embargo and the surge in crude oil prices. This resulted in reduced levels of asphalt supply. During that time, contractors screened asphalt mixes containing 80% reclaimed asphalt pavement (RAP) [1–3]. When the oil prices fell, the proportion of RAP in asphalt mixes decreased to 20%. This trend continued throughout the development of Superpave [1–3]. RAP—removed and processed pavement materials—contains valuable materials (e.g., asphalt binder and aggregate) [3–8]. Between the 1980s and 1990s, recycled asphalt shingles (RAS) were used in the asphaltic mixes [1, 2]. In the mid to late 2000s, oil prices rose again increasing the demand for the use of RAP and RAS to reduce the overall cost [1, 2].

**Comment [AA2]:** This is somewhat not true. The use of high percentages of RAP has negative results on the performance of the asphalt mixture

**Comment [AA3]:** The use of RAP is not only due to the high prices of asphalt

Different methods can be used to extract asphalt binders from the asphaltic mixes. However, the centrifuge extraction method is the most common method used to extract asphalt binders from mixes using solvents [9–11] because of its simplicity and use at room temperature [10–13]. This method is used if the characterizing of extracted asphalt binders (EABs) is necessary. However, one of the main drawbacks of this method is leaving around 4% of the total binder with the aggregate [9, 12, 14]. The used solvents dissolve the asphalt binders during the extraction process, and mineral matters (dust) are getting out with the dissolved

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asphalt binders. Then, the mineral matters are removed using a filterless centrifuge. After removing the mineral matter from the extracted solvent, the asphalt binders are recovered using a distillation process that is achieved by a rotary evaporator (rotavap) [15, 16]. This recovery process was used since the 1970s [17]; however, the overheating process in the rotavap would result in increasing the stiffness of the recovered asphalt binders [18–20]. Furthermore, any remaining solvent in the recovered asphalt binders could result in decreasing the asphalt binders' stiffnesses. It was observed that even 0.5% of the solvent remaining in the recovered asphalt binders could result in a 50% decrease in the viscosity value [20]. Nösler et al. [21] found a decrease in the ring and ball softening point by 6 °C because of the presence of trichloroethylene (TCE) in the recovered binder by a percentage of 0.9% by weight.

Rodezno and Julian [9] investigated the effect of different extraction methods—centrifuge, ignition, automated using the asphalt analyzer, and reflux—on the properties of EABs. Eight mixes were analyzed; those mixes either contained RAP/RAS or none. The testing program was achieved with the collaboration of different laboratories in Wisconsin to evaluate the within-lab and between-lab variability. For the centrifuge extraction method, an average difference between the percentages of the actual and EABs was found to be 0.21% for mixes containing a virgin asphalt binder (VAB) and may reach 0.38% for mixes containing a high percentage of RAP/RAS, recycled binder percentage of 30-35%. The within-lab and between-lab variability were not affected by using RAP/RAS in the asphaltic mixes. Ignition and asphalt analyzer extraction methods had the highest accuracy because the average differences between the percentages of actual and EABs were 0.05% and 0.17%, respectively. However, the ignition method can not be used when the characterization of EAB is necessary. Unfortunately, the asphalt analyzer apparatus is not available in our Missouri S&T lab. Regardless of the extraction method and type of solvent—toluene, TCE, and n-propyl bromide—there was no significant difference in the performance grade (PG) characterization of EABs [9]. However, another research showed that there was a difference in the properties of EABs using the three aforementioned types of solvents [22].

Piérard et al. [23] investigated the effect of the extraction process on the asphalt binder content. Styrene butadiene styrene or ethyl vinyl acetate modified asphalt binders were extracted from fresh, short-term aged, and compacted mixes prepared in the lab. Two sources of asphalt binders and two types of aggregates were used. Different solvents—toluene, dichloromethane, and TCE—were used to extract binders from mixes. The average extracted content of the asphalt binder, regardless of the type of the solvent, was 6.3±0.2% that was considered lower than the initial binder content (6.6%). Fourier transform infrared results showed that the decrease of the asphalt binder content was not related to the decreases in the polymer content because the intensities of the released polymer's peaks were similar for the modified binder used in the preparation of mixes and the recovered one. No appreciable effect of the aggregate type and/or the compaction process on the percentage of EAB was noted. The percentage of EAB from short-term aged mixes depended on the type of the asphalt binder and the used solvent.

Sirin and Tia [24] evaluated the effect of the extraction process using reflux on the extracted percentage of asphalt binder from field and lab asphalt mixes containing crumb rubber modifier (CRM). Mixes modified with CRM had an actual asphalt binder content of 6.34% and a CRM percentage of 0.76%, which was a total of asphalt and CRM content of 7.1% by the weight of the mix. Additionally, conventional mixes with only an asphalt binder percentage of 6.34% were examined. The researchers concluded that the percentage of the EAB was lower than the actual one either for the modified or unmodified mixes. For CRM modified mixes, an average not extracted percentage of asphalt binder and CRM was found to be 0.86%. For conventional mixes, an average not extracted asphalt binder percentage of

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0.25% was discovered. Thus, the average percentage of CRM that remained in the reflux was 0.61% (0.86% − 0.25%) out of a CRM percentage of 0.76% by the weight of the mix. This has proven that utilizing recycled materials such as CRM in asphalt mixes made the extraction process of the asphalt binders from these mixes more difficult.

Using recycled materials in asphalt mixes not only changes the performances of the EABs but also makes the extraction process more difficult. Thus, the main objective of this study was to optimize the asphalt content (AC) percentage extracted from mixes containing RAP and/or RAS using the centrifuge extraction process. Various fabrication methods were used in field, plant, and lab mixes containing different VABs and different asphalt binder replacement (ABR) percentages by RAP and/or RAS. The objective of this study was achieved by comparing the extracted AC percentage using the centrifuge extraction process with the actual AC percentage. The mineral matter determination method (MMDM) could underrate the extracted AC percentage [10]. Therefore, the effect of MMDM on the extracted AC percentage was evaluated. Different fabrication methods used in field, plant, and lab mixes could alter the interactions between VAB and RAP/RAS binder. Using recycling agents (e.g., Evoflex) increased the contribution of the RAP binder in the mixes, which increased the interactions between the RAP binder and VAB [8]. Increasing these interactions could enhance the extracted AC percentage when compared to the actual AC percentage, which was investigated in this study.

## 2. MATERIAL AND METHODS

### 2.1 Materials

#### 2.1.1 Field mixes

Sixty field samples were collected as cores from different routes in two batches: The first batch had 38 samples (Fig. 1) and the second batch had 22 samples (Fig. 2). More details about the first and second batch samples are presented in Tables 1 and 2, respectively. The field cores presented in Table 1 were collected in 2016, and the field cores in Table 2 were sampled in 2019. For field samples taken from routes constructed in 2016, the cores were sampled within two weeks after the pavement construction process in 2016. These mixes contained different ABR percentages by RAP and/or RAS and different additives. The additives' percentages in the job mix formula (JMF) were specified by the net weight of VAB. Some mixes contained neither RAP nor RAS (e.g., US 54-5, US 54-7, and MO 94). The total AC % values in Tables 1 and 2 represent the actual AC %, as defined by the JMF.

#### 2.1.2 Plant mixes

Four asphalt mixes were designed following Superpave, and each mix was mixed in a drum-mix plant. The plant was located near the intersection of Lakeside Rd. and US 54, near Lakeland, Missouri. The asphalt contractor was Magruder Paving, LLC. Twelve plant mixes were sampled from behind the paver during the construction process; these plant mixes represented four asphalt mixes. Plant mixes were reheated to 100 ± 5 °C in the lab before separation, then they were reheated to the compaction temperature specified in the JMF and compacted using Superpave gyratory, as shown in Fig. 2. These mixes contained either RAP or RAS. More details about these mixes are illustrated in Table 3.

**Comment [AA4]:** Not required

#### 2.1.2 Lab mixes

Different lab mixes, presented in Fig. 2, were designed following Superpave using the same materials (e.g., aggregate, asphalt binder, RAP, and additives) used in the US 54-6 and US

**Comment [AA5]:** field, plant, and lab. ?

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63-1 plant or field mixes. The used additives were Morelife, Evoflex, and Evotherm. Using a softer VAB in mixes containing recycled materials (e.g., RAP) is recommended [25] to increase the workability characteristics because of the aged binders in RAP. Thus, a softer asphalt binder with a PG of 46−34 was used in lab mixes to evaluate the effect of using a soft asphalt binder in mixes containing RAP when compared to mixes containing the same materials and a stiffer binder (PG 58−28). To promote the sustainability of mixes containing RAP, rubber was utilized in these mixes. An engineered crumb rubber (ECR), a type of dry-process ground tire rubber, with three percentages—5%, 10%, and 20%—by the net weight of total binder were used in lab mixes. Asphalt binder and ECR were heated to 170 °C then blended in a high-shear mixer at 3500 revolutions per minute for 30 minutes. After mixing aggregates with binders or modified binders, the mixes were short-term aged in the oven at the compaction temperature, specified in the JMF, for two hours before the compaction process. Finally, lab mixes were compacted using a Superpave gyratory compactor. More details about these mixes are shown in Table 4. Field, plant, and lab mixes' codes represent the route name (e.g., MO 13), section number (e.g., 1), and coding system (e.g., F1).

**Comment [AA6]:** non-sequential sentences. confusing to the reader.

**Fig. 1. The first batch of field mixes [5]**

**Comment [AA7]:** Is ref.5 prepared these samples? please, clarify.

**Fig. 2. The second batch of field mixes; plant and lab mixes [5]**

**Comment [AA8]:** There are three types of samples, batch, plant, and lab. Please separate the figure into three figures or a, b, and c.

**Comment [AA9]:** Is ref.5 prepared these samples? please, clarify.

## 2.2 Methods

The centrifuge extraction process for asphalt binders from mixes was performed according to ASTM D2172 / D2172M-17e1 [15], discussed as method A. The TCE solvent was used to extract the asphalt binder from the mixes using a centrifuge extractor model H1460 obtained from Ploog Engineering Co., Inc., Crown Point, IN, U.S.A. The centrifuge extractor is shown in Fig. 3(a). A representative sample of approximately 100 ml of effluent—EAB, TCE, and

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mineral matter—was taken into an ignition dish to quantify the amount of mineral matter in the effluent using the ashing method. To better estimate the extracted percentage of AC, the representative sample of the effluent was taken at least twice in two different ignition dishes at a rate of 100 ml per dish. The amount of the mineral matter was removed and calculated in the remaining effluent using a filterless centrifuge obtained from Ploog Engineering Co., Inc, as presented in Fig. 3(b). The mineral matter using ashing and centrifuge methods are presented in Figs. 3(c) and 3(d), respectively. Hence, the extracted percentage of AC was calculated using the ashing MMDM, centrifuge MMDM, and average ashing & centrifuge MMDMs.

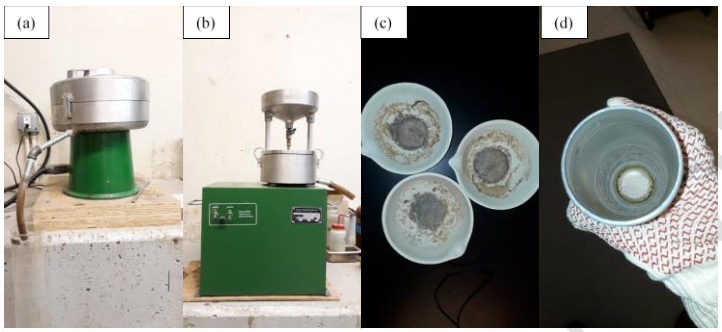


**Fig. 3. (a) Centrifuge extractor, (b) Filterless centrifuge, (c) Ashing dishes containing mineral matter, and (d) Centrifuge metal cup containing mineral matter [5]**

**Comment [AA11]:** ?

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**Table 1. Details of the first batch of field mixes [5]**

| Code | County, Route/Dir, and Location | Virgin Asphalt PG | Virgin AC[a] (%) | Total AC (%) | ABR by RAP–RAS (%) | NMAS[b] (mm) | Const.[c] Year | Additives |
|---|---|---|---|---|---|---|---|---|
| MO 13-1-F1<br>MO 13-1-F2<br>MO 13-1-F3 | Henry, MO 13 NB, and S. of Clinton | 64−22H | 4.4 | 5.7 | 17–0 | 9.5 | 2016 | Morelife T280 0.5% |
| US 54-6-F1<br>US 54-6-F2<br>US 54-6-F3 | Miller, US 54 NB, and N. of Osage Beach | 58−28 | 3.6 | 5.1 | 31–0 | 12.5 | 2016 | Morelife T280 1% |
| US 54-1-F1<br>US 54-1-F2<br>US 54-1-F3 | Miller, US 54 SB, and N. of Osage Beach | 58−28 | 3.6 | 5.2 | 0–33 | 12.5 | 2016 | IPC70 2.5%, PC2106 3.5%, Morelife T280 1.5% |
| US 63-1-F1<br>US 63-1-F2<br>US 63-1-F3 | Randolph, US 63 SB, and S. of Moberly | 58−28 | 3.4 | 5.1 | 35–0 | 12.5 | 2016 | Evotherm 0.5%, Evoflex CA 1.75% |
| US 63-2-F1<br>US 63-2-F2<br>US 63-2-F3 | Macon, US 63 SB, and N. of Macon, near LaPlata | 64−22 | 4.1 | 5.6 | 20–10 | 12.5 | 2008 | Bag house fines (BHF) 1.5%,AD-here HP plus 0.5% |
| US 54-3-F1<br>US 54-3-F2<br>US 54-3-F3 | Miller, US 54, Osage Beach | 58−28 | 3.6 | 5.2 | 18–15 | 12.5 | 2016 | Morelife T280 1% |
| US 54-5-F1<br>US 54-5-F2 | Miller, US 54, and Osage Beach | 64−22H | 5.4 | 5.4 | 0–0 | 12.5 | 2016 | Morelife T280 1% |
| US 54-4-F1<br>US 54-4-F2<br>US 54-4-F3 | Miller, US 54, and Osage Beach | 64−22H | 3.2 | 4.8 | 35–0 | 12.5 | 2016 | PC2106 3%, Morelife T280 1% |
| US 54-2-F1<br>US 54-2-F2<br>US 54-2-F3 | Miller, US 54, Osage Beach | 58−28 | 3.6 | 5.3 | 33–0 | 12.5 | 2016 | Morelife T280 1% |
| US 50-1-F1<br>US 50-1-F2<br>US 50-1-F3 | Moniteau/Morgan, US 50, and Tipton | 64−22 | 3.8 | 5.0 | 25–0 | 12.5 | 2011 | BHF 1.5%, AD-here HP plus 1% |
| MO 52-1-F1 | Morgan, MO 52, and | 64−22 | 3.7 | 4.8 | 0–34 | 12.5 | 2010 | BHF 1.5%, AD-here HP |

Comment [AA12]: ?

Comment [AA13]: Note for the four tables: These two columns confuse the reader. A third column can be added to number the samples, i.e. sample 1, 2, 3, and so on. Thus, the samplel number is indicated instead of the code, country, direction and location.

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|  |  |  |  |  |  |  |  |  |
|---|---|---|---|---|---|---|---|---|
| MO 52-1-F2<br>MO 52-1-F3 | Versailles |  |  |  |  |  |  | plus 0.8% |
| US 54-7-F1<br>US 54-7-F2<br>US 54-7-F3 | Cole, US 54 WB, and Brazito | 64−22 | 6.2 | 6.2 | 0–0 | 12.5 | 2003 | LOF 65-00LS1 0.25% |
| US 54-8-F1<br>US 54-8-F2<br>US 54-8-F3 | Cole, US 54, and S. of Jeff City | 70−22 | 5.1 | 5.6 | 9–0 | 12.5 | 2006 | AD-here HP plus 0.5% |

*[a] AC: Asphalt Content, [b] NMAS: Nominal Maximum Aggregate Size, and c Const.: Construction.*
*Morelife T280, AD-here HP Plus, LOF 65-00LS1, and IPC-70: anti-stripping agents.*
*Evotherm and PC 2106: warm-mix additives.*
*Evoflex CA: rejuvenator additive.*

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**Table 2. Information on the second batch of field mixes [5]**

| Code | Route/Dir | Virgin Asphalt PG | Total AC (%) | ABR by RAP–RAS (%) | NMAS (mm) | Date of Most Recently Overlay |
|---|---|---|---|---|---|---|
| MO 151-F1<br>MO 151-F2<br>MO 151-F3<br>MO 151-F4<br>MO 151-F5 | MO 151 | 64−22 | 4.7 | 16–15 | 12.5 | 2014 |
| US 61 N-F1<br>US 61 N-F2<br>US 61 N-F3 | US 61 N | 64−22H | 5.3 | 30–0 | 9.5 | 2013 |
| US 54-F1<br>US 54-F2<br>US 54-F3 | US 54 E | 70−22 | 5.7 | 12–0 | 12.5 | 2010 |
| MO 6-F1<br>MO 6-F2<br>MO 6-F3<br>MO 6-F4<br>MO 6-F5 | MO 6 W | 58−28 | 5.9 | 30–0 | 4.75 | 2015 |

| | | | | | | |
|---|---|---|---|---|---|---|
| MO 94-F1<br>MO 94-F2<br>MO 94-F3 | MO 94 | 64−22 | 5.6 | 0–0 | 12.5 | 2005 |
| US 36-F1<br>US 36-F2<br>US 36-F3 | US 36 E | 64−22 | 5.1 | 25–0 | 12.5 | 2011 |

**Table 3. Plant mixes' information [5]**

| Code | County, Route/Dir, and Location | Virgin Asphalt PG | Virgin AC (%) | Total AC (%) | ABR by RAP–RAS (%) | NMAS (mm) | Const. Year | Additives |
|---|---|---|---|---|---|---|---|---|
| MO 13-1-P1<br>MO 13-1-P2<br>MO 13-1-P3 | Henry, MO 13 NB, and S. of Clinton | 64−22H | 4.4 | 5.7 | 17–0 | 9.5 | 2016 | Morelife T280 0.5% |
| US 54-6-P1<br>US 54-6-P2<br>US54-6-P3 | Miller, US 54 NB, and N. of Osage Beach | 58−28 | 3.6 | 5.1 | 31–0 | 12.5 | 2016 | Morelife T280 1% |
| US 54-1-P1<br>US 54-1-P2<br>US 54-1-P3 | Miller, US 54 SB, and N. of Osage Beach | 58−28 | 3.6 | 5.2 | 0–33 | 12.5 | 2016 | IPC70 2.5%, PC2106 3.5%, Morelife T280 1.5% |
| US 63-1-P1<br>US 63-1-P2<br>US 63-1-P3 | Randolph, US 63 SB, and S. of Moberly | 58−28 | 3.4 | 5.1 | 35–0 | 12.5 | 2016 | Evotherm 0.5%, Evoflex CA 1.75% |

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**Table 4. Lab asphalt mixes' information [5]**

| Code | Virgin AC (%) | Total AC (%) | Virgin Asphalt PG | ABR by RAP–RAS (%) | ECR[a] (%) | Additives |
|---|---|---|---|---|---|---|
| | | | US 54-6 Lab Mixes | | | |
| US 54-6-L1 | 3.6 | 5.1 | 58−28 | 31–0 | 0 | |
| US 54-6-L2 | | | | | | |
| US 54-6-L3 | | | | | | |
| US 54-6-R[b]-L1 | | | | | | 3% Evoflex |
| US 54-6-R-L2 | | | | | | |
| US 54-6-SB[c]-L1 | | | 46−34 | | | |
| US 54-6-SB-L2 | | | | | | |
| US 54-6-SB-E5[d]-L1 | 3.7 | 5.2 | | | 5 | |
| US 54-6-SB-E5-L2 | | | | | | |
| US 54-6-SB-E5-L3 | | | | | | |
| US 54-6-SB-E20[e]-L1 | 4.0 | 5.5 | | | 20 | |
| US 54-6-SB-E20-L2 | | | | | | |
| | | | US 63-1 Lab Mixes | | | |
| US 63-1-R-L1 | 3.4 | 5.1 | 58−28 | 35–0 | | 1.75% Evoflex & 0.5% Evotherm |
| US 63-1-R-L2 | | | | | | |
| US 63-1-R-L3 | | | | | | |
| US 63-1-SB-L1 | | | 46−34 | | | |
| US 63-1-SB-L2 | | | | | | |
| US 63-1-SB-L3 | | | | | | |
| US 63-1-SB-R-L1 | | | | | | 1.75% Evoflex & 0.5% Evotherm |
| US 63-1-SB-R-L2 | | | | | | |
| US 63-1-SB-R-L3 | | | | | | |
| US 63-1-SB-E10-L1 | 3.6 | 5.3 | | | 10 | |
| US 63-1-SB-E10-L2 | | | | | | |
| US 63-1-SB-E20-L1 | 3.8 | 5.5 | | | 20 | |
| US 63-1-SB-E20-L2 | | | | | | |

*[a] ECR: Engineered Crumb Rubber, [b] R: Rejuvenator, [c] SB: Soft Binder, [d] E5: 5% ECR, and [e] E20: 20% ECR.*

## 3. RESULTS AND ANALYSIS

### 3.1. Extraction of Asphalt Binders from Plant Mixes

The actual AC versus the extracted AC percentages for plant mixes using ashing MMDM, centrifuge MMDM, and average ashing & centrifuge MMDMs are illustrated in Fig. 4. Only two samples had the same actual AC and extracted AC using ashing MMDM as illustrated in Fig. 4(a). Moreover, around 60% of the samples had extracted AC values lower than the actual AC percentages. This illustrates that the ashing MMDM underestimated the extracted AC percentage. By using the centrifuge MMDM, Fig. 4(b), one-third of the samples had extracted AC percentages with the same values as the actual AC percentages. Fig. 4(c) shows the extracted versus actual AC percentages; the extracted AC percentages were calculated using the average ashing & centrifuge MMDMs. The extracted AC percentages using the average ashing & centrifuge MMDMs had more accurate results when compared to the extracted AC percentages using the ashing MMDM and less accurate results when compared to extracted AC percentages using the centrifuge MMDM.

**Comment [AA14]:** An explanation for these behaviors is required

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The one-way analysis of variance (ANOVA) presented in Table 5 was calculated using JMP Pro software. An ANOVA was conducted to compare the means of the different extracted AC percentages using different MMDMs and the mean of the actual AC percentages. There was no significant difference between the means of the actual AC percentages and extracted AC percentages using the ashing MMDM, centrifuge MMDM, or the average of ashing & centrifuge MMDMs. This was illustrated by the Prob > F (p-value) because it was greater than the significance level α (0.05).

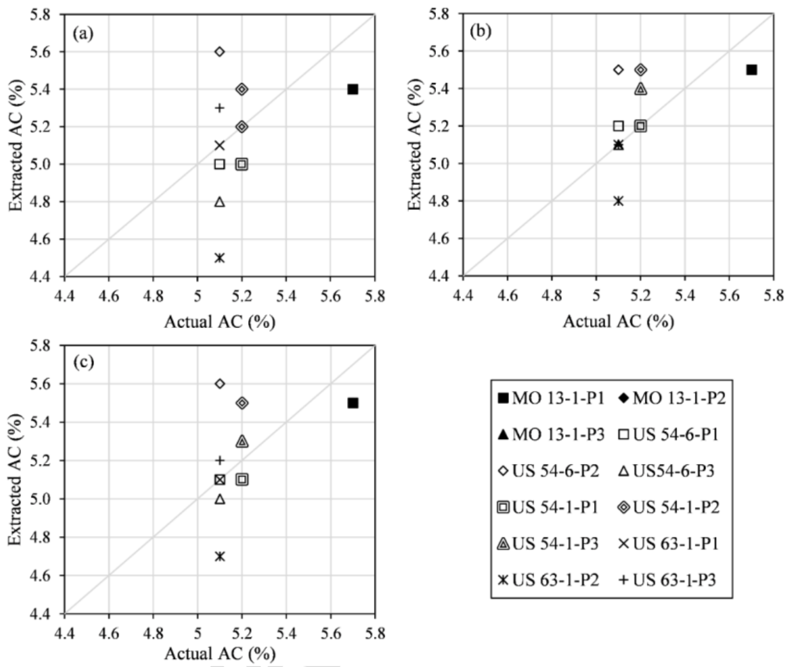


**Fig. 4. Actual versus extracted AC percentages for plant mixes; (a) Ashing MMDM, (b) Centrifuge MMDM, and (c) Average ashing & centrifuge MMDMs**

**Comment [AA15]:** Not all the samples indicated in the key are shown in the figures.

**Table 5. ANOVA results: actual and extracted AC percentages for plant mixes**

| Source | DF[a] | Sum of Squares | Mean Square | F Ratio | Prob > F |
|---|---|---|---|---|---|
| Method | 3 | 0.089 | 0.030 | 0.406 | 0.749 |
| Error | 44 | 3.211 | 0.073 | | |
| C. Total | 47 | 3.300 | | | |

[a] *DF: degrees of freedom*

**Comment [AA16]:** d.f.

Fig. 5 shows the extracted AC per actual AC percentages for plant mixes using different MMDMs. For around 75% of plant mixes, it was noted that the extracted AC percentages using the centrifuge MMDM were higher than the extracted AC percentages using the ashing MMDM. By using average ashing & centrifuge MMDMs, the extracted AC per actual AC percentages were between 91% and 109% for mixes containing RAP, and between 98%

and 105% for mixes containing RAS. Consequently, the extracted AC percentages from mixes containing RAS were more accurate than the extracted AC percentages from mixes containing RAP.

**Comment [AA17]:** An explanation for these behaviors is required

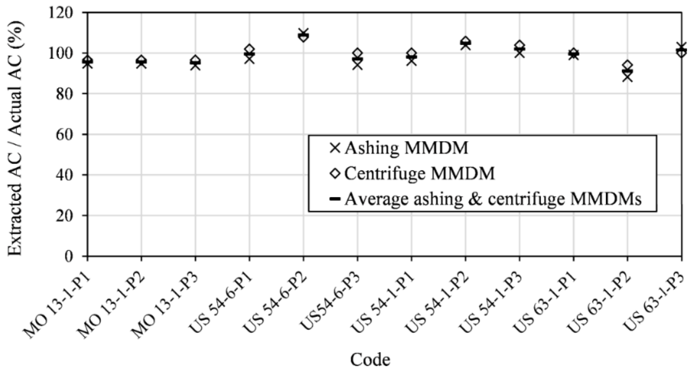


**Fig. 5. Extracted per actual AC percentages for plant mixes**

### 3.2. Extraction of Asphalt Binders from Field Mixes Constructed before 2016

Fig. 6 illustrates the actual AC versus the extracted AC percentages using different MMDMs for field mixes constructed before 2016. Field mixes contained RAP and/or RAS, and some field mixes contained neither RAP nor RAS (e.g., US 54-7 and MO 94). The actual AC percentages were between 4.7% and 6.2%. Using the ashing MMDM, the extracted AC percentages were between 4.3% and 6.8%. The extracted AC percentages using the centrifuge MMDM were between 4.6% and 6.8%. The extracted AC percentages using the average ashing & centrifuge MMDMs were between 4.5% and 6.8%. The ashing MMDM underestimated the extracted AC percentages because most of the extracted AC percentages were located under the inclined line. This inclined line indicates samples that had the same actual and extracted AC percentages.

**Comment [AA18]:** Why?

**Comment [AA19]:** self-evident

To understand the effect of MMDMs on the extracted AC%, the ANOVA was implemented and presented in Table 6. The Prob > F was found to be 0.869, which was greater than the significance level α (0.05). Thus, no significant difference between means of the actual AC and extracted AC using the ashing MMDM, centrifuge MMDM, or the average of ashing & centrifuge MMDMs was observed.

The extracted AC per actual AC percentages for field mixes using different MMDMs are presented in Fig. 7. For most samples—71% of the samples—the extracted AC using centrifuge MMDM per actual AC percentages was higher than the extracted AC using the ashing MMDM per actual AC percentages. The highest extracted AC per actual AC percentages were recorded for extracted binders from the MO 6 mixes. These mixes were recently constructed in 2015 and contained VAB with a PG of 58−28, which was softer than VABs used in the other mixes. However, these mixes contained a high ABR percentage by RAP (30%). Therefore, using a soft VAB in the mixes facilitated the extraction process especially if those mixes contained a high percentage of RAP and/or RAS.

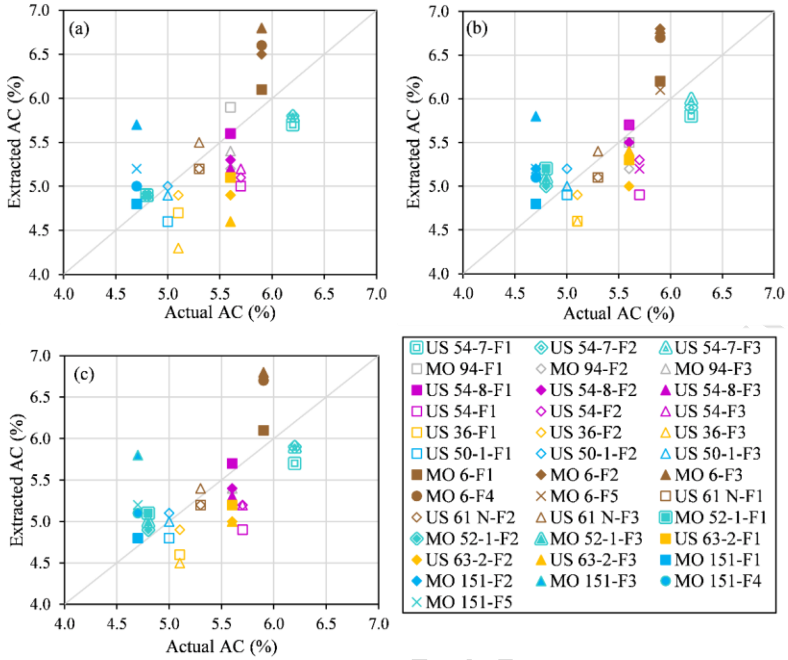


**Fig. 6. Actual versus extracted AC percentages for field mixes constructed before 2016; (a) Ashing MMDM, (b) Centrifuge MMDM, and (c) Average ashing & centrifuge MMDMs**

**Table 6. ANOVA results: actual and extracted AC percentages for field mixes constructed before 2016**

| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
|---|---|---|---|---|---|
| Method | 3 | 0.213 | 0.071 | 0.239 | 0.869 |
| Error | 144 | 42.829 | 0.297 | | |
| C. Total | 147 | 43.042 | | | |

### 3.3. Extraction of Asphalt Binders from Field, Plant, and Lab Mixes

The actual AC and extracted AC percentages using different MMDMs are presented in Fig. 8 for the US 54-6 field, plant, and lab mixes. These mixes contained 31% ABR percentage by RAP. The actual AC percentage for field, plant, and lab mixes was 5.1%; however, some lab mixes had actual AC percentages of 5.2% or 5.5%. The extracted AC percentages using ashing MMDM, presented in Fig. 8(a), were between 4.4% and 6.0%. The centrifuge MMDM, Fig. 8(b), showed more accurate extracted AC percentages that were between 4.8% and 5.6%. Fig. 8(c) shows the extracted AC percentages using the average ashing & centrifuge MMDMs. The ashing MMDM underestimated the extracted AC percentages. Thus, for more than 71% of the samples in Figs. 8(a) or 8(c), the extracted AC percentages were lower than the actual AC percentages.

**Comment [AA20]:** Explain the reasons for this difference

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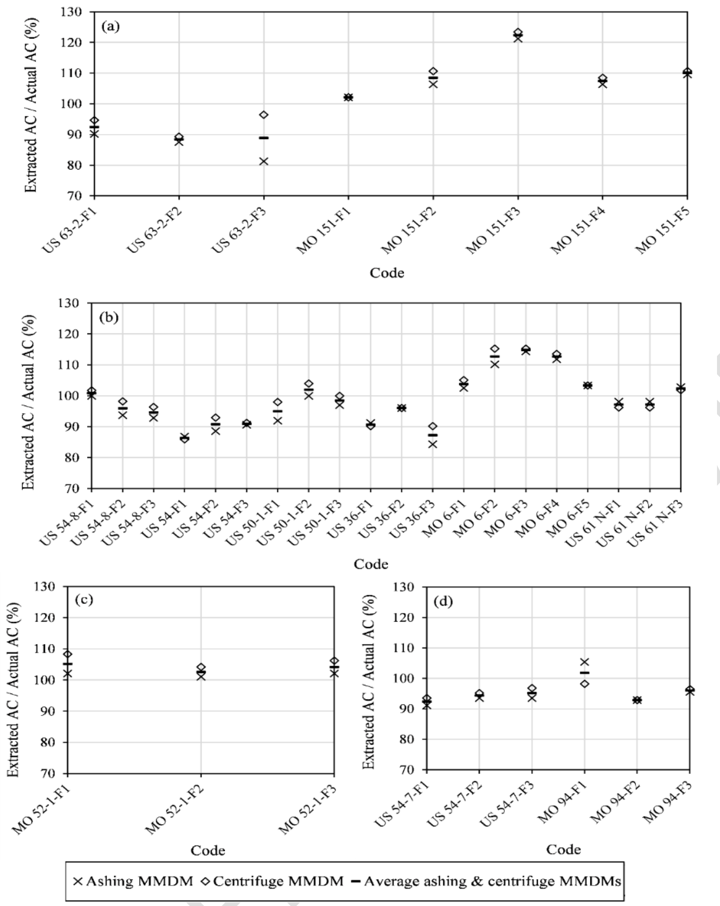


**Fig. 7. Extracted per actual AC percentages for field mixes constructed before 2016; (a) Mixes containing RAP and RAS, (b) Mixes containing RAP, (c) Mixes containing RAS, (d) Mixes containing neither RAP nor RAS**

**Comment [AA21]:** The difference in results for the four cases was not explained

The actual AC versus extracted AC percentages using different MMDMs for the US 63-1 field, plant, and lab mixes are illustrated in Fig. 9. These mixes contained 35% ABR percentages by RAP. The actual AC for field, plant, and lab mixes was 5.1%; however, some lab mixes had actual AC% of 5.3% and 5.5%. The extracted AC percentages using the ashing MMDM, Fig. 9(a), were between 4.5% and 6.0%. The centrifuge MMDM, Fig. 9(b), presented more accurate extracted AC percentages that had values between 4.7% and 5.8%. Using both ashing and centrifuge MMDM, Fig. 9(c), resulted in extracted AC percentages between 4.6% and 5.9%.

To better understand the effect of MMDMs on the extracted AC percentages, the means of the extracted AC using different MMDMs were compared with the mean of the actual AC. The ANOVA results are shown in Table 7. The p-value (Prob > F) is 0.383, which was greater than the 0.05 significance level. Thus, no significant difference was found between the means of the extracted AC using different MMDMs when compared to the mean of the actual AC.

Comment [AA22]: This is a review of the results without giving reasons for these differences, although there are fundamental differences in each mixture

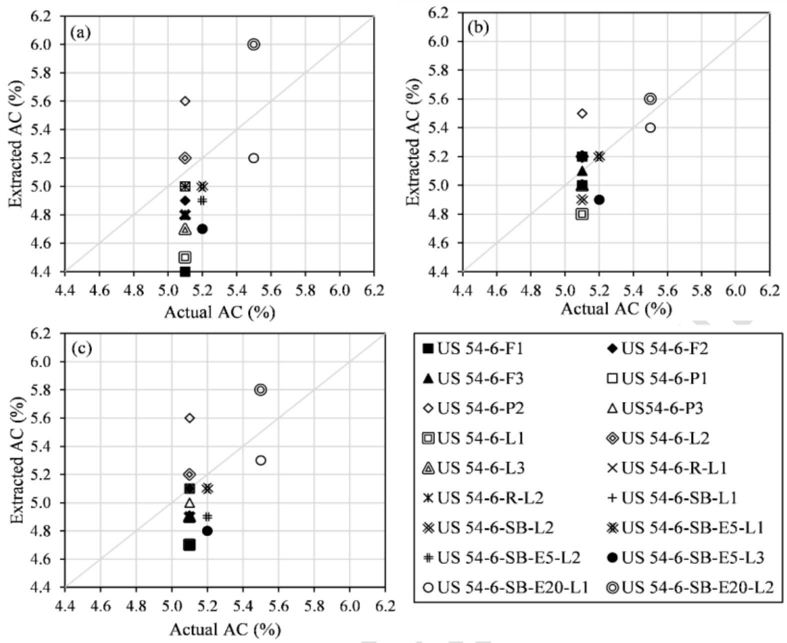


**Fig. 8. Actual versus extracted AC percentages for the US 54-6 field, plant, and lab mixes; (a) Ashing MMDM; (b) Centrifuge MMDM, and (c) Average ashing & centrifuge MMDMs**

Fig. 10 presents the extracted AC per actual AC percentages for the US 54-6 field, plant, and lab mixes using different MMDMs. The extracted AC per actual AC percentages were between 85% and 110%. The centrifuge MMDM showed higher extracted AC than the ashing MMDM. Additionally, the highest extracted AC per actual AC percentages were found for the US 54-6 plant mixes. More interactions between VAB and RAP binder occurred in plant mixes due to reheating these mixes in the lab before the compaction process. These interactions increased the extracted AC percentages.

Fig. 11 presents the extracted AC per actual AC percentages for the US 54-6 lab mixes using different MMDMs. The extracted AC per actual AC percentages were between 88% and 110%. The centrifuge MMDM had higher extracted AC when compared to the ashing MMDM for most samples (more than 83% of the samples). Adding 3% Evoflex increased the extracted AC per actual AC percentages, which reflected the role of Evoflex in enhancing the contribution of the recycled materials in the mixes. This contribution increased the interactions between the RAP binder and VAB. The same results were observed with a softer VAB (PG 46−34); fewer variations were observed for the extracted AC per actual AC percentages using different MMDMs.

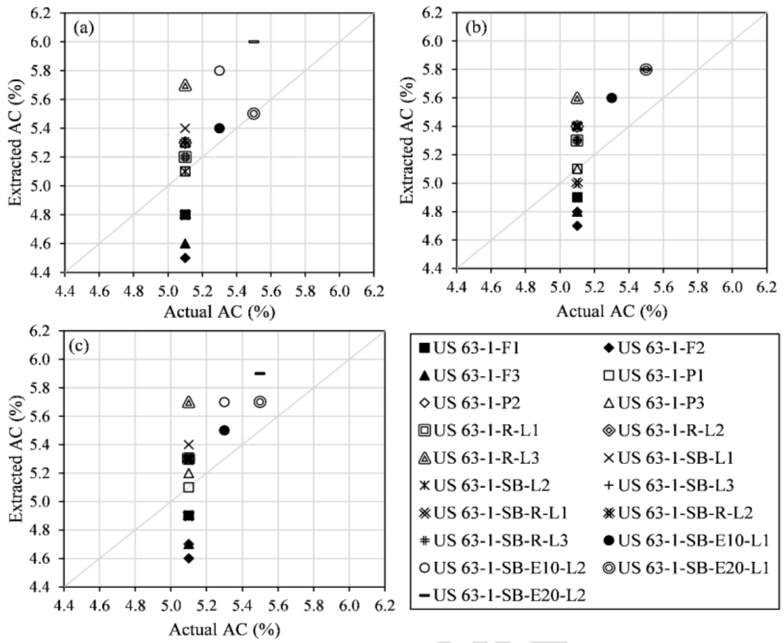


**Fig. 9. Actual versus extracted AC percentages for the US 63-1 field, plant, and lab mixes; (a) Ashing MMDM, (b) Centrifuge MMDM, and (c) Average ashing & centrifuge MMDMs**

**Table 7. ANOVA results: actual and extracted AC percentages for the US 54-6 and US 63-1 field, plant, and lab mixes**

| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
|---|---|---|---|---|---|
| Method | 3 | 0.294 | 0.098 | 1.025 | 0.383 |
| Error | 144 | 13.774 | 0.096 | | |
| C. Total | 147 | 14.068 | | | |

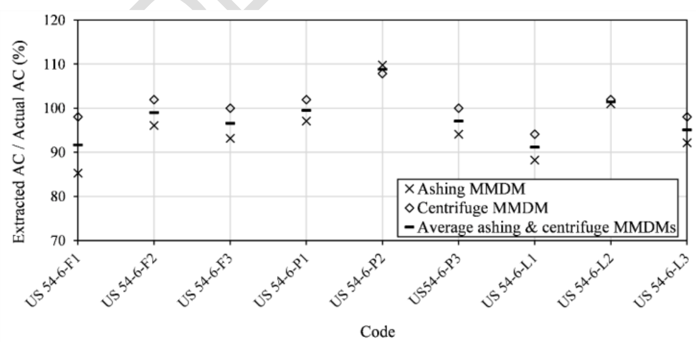


**Fig. 10. Extracted per actual AC percentages for the US 54-6 field, plant, and lab mixes**

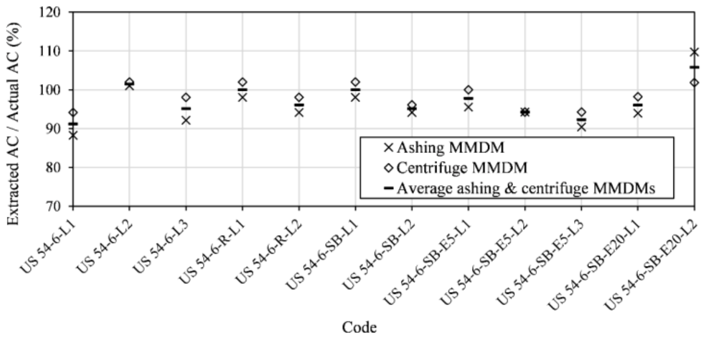


**Fig. 11. Extracted per actual AC percentages for the US 54-6 lab mixes**

Fig. 12 shows the extracted AC per actual AC percentages for the US 63-1 field, plant, and lab mixes using different MMDMs. The extracted AC per actual AC percentages were between 88% and 112%. The centrifuge MMDM demonstrated higher extracted AC than the ashing MMDM for most samples (78% of samples). The highest extracted AC per actual AC percentages were recorded for plant and lab mixes. More interactions between RAP binder and VAB happened in plant and lab mixes when compared to interactions that occurred in field mixes. The fabrication mechanism used in lab mixes and reheating plant mixes to the compaction temperature in the lab increased the interactions between RAP binder and VAB, resulting in higher extracted AC percentages compared to those extracted from field mixes.

**Comment [AA23]:** Is it just this reason?

Fig. 13 depicts the extracted AC per actual AC percentages for the US 63-1 lab mixes using different MMDMs. The extracted AC per actual AC percentages were between 95% and 112%. The centrifuge MMDM showed higher extracted AC when compared to the ashing MMDM for most samples (70% of the samples).

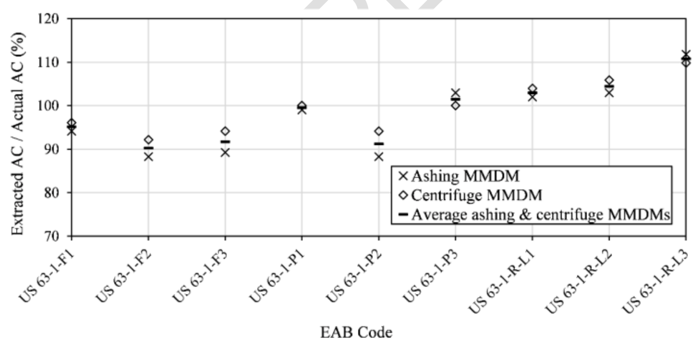


**Fig. 12. Extracted per actual AC percentages for the US 63-1 field, plant, and lab mixes**

For lab mixes containing ECR, it was observed that part of the rubber particles remained with the aggregate, the second part of the particles dissolved in the asphalt binder, and the third part was extracted with the effluent. Fig. 14 illustrates the ECR particles that remained with the aggregate and were extracted with the effluent. The first part of the rubber particles was observed with the aggregate particles during the sieve analysis. The second part of the rubber particles dissolved in the asphalt binders was the reason for enhancing EABs' stiffness and elasticity [8]. The third part was observed in the metal cup with the mineral matter after the filterless centrifuge process.

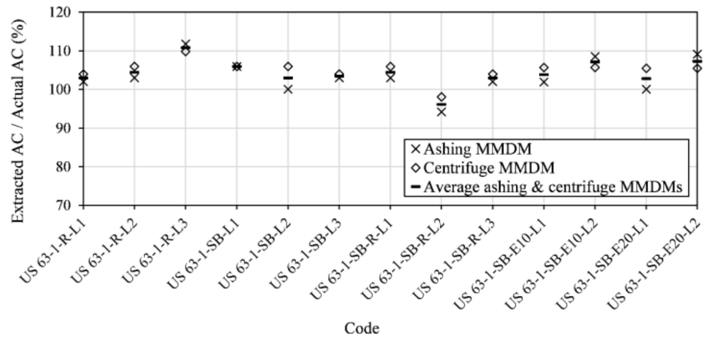


**Fig. 13. Extracted per actual AC percentages for the US 63-1 lab mixes**

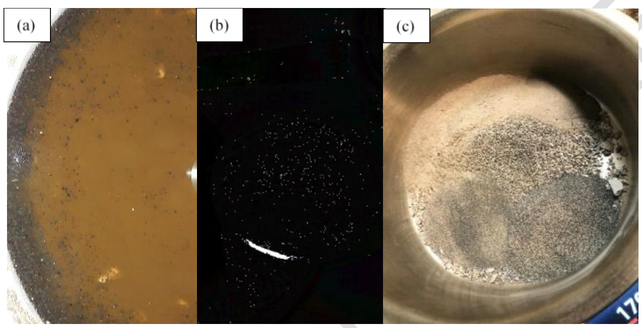


**Fig. 14. The extracted rubber particles; (a) Particles suspended with TCE into the extractor bowl, (b) Particles extracted with the mineral matter after the filterless centrifuge process, (c) Particles remained with the aggregate [5]**

## 3.4. Extraction of Asphalt Binders from Field Mixes Constructed in 2016

The actual AC and extracted AC percentages for field mixes, constructed in 2016, using different MMDMs are illustrated in Fig. 15. The actual AC percentages were between 4.8% and 5.7%. The extracted AC percentages using the ashing MMDM, presented in Fig. 15(a), were found to be between 4.4% and 5.3%. Most samples (87%) had extracted AC percentages less than the actual AC percentages. Therefore, the ashing MMDM underestimated the extracted AC percentages. Using the centrifuge MMDM, presented in Fig. 15(b), increased the accuracy of the extracted AC percentages. The extracted AC percentages using the centrifuge MMDM were between 4.7% and 5.6%. Hence, the centrifuge MMDM presented more accurate extracted AC percentages. The extracted AC percentages were also calculated using the average ashing and centrifuge MMDMs, as represented in Fig. 15(c). The extracted AC percentages using the average ashing and centrifuge MMDMs were between 4.6% and 5.5%.

To show the effect of different MMDMs on the extracted AC percentages, the ANOVA results are presented in Table 8. The p-value (Prob > F) was 0.0028, which was lower than the 0.05

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significance level. Thus, there was a significant difference between the means of the extracted AC percentages using different MMDMs when compared to the mean of the actual AC percentages. To understand which MMDM had a significant difference, the Tukey honestly significant difference (HSD) test was implemented. The connecting letters report using the Tukey HSD test is illustrated in Table 9. Levels not connected by the same letter were significantly different. This reflected that the mean of the extracted AC percentages using the ashing MMDM was significantly different when compared to the means of the actual AC or extracted AC percentages using the centrifuge MMDM.

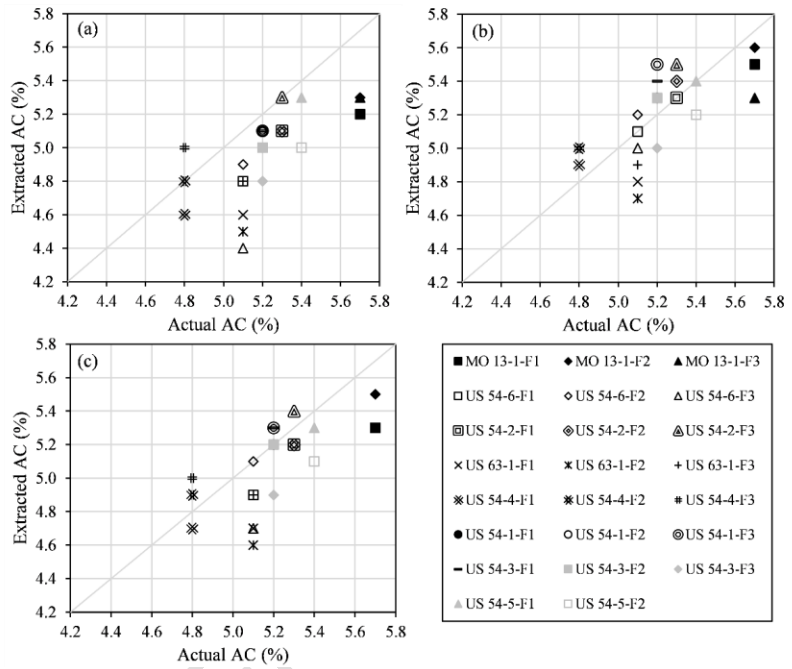


**Fig. 15. Actual versus extracted AC percentages for field mixes constructed in 2016; (a) Ashing MMDM, (b) Centrifuge MMDM, and (c) Average ashing & centrifuge MMDMs**

**Table 8. ANOVA results: actual and extracted AC percentages for field mixes constructed in 2016**

| Source | DF | Sum of Squares | Mean Square | F Ratio | Prob > F |
|---|---|---|---|---|---|
| Method | 3 | 0.978 | 0.326 | 5.067 | 0.0028 |
| Error | 88 | 5.661 | 0.064 | | |
| C. Total | 91 | 6.639 | | | |

**Table 9. Tukey HSD test results**

| Level | | | Mean |
|---|---|---|---|
| Actual AC% | A | | 5.22 |
| Extracted AC% using the centrifuge MMDM | A | | 5.20 |
| Extracted AC% using average ashing & centrifuge MMDMs | A | B | 5.08 |
| Extracted AC% using the ashing MMDM | | B | 4.96 |

*Note: Levels not connected by the same letter are significantly different.*

## 4. CONCLUSION

In this study, asphalt binders were extracted from field, plant, and lab mixes containing reclaimed asphalt pavement (RAP) and/or recycled asphalt shingles. Extraction was performed using a centrifuge extractor, and percentages of asphalt content (AC) were evaluated using two mineral matter determination methods (MMDMs). The extracted AC percentages using ashing MMDM, centrifuge MMDM, and average ashing & centrifuge MMDMs were compared with the actual AC percentages. The effect of the different fabrication methods used in the mixes on the extracted AC percentages was analyzed. The effect of using a soft virgin asphalt binder (VAB) or Evoflex as a recycling agent on the extracted AC percentages was explored. Based on this study, the following points were concluded:

1. The ashing MMDM is sensitive and requires a skilled operator. It could underrate the extracted AC percentages when compared to the actual AC percentages. Using the ashing MMDM, the mineral matter in the total extracted effluent is calculated using a representative sample of 100 ml. Consequently, if this 100 ml sample is non-representative and contains more mineral matter, this underestimates the extracted AC percentages.
2. The centrifuge MMDM showed more accurate extracted AC percentages than those of the ashing MMDM. The centrifuge MMDM does not depend on the skills and accuracy of the operator. Moreover, in the centrifuge MMDM, the total mineral matter in the extracted effluent is estimated and not based on a representative sample like what happens in the ashing MMDM.
3. The use of soft VAB in mixes containing RAP simplified the extraction process when compared to binders extracted from mixes containing a stiffer VAB.
4. Reheating plant mixes in the lab to the compaction temperature increased the interactions between VAB and RAP binder, resulting in increased the extracted AC percentages when compared to extracted AC percentages from the same mixes collected from the field.
5. The use of Evoflex increased the interactions between the RAP binder and VAB, which increased the extracted AC percentages.

**Comment [AA24]:** This is not a conclusion

**Comment [AA25]:** This is bad work that does not comply to the specifications.

**Comment [AA26]:** How much?

**Comment [AA27]:** From where did you get this conclusion? from this paper?

**Comment [AA28]:** In centrifuge MMDM, a sample that represents the entire sample should be taken. Otherwise, the test result is wrong and cannot be relied upon

**Comment [AA29]:** How much? This is self-evident

**COMPETING INTERESTS DISCLAIMER:**

**AUTHORS HAVE DECLARED THAT NO COMPETING INTERESTS EXIST. THE PRODUCTS USED FOR THIS RESEARCH ARE COMMONLY AND PREDOMINANTLY USE PRODUCTS IN OUR AREA OF RESEARCH AND COUNTRY. THERE IS ABSOLUTELY NO CONFLICT OF INTEREST BETWEEN THE AUTHORS AND PRODUCERS OF THE PRODUCTS BECAUSE WE DO NOT INTEND TO USE THESE PRODUCTS AS AN AVENUE FOR ANY**

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**LITIGATION BUT FOR THE ADVANCEMENT OF KNOWLEDGE. ALSO, THE RESEARCH WAS NOT FUNDED BY THE PRODUCING COMPANY RATHER IT WAS FUNDED BY PERSONAL EFFORTS OF THE AUTHORS.**

## REFERENCES


[1] Newcomb DE, Epps JA, Zhou F. Use of RAP & RAS in high binder replacement asphalt mixtures: a synthesis. Special report 213. National Asphalt Pavement Association (NAPA). Lanham, MD, USA; 2016.

[2] West RC and Willis JR. Case studies on successful utilization of reclaimed asphalt pavement and recycled asphalt shingles in asphalt pavements. Final report: NCAT Report 14-06. National Center for Asphalt Technology. Auburn University. Auburn, AL, USA; July 2014.

[3] Copeland A. Reclaimed asphalt pavement in asphalt mixtures: state of the practice. Final report: FHWA-HRT-11-021. Office of Infrastructure Research and Development. Federal Highway Administration. McLean, VA, USA; April 2011.

[4] Wang Z, Wang P, Guo H, Wang X, Li G. Adhesion improvement between RAP and emulsified asphalt by modifying the surface characteristics of RAP. Adv Mater Sci Eng. April 2020;2020:1–10. doi:10.1155/2020/4545971.

[5] Buttlar WG, Abdelrahman M, Majidifard H, Deef-Allah E. Understanding and improving heterogeneous, modern recycled asphalt mixes. Final report: cmr 21-007. University of Missouri. Columbia, Missouri, USA; August 2021.

[6] De Lira RR, Cortes DD, Pasten C. Reclaimed asphalt binder aging and its implications in the management of RAP stockpiles. Constr Build Mater. December 2015;101(1):611–616. doi:10.1016/j.conbuildmat.2015.10.125.

[7] Deef-Allah E, Abdelrahman M. Characterization of asphalt binders extracted from field mixtures containing RAP and/or RAS. World J Adv Res Rev. January 2022;13(01):140–152. doi:10.30574/wjarr.2022.13.1.0729.

[8] Deef-Allah E, Abdelrahman M. Interactions between RAP and virgin asphalt binders in field, plant, and lab mixes. World J Adv Res Rev. January 2022;13(01):231–249. doi:10.30574/wjarr.2022.13.1.0744.

[9] Rodezno C, Julian G. Asphalt binder extraction protocol for determining amount & PG characteristics of binders recovered from asphalt mixtures. Final report: WHRP 0092-16-02. National Center for Asphalt Technology at Auburn University. Auburn, AL, USA; January 2018.

[10] Hemida A, Abdelrahman M, Deef-Allah E. Quantitative evaluation of asphalt binder extraction from hot mix asphalt pavement using ashing and centrifuge methods. Transp Eng. March 2021;3. doi:10.1016/j.treng.2021.100046.

[11] Deef-Allah E, Abdelrahman M. Investigating the relationship between the fatigue cracking resistance and thermal characteristics of asphalt binders extracted from field mixes containing recycled materials. Transp Eng. June 2021;4. doi:10.1016/j.treng.2021.100055.

[12] Mehta Y, Nolan A, Coffey S, DuBois E, Norton A, Reger D et al. High reclaimed asphalt pavement in hot mix asphalt. Final report: FHWA-NJ-2012-005. Rowan University. Glassboro. NJ, USA; July 2012.

[13] Mikhailenko P, Ataeian P, Baaj H. Extraction and recovery of asphalt binder: a literature review. Int J Pavement Res Technol. 2020;13:20–31. doi:10.1007/s42947-019-0081-5.

[14] Stroup-Gardiner M, Nelson JW. Use of n-propyl bromide solvents for extraction and recovery of asphalt cements. J Test Eval. September 2001;29(5):432–441. doi:10.1520/jte12273j.

[15] ASTM D2172 / D2172M-17e1. Standard test methods for quantitative extraction of asphalt binder from asphalt mixtures. ASTM International. West Conshohocken, PA, USA, 2017, doi:10.1520/D2172_D2172M-17E01.


UNDER PEER REVIEW

[16] ASTM D5404 / D5404M-12(2017). Standard practice for recovery of asphalt from solution using the rotary evaporator. ASTM International. West Conshohocken, PA, USA; 2017, doi:10.1520/D5404_D5404M-12R17.
[17] Collins-Garcia H, Tia M, Roque R, Choubane B. Alternative solvent for reducing health and environmental hazards in extracting asphalt: an evaluation. Transp Res Rec. January 2000;1712(1):79–85. doi:10.3141/1712-10.
[18] Li H, Wu Y, Guo Y. Validation of reclaimed shingles asphalt binder extraction and recovery methods. in: Advanced Characterization of Asphalt and Concrete Materials. American Society of Civil Engineers. Reston, VA, USA, 17–23; July 2014, doi:10.1061/9780784478554.003.
[19] Zhou F, Li H, Hu S, Button JW, Epps JA. Characterization and best use of recycled asphalt shingles in hot-mix asphalt. Final report: FHWA/TX-13/0-6614-2. Texas A&M Transportation Institute. College Station, Texas, USA; July 2013.
[20] Burr BL, Davison RR, Glover CJ, Bullin JA. Solvent removal from asphalt. Transp Res Rec. 1990;1269:1–8.
[21] Nösler I, Tanghe T, Soenen H. Evaluation of binder recovery methods and the influence on the properties of polymer modified bitumen. in: E&E Conference. Copenhagen; 2008.
[22] AbuHassan Y, Alin M, Iqbal T, Nazzal M, Abbas AR. Effect of extraction solvents on rheological properties of recovered asphalt binders. J Transp Eng B: Pavements. March 2019;145(1). doi:10.1061/JPEODX.0000096.
[23] Piérard N, Vansteenkiste S, Vanelstraete A. Effect of extraction and recovery procedure on the determination of PmB content and on the properties of the recovered binder. Road Mater Pavement Des. 2010;11(1):251–279. doi:10.1080/14680629.2010.9690334.
[24] Sirin O, Tia M. Investigation of problems in binder extraction from conventional and rubber modified asphalt mixtures. in: 6th RILEM Symposium PTEBM'03. Zurich; 2003, doi: 10.1617/2912143772.025.
[25] MoDOT. Missouri standard specifications for highway construction. MoDOT. Missouri, USA; 2018.

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