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Ahmed Hemida

Magdy Abdelrahman

Missouri University of Science and Technology, [abdelrahmanm@mst.edu](mailto:abdelrahmanm@mst.edu)

Eslam Deef-Allah

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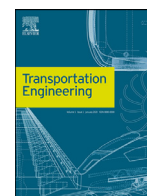
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# Quantitative evaluation of asphalt binder extraction from hot mix asphalt pavement using ashing and centrifuge methods

Ahmed Hemida<sup>a,\*</sup>, Magdy Abdelrahman<sup>b</sup>, Eslam Deef-Allah<sup>a</sup>

<sup>a</sup> Department of Civil, Architectural, and Environmental Engineering, Missouri University of Science and Technology (Missouri S&T), Rolla, MO 65409, USA

<sup>b</sup> Missouri Asphalt Pavement Association (MAPA) Endowed Professor, Department of Civil, Architectural, and Environmental Engineering, Missouri University of Science and Technology (Missouri S&T), Rolla, MO 65409, USA



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## ABSTRACT

Asphalt binder requires more investigation to be accurately and precisely extracted since it is an effective procedure for quality assurance quality control (QA/QC) and subsequent binder characterization. In this research, the authors presented a hands-on experience with binder extraction to deliver recommendations concerning the sensitive steps that may affect the outcomes (extracted binder content,  $P_{be}$  %). Two mineral matter determination methods (ashing and centrifuge) were addressed based on the centrifuge extraction method. Field cores were investigated by comparing the  $P_{be}$  % to the actual binder content,  $P_{ba}$  %. Analysis of variance (ANOVA) and Tukey Post-Hoc statistical analyses, in addition to linear least square regression analysis, were used to show the significance of difference according to 38 variant cores randomly obtained from the field segments (in-service roads) via the first two weeks from their construction dates. Such cores involved reclaimed asphalt pavement (RAP), reclaimed asphalt shingles (RAS), and a wide range of additives. The two extraction methods were compared, concluding that the centrifuge method was highly recommended based on a quantitative evaluation, which delivered the same average  $P_{ba}$  % based on the 38 cores. Furthermore, the centrifuge method provided much saving in the experimental time (almost half the time required for the ashing method). It was found that the ashing outcomes were equal to the centrifuge outcomes when disregarding the ammonium carbonate addition. Thus, it could be recommended to reassess the ammonium carbonate addition as it might excessively compensate for minerals that have not been lost by the ignition oven.

## 1. Introduction

Asphalt binder extraction from hot mix asphalt (HMA) pavement mixes has not been sufficiently addressed in literature [1, 2]. In compliance with Burr et al. (1993), the binder extraction methods still need further research to deliver a good accuracy (measurements proximity to the actual value) and precision (the degree to show the same results of repeated measurements under no changed conditions) of the extracted asphalt content [3]. Literature reported that the most common binder extraction methods are centrifuge extraction (the terminology of *centrifuge extraction* denoted here the initial separation of binder from aggregate) and reflux [2]. However, the centrifuge extraction method is the only standard method potentially followed by representative binder characterization [3]. Due to a literature gap [2, 4], this study focuses on the quantitative evaluation of binder extracted from HMA using the centrifuge extraction method based on two mineral matter determination methods: ashing and centrifuge. This study quantitatively compares the

two methods and provides recommendations concerning the sensitive steps that may affect the extracted binder content ( $P_{be}$  %).

The outcomes of extraction methods are still not reproducible to evaluate the binder content ( $P_b$  %) with respect to quality assurance and quality control (QA/QC) processes [1]. The variation between results could be significant when comparing the  $P_{be}$  % to the actual binder content ( $P_{ba}$  %) or even among the same source's  $P_{be}$  % outcomes. Literature has shown that such a high variation in results could be due to the aggregate absorption of the binder in the aggregate-binder mix, so the binder is not entirely extracted [3]. Subsequently, evaluating  $P_b$  % in addition to measuring extracted binders' physical and chemical properties require representative sample extraction and recovery [1].

The first step of the asphalt binder QA/QC process is accurately and precisely extract the asphalt mix [2]. Such an extraction process is required for asphalt binder evaluation after exposure to the construction process and in-service. The depletion of crude oil, recycled materials, and new resources could contribute to the asphalt industry

\* Corresponding author.

E-mail addresses: [amhnc@mst.edu](mailto:amhnc@mst.edu) (A. Hemida), [m.abdelrahman@mst.edu](mailto:m.abdelrahman@mst.edu) (M. Abdelrahman), [emddkc@mst.edu](mailto:emddkc@mst.edu) (E. Deef-Allah).

[5, 6]. Thus, there is a need for evaluating recycled asphalt materials such as reclaimed asphalt pavement (RAP) [7, 8], so it may be assessed for recycling purposes [1, 2, 7]. An advanced assessment could follow to evaluate the effect of the current asphalt additives (e.g., crumb rubber modifier) [9] and new additives or replacers (e.g., bio-based binders) [5, 10, 11] on the binder after exposing to the field (in-service roads). The binder content determination could be evaluated by several methods, such as solvent-based extraction (extraction and recovery), pycnometer, nuclear asphalt content gage, automatic recordation, [2, 12], and ignition [2, 13]. Nevertheless, the only standard method that allows binder characterization is the extraction and recovery method [2, 14]. Physical and chemical measurements of asphalt binders extracted from asphalt mix require accurate and precise extraction and recovery procedures [3].

As mentioned above, the centrifuge (Method A in ASTM D2172 [15]) and reflux (Method B in ASTM D2172 [15]) methods are the most common binder extraction methods because of their simplicity and prevalence in the industry [2]. Several researchers agreed that the centrifuge technique is the most convenient method for many reasons [2, 8]. It is a cold method, so the binder is significantly less aged than the reflux method (hot method) [2]. The centrifuge extraction technique is safer to operate [2]. Besides, it is the only standard technique followed by representative binder characterization since the binder could be recovered [3]. Therefore, it is the most convenient method used for binder extraction [8]. The centrifuge extraction method was developed in the 1920s [3, 16, 17] but adopted by ASTM D2172 in 1963 [3]. Recovered binder characterization (out of the scope of this study) needs significant research in the future to improve the QA/QC process of the asphalt industry [8]. Solvent-based binder extraction is inevitable if the binder characterization is required [7]. Binder characterization is essential not only for QA/QC processes but also for evaluating the RAP for recycling efforts positively reflected on the environmental benefit and cost reduction of the asphalt industry [7]. Subsequently, further attention to the centrifuge method could be highly beneficial in asphalt sustainability research.

Asphalt binder extraction and recovery method is used by many laboratories [2]. One of them is Missouri Department of Transportation (MoDOT), which provided the investigated core samples in this research. However, these core samples were investigated in the asphalt laboratory at Missouri University of Science and Technology. The same laboratory results could be controlled and consistent due to the low variation controlled by the relative consistency of the human and instrument components. Nevertheless, it is not easy to maintain the variation between different laboratories [3]. Literature listed some reasons that may yield high variations [3]:

- 1- Binder in aggregate-binder mix not completely extracted due to aggregate absorption,
- 2- Solvent aging: interaction between binder and solvent during the recovery process,
- 3- Remaining solvent in recovered binder after the recovery process.

The main concern in asphalt binder extraction is the precision and accuracy of outcomes [3]. In this research, the authors aimed to evaluate the binder extraction process using the most popular method, the centrifuge extraction method, with two mineral matter determination methods [ashing and centrifuge (centrifuge) denoted here is the mineral matter separation from the extract]). Based on the analysis of variance (ANOVA) and Tukey Post-Hoc statistical analyses [18], this research evaluated the accuracy and precision of the  $P_{be}$ % compared to  $P_{ba}$ %. According to hands-on experiences, modifications to the known specification methods were provided in order to address the unclear or unmentioned steps. Subsequently, the most efficient extraction method was recommended for better accuracy and precision.

## 2. Materials

Asphalt core samples were randomly withdrawn from thirteen pavement segments belonging to five different paved roads in Missouri (MO13, MO52, US50, US54, and US63) to evaluate the extraction processes and compare the  $P_{be}$ % to the  $P_{ba}$ %. Binder content was investigated for a total of 38 Superpave mix core samples. Each core set comprised an average of three core samples from random locations in each segment. Some of these sets contained recycled materials (RAP and reclaimed asphalt shingles (RAS)) and a wide range of additives. Others were based on virgin asphalt binder with additives. The aggregate included in all field core samples had a nominal maximum aggregate size (NMAS) of 12.5 mm, except for MO13-1 (NMAS = 9.5 mm). Missouri requirements for Superpave aggregate gradation (SP125, Superpave 12.5-mm NMAS, and SP095, Superpave 9.5-mm NMAS) were followed. All core samples were withdrawn within two weeks after the culmination of the construction process. Eight of the pavement segments were constructed in 2016, and the others were constructed before 2016. Further details are provided in the Results and Discussion section.

## 3. Methods

Method A (centrifuge extraction method) in ASTM D2172 [15] was used to evaluate the extraction process. Two mineral-matter determination methods were used for comparison: ashing and centrifuge. Some modifications to the specifications were followed based on hands-on experience, as discussed later. The interpretations here focused on these modifications. Other than that, the standard procedures in ASTM D2172 were followed. To evaluate the amount of minerals extracted during the binder extraction process, the extracted asphalt-solvent solution was poured into three portions: two ignition dishes and one filterless centrifuge cup. Each ignition dish was filled with approximately 100 ml of the asphalt-solvent solution (called aliquot), and the remaining amount (i.e., all solution except approximately 200-ml aliquot) went through the filterless centrifuge device (with proportioning the withdrawn 200-ml aliquot for the centrifuge method calculations). Therefore, the extraction process could be evaluated based on the average of two ashing outcomes and one centrifuge outcome.

The study evaluated individual core weights of 1755-g average, 1228-g minimum, and 2223-g maximum based on the received field samples. The time required to loosen the asphalt core was 1–2 h at 120 °C. The higher the binder grade, the longer the required time to loosen the core. The core was loosened, as shown in Fig. 1a. It was found that the smaller the separated, coated aggregate particles (Fig. 1b), the easier the binder extraction was achieved due to a higher surface area with interfacial binder-solvent contact. The loose mix was evenly distributed in the extraction bowl (Fig. 1c) to balance the centrifuge extraction process. A sufficient amount of petroleum solvent was added to cover the mix. Trichloroethylene (TCE), a high-purity industrial grade solvent (>99.9%), was used as one of the most common binder-extraction solvents [4]. The TCE was added to cover the mix surface (Fig. 1d). A 52.5-minute duration was applied to keep it consistent for all mixes and to be less than one hour recommended in ASTM D2172. The binder was almost entirely dissolved in TCE. The sample was covered by the lid to minimize the solvent volatilization. It was found that stirring the mix three times in approximately equal intervals (15 min each) was remarkable to expedite the binder extraction process. The TCE thoroughly disintegrated the coated aggregate. However, it was important not to stir the mix in the last 15–20 min, so the minerals had enough time to precipitate. Therefore, the minerals extracted were minimized. The minimization of the extracted minerals was beneficial for two reasons: (1) reduce the error associated with the ashing method calculation and (2) reduce the amount of saturated ammonium carbonate solution (SACS) used to compensate for the minerals lost in the ignition oven. The preparation method and use of SACS are presented later.

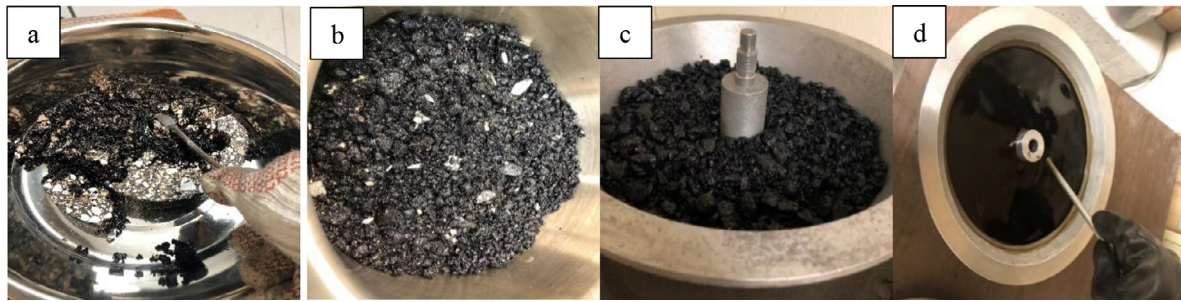


Fig. 1. Loose asphalt mix exposed to the solvent for binder extraction process: (a) loosening the preheated HMA pavement core, (b) mix after being loosened, (c) even distribution of asphalt mix, and (d) asphalt mix soaked in TCE.



Fig. 2. Centrifuge extraction apparatus: extraction of asphalt-solvent solution.

An oven-dried (all oven-drying processes conducted at 110 °C in this study) filter ring was put between the bowl and its lid with the thoroughly tightened setscrew. Tightening the setscrew properly minimized the minerals extracted. Fig. 2 shows the solution extraction using the centrifuge extraction apparatus. The speed mentioned in ASTM D2172—up to 3600 rpm—was found to be too high for the apparatus capabilities, particularly for the large samples (higher than 2000-g). The authors applied several trials on two centrifuge extraction apparatuses with no stabilization, for both, at that high speed. Instability to that degree could lead to unrestrained vibration associated with excessive minerals extracted. Subsequently, it is recommended to use a speed ranging 1500–2000 rpm because it is sufficient to extract approximately the entire solution. It was found that beginning with a low speed to centrifuge the solution into the beaker slowly was efficient because it provided the TCE time to wash the aggregate from the binder, then it was appropriate to gradually increase the speed with the last droplets. Such a process was repeated for the subsequent washes. Extra TCE (approx. 200 ml) was added from the lid holes with a waiting time of  $4 \pm 1$  min before centrifuging to ensure the solvent washing process was efficient. In all cases, such an operation had to be repeated until it reached a light straw color of extracted TCE [15], indicating pure aggregate in the bowl. It was noticed and noted that generally three solvent additions were not enough to wash the aggregate when testing old samples thoroughly. The authors found that a range of 6–10 200-ml solvent washes were sufficient for the field core samples with an average of 8 washes concerning the 38 investigated samples.

### 3.1. Ashing method

One of the most critical aspects of the ashing method is making the solution homogeneous and equally distributed in the 2000-ml longitudinal beaker because each aliquot only represents approximately 100-ml TCE, out of at least 1800 ml. Thus, if such a small sample was not representative, it would yield a significant  $P_{be}$ % error. After a lengthy investigation and several trials, it was found that the over stirring might take the minerals to the top of the beaker. Thus, a higher concentration of minerals might be attributed to the aliquot. Such mineral distribution might end up with unreal ash representation, which leads to a very low  $P_{be}$ % compared to the  $P_{ba}$ %. That is why it is recommended for potential efficiency that researchers use a moderate stirring with a thin agitator (e.g., glass) with no direct pour after the stirring process but followed by approximately 5 min to ensure a homogeneous distribution. The results were much rational in such a case, as discussed further in the Results and Discussion section. Since the used beaker had a 2000-ml capacity, the extracted solution might exceed such amount; thus, another beaker might be used for the remaining portion of the solution. In that case, all of the solution had to be decanted through the two beakers multiple times (3–5 times) to ensure an even distribution of minerals in the two beakers. Simultaneously, two oven-dried ignition dishes were filled with aliquots after putting them in a desiccator for 10 min. Such duration was required to cool them and acquire a stabilized weight. Typically, the beaker had a 20-ml accuracy, and it was found that using the sense to take the reading is recommended for more accurate results when located between two signs. The remaining solution went through the filterless centrifuge device (for centrifuge method), as discussed later.

#### 3.1.1. Mineral matter extraction

The filled ignition dishes with aliquots were heated on hot plates to an adequate temperature (Fig. 3a). Due to the TCE comprised in the solution, which is flammable, this process had to be completed in a well-ventilated fume hood, and it thusly minimized the solution boiling. The TCE evaporated in a short period (probably 25% of the time, if the hot plate was left in the open air). Put simply, the suction process rids the solution of the evaporated TCE quickly; thus, there was no opportunity for TCE to catch fire in the fume hood atmosphere even at extreme temperature exposure (275–300 °C on the hot plate surface, much less in the ignition dish). In addition to minerals, the liquid binder remained with almost no TCE. Subsequently, the ignition dish, including the extracted liquid binder plus minerals, was exposed to a 600 °C ignition oven until yielding the mineral ash (Fig. 3b). It was found that 75 min was enough time to ensure no liquid binder remained. Due to the ignition dishes' heat level, they were kept at room temperature for about 10 min to be easily handled, followed by 10 additional minutes in the desiccator to rid them of external moisture (Fig. 3c). The SACS was uniformly distributed on the ash surface based on 5 ml per gram of ash after acquiring ignition dish weights; it was left for 1 hour to be digested [15], followed by oven-drying for 1–1.5 h. The ignition dishes were cooled down in the desiccator for 10 min before acquiring their weights.

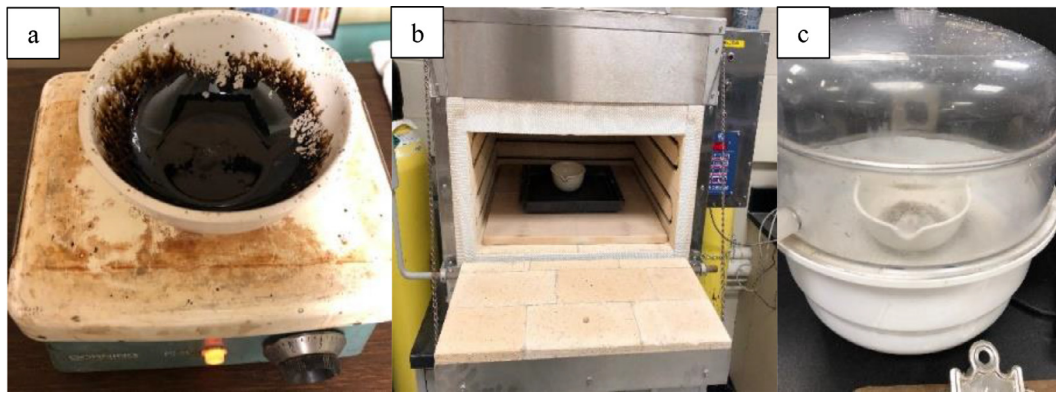


Fig. 3. Mineral matter determination: (a) ignition dish with aliquot heated on a hot plate, (b) ignition dish in an ignition oven after removing TCE, and (c) ignition dish in a desiccator to cool after removing binder.



Fig. 4. Filterless centrifuge device.

### 3.1.2. Saturated ammonium carbonate solution preparation

The requirements to prepare approximately 100 ml of the SACS were 100-g ammonium carbonate and deionized water. Ammonium carbonate was well-grounded until acquiring a high fineness due to the increased surface area as it facilitated its dissolution in water. The 100-g ground ammonium carbonate was poured in an empty, clean beaker, and it was followed by deionized water to acquire a final volume of 100 ml. A magnetic stirring hot plate was used to stir the solution at 20 °C (around room temperature, i.e., storing and testing temperature) until reaching a homogeneous solution with all particles dissolved in the deionized water to ensure a saturated solution of ammonium carbonate [19]. The ammonium carbonate crystals were slowly dissolved in the deionized water. Thus, the desired solution took a long time to achieve. Ultimately, it was no problem if some of the ammonium carbonate crystals precipitated on the container's bottom. Such precipitation indicated reaching the saturation status.

### 3.2. Centrifuge method

The remaining binder-solvent solution went through the filterless centrifuge device (Fig. 4). This device was extremely efficient in splitting the minerals from the binder-solvent solution. The 2000-ml beaker(s) were washed using extra clean TCE across the filterless centrifuge funnel to ensure that all minerals were embedded in the filterless centrifuge cup. It was found that it was bet to gradually decant the solution through the funnel to prevent blockage. Ultimately,

the product extracted, which was a pure binder-solvent solution (no minerals involved), was ready for the binder recovery process (that is out of the scope of this study). According to ASTM D1856 [20] and AASHTO T170 [21], one should end the recovery process within eight hours after blending asphalt mix with the solvent to minimize the undesired binder-solvent interaction. By contrast, after oven-drying the filterless centrifuge cup for approximately 1 hour, minerals' net weight was obtained. The weighing process was implemented after cooling down (about 10 min in the desiccator). This process is based on minerals' overall weight with the extract (unlike the ashing method); hence, the measured  $P_b\%$  error is minimized. The results and discussion compared the two methods in a quantitative analysis based on the statistical significance of difference using ANOVA, Tukey Post-Hoc statistical analyses [18], and least squares regression analysis.

## 4. Results and discussion

Table 1 presents data for the 38 pavement core samples (i.e., 13 core sets). The core code was recognized as follows: the first two letters represented the road designation, either MO or US, followed by the road and segment numbers, respectively, hence the core number. The provided information included the sample weight, RAP%, RAS% (if any), virgin contract grade, virgin and total  $P_{ba}\%$ , and  $P_{be}\%$  by both, ashing method, an average of two (labeled  $P_{bea}\%$ ), and centrifuge method (labeled  $P_{bec}\%$ ). It was noticed that the  $P_{ba}\%$  was greater than the  $P_{be}\%$  in some core sets. This was most likely yielded from the difference between the actual binder content, which was the HMA-designed binder content according to the job mix formula (JMF), and the binder content conducted in the field.

Fig. 5 summarizes the minimum, maximum, and average binder content ( $P_b\%$ ) based on the actual binder, the extracted binder by the ashing method, and the extracted binder by the centrifuge method. It was noticed that the average  $P_{ba}\%$  was equal to the average  $P_{bec}\%$  (i.e., 5.3% both). However, the ashing method yielded a  $P_{be}\%$  of 5.0%, resulting in an average of 0.3% lower than the average of either the  $P_{bec}\%$  or the  $P_{ba}\%$ , according to the 38 investigated pavement core samples.

### 4.1. ANOVA and Tukey Post-Hoc statistical analysis

One-way ANOVA was used to evaluate the significance of difference among the compared binder contents, as presented in Table 2. The three levels of the same independent variable treatment are the  $P_{ba}\%$ ,  $P_{bea}\%$ , and  $P_{bec}\%$ . Accordingly, the Tukey Post-Hoc analysis was provided to show the statistical significance between  $P_{ba}\%$  and  $P_{be}\%$  by ashing and centrifuge methods, as presented in Fig. 6. ANOVA analysis was established based on  $\alpha = 0.05$ . The sample US54-5 was avoided from the ANOVA and Tukey statistical analyses due to the limited availability, which implied two values.

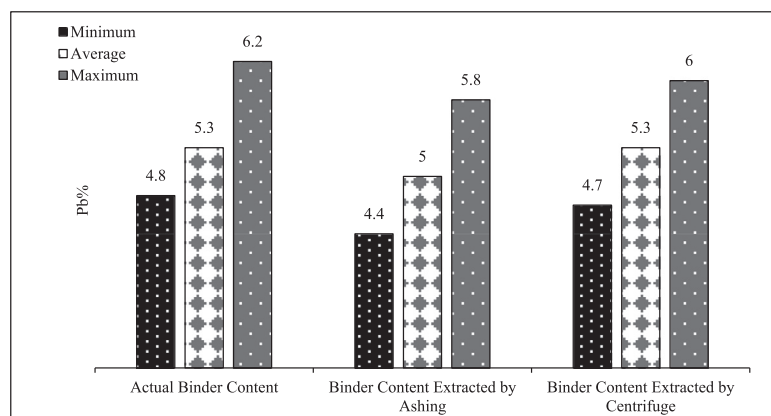
**Table 1**  
HMA core samples data and extracted binder content ( $P_{be}$ %) by ashing and centrifuge methods.

Set	Sample Label <sup>1</sup>	Construction Year	Sample Weight	RAP%	RAS%	Virgin PG	Contract grade	Additive <sup>3</sup>	Virgin AC%	Total $P_{ba}$ %	$P_{be}$ %	
											Ashing (Avg)	Centrifuge
1	MO13-1(1)	2016	1228	17	0	64-22H	70-22	A: 0.5%	4.4	5.7	5.3	5.3
	MO13-1(2)		1315								5.2	5.5
	MO13-1(3)		1255								5.3	5.6
2	MO52-1(1)	2010	1677	0	34	64-22	NA <sup>2</sup>	B: 1.5% C: 0.8%	3.7	4.8	4.9	5.0
	MO52-1(2)		1720								4.9	5.2
	MO52-1(3)		1548								4.9	5.1
3	US50-1(1)	2011	1630	25	0	64-22	NA	B: 1.5% C: 1%	3.8	5	4.9	5.0
	US50-1(2)		1760								5.0	5.2
	US50-1(3)		1550								4.6	4.9
4	US54-1 (1)	2016	2066	0	33	58-28	NA	D: 2.5% E: 3.5% A: 1.5%	3.6	5.2	5.1	5.3
	US54-1(2)		2098								5.0	5.3
	US54-1(3)		1905								5.1	5.5
5	US54-2(1)	2016	1842	33	0	58-28	NA	A: 1%	3.6	5.3	5.3	5.5
	US54-2(2)		1970								5.1	5.4
	US54-2(3)		1973								5.1	5.3
6	US54-3(1)	2016	1867	18	15	58-28	NA	A: 1%	3.6	5.2	4.8	5
	US54-3(2)		2223								5.0	5.3
	US54-3(3)		2115								5.1	5.4
7	US54-4 (1)	2016	2186	35	0	64-22H	NA	E: 3% A: 1%	3.2	4.8	4.8	5
	US54-4(2)		2205								4.6	4.9
	US54-4(3)		1841								5.0	5
8	US54-5(1)	2016	2044	0	0	64-22H	64-22	A: 1%	5.4	5.4	5.3	5.4
	US54-5(2)		2036								5.0	5.2
9	US54-6(1)	2016	1782	31	0	58-28	70-22	A: 1%	3.6	5.1	4.4	5
	US54-6(2)		1490								4.9	5.2
	US54-6(3)		1721								4.8	5.1
10	US54-7(1)	2003	1976	0	0	64-22	NA	F: 0.25%	6.2	6.2	5.8	5.9
	US54-7(2)		1655								5.8	6.0
	US54-7(3)		1698								5.7	5.8
11	US54-8(1)	2006	1576	9	0	70-22	NA	C: 0.5%	5.1	5.6	5.2	5.4
	US54-8(2)		1695								5.3	5.5
	US54-8(3)		1600								5.6	5.7
12	US63-1(1)	2016	1633	35	0	58-28	70-22	G: 0.5% H: 1.75%	3.4	5.1	4.8	4.9
	US63-1(2)		1664								4.5	4.7
	US63-1(3)		1607								4.6	4.8
13	US63-2(1)	2008	1433	20	10	64-22	NA	B: 1.5% C: 0.5%	4.1	5.6	4.6	5.4
	US63-2(2)		1707								4.9	5.0
	US63-2(3)		1399								5.1	5.3

<sup>1</sup> All pavement core samples denoted Superpave bituminous mixtures of SP125 (NMAS = 12.5 mm) except for MO13-1, which included SP095 (NMAS = 9.5 mm).

<sup>2</sup> NA: Not Available.

<sup>3</sup> A: Anti-stripping agent (Morelife T280); B: Bag house fines; C: Anti-stripping agent (AD-here HP Plus); D: Anti-stripping agent (IPC-70); E: Warm mix additive (PC 2106); F: Anti-stripping agent (LOF 65-00LS1); G: Warm-mix additive (Evotherm); H: Rejuvenator (EvoFlex CA).

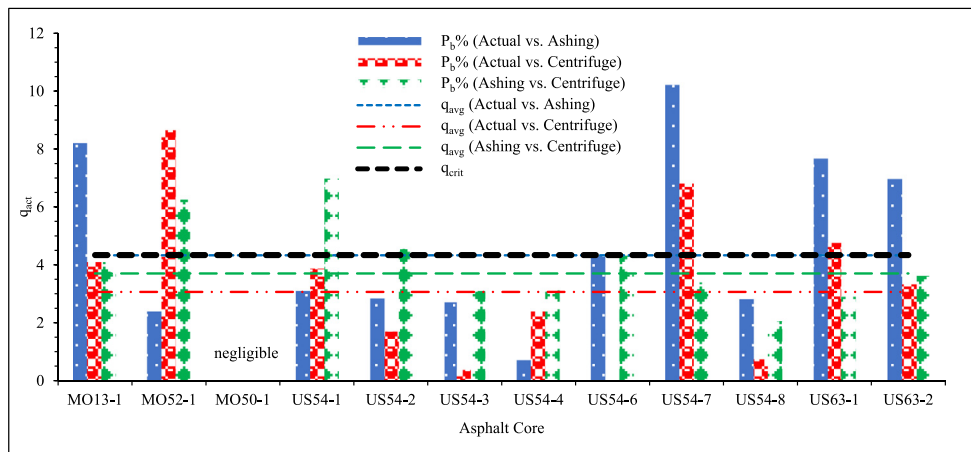


**Fig. 5.** Minimum, average, and maximum binder content ( $P_b$ %) based on the actual binder, extracted by the ashing method, and extracted by the centrifuge method.

**Table 2**  
One-way ANOVA results for actual and extracted (by ashing and centrifuge) binder contents.

Sample	Asphalt Binder Replacement			Source of Variation Between Groups			Within Groups			F	P-value	F <sub>crit</sub>
	RAP%	RAS%	sum	SS <sup>1</sup>	DF <sup>2</sup>	MS <sup>3</sup>	SS <sup>1</sup>	DF <sup>2</sup>	MS <sup>3</sup>			
US54-7	0	0	0	0.315	2	0.158	0.035	6	0.006	27	0.001	5.1
MO52-1	0	34	34	0.144	2	0.072	0.022	6	0.072	19.9	0.002	5.1
MO13-1	17	0	17	0.327	2	0.163	0.058	6	0.010	16.8	0.003	5.1
US63-1	35	0	35	0.357	2	0.179	0.072	6	0.012	15	0.005	5.1
US54-1	0	33	33	0.136	2	0.068	0.033	6	0.006	12.2	0.008	5.1
US63-2	20	10	30	0.882	2	0.441	0.218	6	0.036	12.1	0.008	5.1
US54-6	31	0	31	0.376	2	0.188	0.182	6	0.030	6.2	0.035	5.1
US54-2	33	0	33	0.109	2	0.054	0.062	6	0.010	5.3	0.047	5.1
US54-3	18	15	33	0.127	2	0.063	0.133	6	0.022	2.9	0.135	5.1
US54-4	35	0	35	0.077	2	0.039	0.087	6	0.014	2.7	0.148	5.1
US54-8	9	0	9	0.101	2	0.050	0.142	6	0.024	2.1	0.2	5.1
US50-1	25	0	25	0.082	2	0.041	0.128	6	0.021	1.9	0.228	5.1

<sup>1</sup> Sum of squares.  
<sup>2</sup> Degree of freedom.  
<sup>3</sup> Mean square.



**Fig. 6.** Tukey analysis results of comparative actual and extracted binder contents.

It was noticed that the statistical significance did not rely on the binder condition (virgin or modified). For instance, the highest statistical significance was attributed to a virgin asphalt mix (US54-7 with  $F = 27$ ). Additionally, the F statistic rank did not rely on the RAP/RAS concentrations. Table 2 was built based on the descending order of the F statistic. As shown in the last four rows in Table 2, the US54-3, US54-4, US54-8, and US50-1 core samples provided the lowest significance (less than F critical) with no distinction of the modification impact on the variation between the  $P_{be}$ % and  $P_{ba}$ %. This analysis demonstrated the efficiency of the used solvent (TCE) with the virgin asphalt cement, RAP, or RAS, including the used additives.

The Tukey tests present three pairwise comparisons:  $P_{ba}$ % against  $P_{bea}$ %,  $P_{ba}$ % against  $P_{bec}$ %, and  $P_{bea}$ % against  $P_{bec}$ %. In compliance with the one-way ANOVA analysis, the Tukey analysis revealed no significant difference of all pairwise comparisons of US54-3, US54-4, US54-8, and US50-1. Additionally, no significant difference of all pairwise comparisons of US54-6 was observed despite the significant difference shown by the ANOVA analysis. Four asphalt cores resulted in a statistically significant difference between  $P_{ba}$ % and  $P_{bea}$ %, which were MO13-1 ( $q_{act} = 8.198$ ), US54-7 ( $q_{act} = 10.205$ ), US63-1 ( $q_{act} = 7.660$ ), and US63-2 ( $q_{act} = 6.961$ ); whereas  $q_{crit} = 4.339$ . Three cores resulted in a statistically significant difference between  $P_{ba}$ % and  $P_{bec}$ %, which were MO52-1 ( $q_{act} = 8.647$ ), US54-7 ( $q_{act} = 6.803$ ), and US63-1 ( $q_{act} = 4.754$ ). Alternatively, the statistical significance between ashing and centrifuge methods resulted in three core samples, which were MO52-1 ( $q_{act} = 6.245$ ), US54-1 ( $q_{act} = 6.971$ ), and US54-2 ( $q_{act} = 4.556$ ).

With monitoring the pairwise comparisons above the dotted line ( $q_{crit}$ ) in Fig. 6, one could notice relatively higher significant differences of  $P_b$ % (actual vs. ashing) compared to  $P_b$ % (actual vs. centrifuge) and  $P_b$ % (ashing vs. centrifuge). Likewise, the  $q_{act}$  average lines (labeled  $q_{avg}$  in Fig. 6) indicated a significant difference among the three pairwise comparisons. Upon which, the  $q_{avg}$ s of  $P_b$ % (Actual vs. Ashing),  $P_b$ % (Actual vs. Centrifuge), and  $P_b$ % (Ashing vs. Centrifuge) were located at 4.330, 3.063, and 3.704, respectively. These averages could explain the overall rank of the significant difference of the three pairwise comparisons, indicating a higher significant difference of  $P_b$ % (Actual vs. Ashing), followed by  $P_b$ % (Ashing vs. Centrifuge), then  $P_b$ % (Actual vs. Centrifuge). Such statistical analysis illustrated higher credibility towards the centrifuge method than the ashing method and less significant difference between centrifuge outcomes and actual binder contents.

4.2. Least squares regression analysis

For more clarity, a linear least squares regression for ashing  $P_{be}$ % results and centrifuge  $P_{be}$ % results compared to  $P_{ba}$ % simply depict to what extent each extraction method outcomes were close to the  $P_{ba}$ %. Fig. 7 illustrates such a comparison using the 38 core sample size. The line of best fit from the  $P_{bec}$ % was closer to the identity line, compared to the ashing  $P_{be}$ %. Such a comparison indicated high reliability associated with the centrifuge method by considering that the ashing results were enhanced by some adjustments made to the specs to optimize its attributed outcomes.

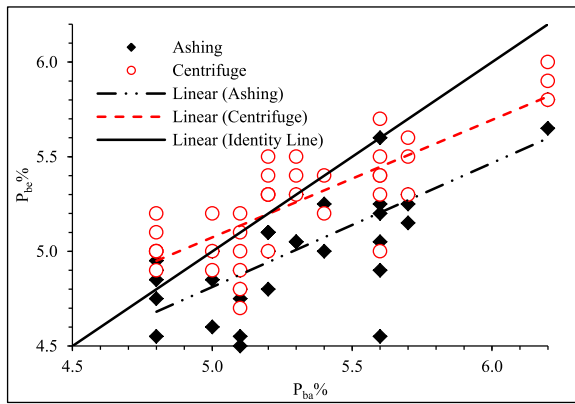


Fig. 7. Linear least-squares regression: extracted binder content ( $P_{be}$ %) by ashing and centrifuge compared to actual binder content ( $P_{ba}$ %).

## 5. Recommendations

The authors of this research believe that Method A (centrifuge extraction method) in ASTM D2172 is highly efficient, yielding extraction for approximately 100% of the binder. Additionally, it is the only standard method that could be followed by the extracted binder characterization. However, the  $P_{be}$ % calculations are most likely related to the determination of minerals extracted with the asphalt-solvent solution, as clarified in this paper by comparing the ashing method to the centrifuge method. Asphalt binder extraction needs hands-on experimentation to provide consistent extraction outcomes. Thus, the variation of results could be minimized when comparing the  $P_{be}$ % to the  $P_{ba}$ %, or even among the  $P_{be}$ % (by using different extraction methods) from the same asphalt mix source. The ashing method is not highly recommended. This is due to its dependency on a small amount of binder-solvent solution (100-ml aliquot) out of 1800 ml, at the very least (i.e., a maximum of 5% by the solution volume). Therefore, the calculation error had more potential to occur. The centrifuge method is based on evaluating the minerals in the overall binder-solvent solution. Thus, the calculation error is minimized to a high extent. Furthermore, the centrifuge method provided savings in experimental time (about 3.5 h) compared to the ashing method (about 7 h) from the time the sample is loosened and ready for the solvent addition. Ashing outcomes ( $P_{bea}$ %) were equal to the centrifuge outcomes ( $P_{bec}$ %) in the case of disregarding the addition of SACS. Thus, it is recommended to reassess the SACS addition because it might excessively compensate for minerals that have not been lost by the ignition oven.

## 6. Conclusion

This study quantitatively evaluated the binder extraction from HMA by two binder extraction methods: ashing and centrifuge. The evaluation was conducted using one-way ANOVA, Tukey, and least square regression analyses. The following points conclude the observations made in this study:

- Among the three investigated levels ( $P_{ba}$ %,  $P_{bea}$ %, and  $P_{bec}$ %), using one-way ANOVA to analyze twelve asphalt core sets at  $\alpha = 0.05$ , eight sets resulted in a significant difference ( $F$ -statistic  $> F_{crit}$ ). The other four sets resulted in no significant difference ( $F$ -statistic  $< F_{crit}$ ). The study did not distinguish a significant influence of RAP, RAS, or additive concentrations on the extraction outcomes by either ashing or centrifuge methods.
- Tukey analysis (which provided pairwise comparisons) distinguished the preference of the centrifuge method ( $q_{act}$  average of all core sets = 3.063 when comparing  $P_{ba}$ % to  $P_{bec}$ %) against the ashing method ( $q_{act}$  average of all core sets = 4.330 when comparing  $P_{ba}$ % to  $P_{bea}$ %).

- Although neither ashing nor centrifuge methods resulted in identical outcomes against  $P_{ba}$ %, the centrifuge method yielded an average  $P_{be}$ % of 5.3%, which complied with the average  $P_{ba}$ % (5.3%)
- Ashing method yielded an average  $P_{be}$ % of 5.0%, indicating an average of 0.3% less than the  $P_{ba}$ %.
- Ashing outcomes ( $P_{bea}$ %) were equal to the centrifuge outcomes ( $P_{bec}$ %) in the case of disregarding the addition of SACS. Thus, it is recommended to reassess the SACS addition because it might excessively compensate for minerals that have not been lost by the ignition oven.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## CRedit authorship contribution statement

**Ahmed Hemida:** Conceptualization, Methodology, Validation, Formal analysis, Investigation, Data curation, Software, Writing - original draft, Writing - review & editing, Visualization. **Magdy Abdelrahman:** Conceptualization, Methodology, Validation, Resources, Data curation, Writing - review & editing, Visualization, Supervision. **Eslam Deef-Allah:** Investigation.

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