05 Oct 2020

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Word Count: 4797 words + 3 tables (250 words per table) = 5547 words

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ABSTRACT

Asphalt binder requires more investigation to be accurately and precisely extracted since it is a significant procedure for quality control quality assurance (QC/QA) and subsequent binder characterization. In this research, the authors provided a hands-on experience with binder extraction to deliver recommendations concerning the sensitive steps that may affect the outcomes (extracted binder content, P_{be}%). Based on the extraction by the centrifuge method, two mineral matter determination methods (ashing and centrifuge) were addressed. Field cores were investigated with 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 the construction date. Such cores involved reclaimed asphalt pavement (RAP), reclaimed asphalt shingles (RAS), and a wide range of additives. The two extraction methods were compared with 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 with disregarding the ammonium carbonate addition. Thus, it could be recommended to reassess the ammonium carbonate addition as it might excessively compensate for fake minerals that have not been lost by the ignition oven.

Keywords: ANOVA, Asphalt Binder Extraction, Quality Assurance, Quality Control, RAP, RAS

1. INTRODUCTION

Asphalt binder extraction from hot mix asphalt (HMA) pavement mixes has not been sufficiently addressed in literature. The outcomes of extraction methods are still not reproducible to accurately and precisely evaluate the binder content (P_b%) with respect to quality control and quality assurance (QC/QA) process (*1*). The variation between results could be significant when comparing the extracted binder content (P_{be}%) to the actual binder content (P_{ba}%), or even among the P_{be}% outcomes from the same source. Literature has shown that such a high variation in results could be because of binder in the aggregate-binder mix not completely extracted due to aggregate absorption (*2*). Subsequently, evaluating P_b% in addition to measuring extracted binder physical and chemical properties requires representative sample extraction and recovery (*1*).

The first step for the asphalt binder QC/QA process is a precise and accurate extraction from the asphalt mix (*3*). Such an extraction process is significantly required for asphalt binder evaluation after exposing to the construction process and being in-service. Besides, due to the diminishment in the crude oil, recycled materials, and new resources could provide contributions to the sustainable development of the asphalt industry (*4; 5*). Thus, there is a need for evaluating recycled asphalt materials such as reclaimed asphalt pavement (RAP) (*6; 7*), so it could be accurately and precisely assessed for recycling purposes (*1; 3; 6; 8*). An advanced assessment could be followed to evaluate the effect of the current asphalt additives such as crumb rubber modifier (*9*) and new additives or replacers such as bio-based binders (*4; 10; 11*) on the binder after exposing the field (in-service roads). The binder content determination could be evaluated by several methods, such as solvent extraction (extraction and recovery), pycnometer, nuclear asphalt content gauge, automatic recordation, (*3; 12*), and the ignition method (*3; 13*). Nevertheless, the only method that allows binder characterization (is the extraction and recovery method (*3; 14*). Physical and chemical measurements of asphalt binder extracted from asphalt mix require accurate and precise extraction and recovery procedure (*2*). The extraction by centrifuge method was found to be safe (based on cold extraction) and significantly effective (*3*). Therefore, it is the most common method used for binder extraction (*7*). Centrifuge method was developed in the 1920s (*2; 15; 16*) but adopted by ASTM D2172 as method A in 1963 (*2*). Another extraction method is the reflux method recognized in ASTM D2172 as method B (ASTM D2172 (*17*)). However, it is not compatible if the recovered asphalt characterization is required (*2*).

For the QC/QA process in addition to asphalt research, the asphalt binder extraction and recovery are used by many laboratories (*3*). One of them is Missouri Department of Transportation (MoDOT) who 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 results in the same laboratory could be controlled and consistent due to the low variation controlled by the relatively human being and instrument consistency. Nevertheless, it is not easy to control the variation between different laboratories (*2*). Literature listed some reasons that may result in such a high variation (*2*):

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.

As mentioned above, the main concern in asphalt binder extraction is the precision and accuracy of outcomes that may not exist (*2*). In this research, the authors aimed to evaluate the binder extraction process using the most popular method, which is the centrifuge method, with two mineral matter determination methods (ashing and centrifuge). Based on the analysis of variance (ANOVA) and Tukey Post-Hoc statistical analyses (*18*), this research may provide the evaluation of the accuracy and precision of the P_{be}% compared to the P_{ba}%. According to hands-on experience, modifications to the known specification methods have been provided in order to address the unclear or unmentioned steps. Therefore, the most efficient extraction method could be recommended for better accuracy and precision.

2. MATERIALS AND METHODS

2.1 Materials

Asphalt core samples were randomly withdrawn from thirteen pavement segments belonged to five different pavement roads in Missouri (MO13, MO52, US50, US54, and US63) to evaluate the extraction process, and compare the P_{be}% to the P_{ba}%. Binder content was investigated for a total of 38 pavement core samples (**Figure 1**). 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). All core samples were withdrawn within two weeks after the construction process. Seven 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.

Figure 1. The 38 investigated HMA pavement core samples

2.2 Methods

Method A (Centrifuge Extraction) in ASTM D2172 (*17*) 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. In order 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, we could evaluate the extraction process based on the average of two ashing outcomes and one centrifuge outcome.

2.1.1 Extracted Binder

Although the ASTM D2172 requires a minimum sample mass of 1500 g in the case of 12.5-mm NMAS and 1000 g in the case of 9.5-mm NMAS (*17*), the study evaluated individual core weights of 1755 g average, 1228 g minimum, and 2223 g maximum based on the received field samples. It was found that the time required to loosen the HMA core sample was in a range of 1–2 hours at 120℃, mainly based on the contract grade. In other words, the higher the binder grade was, the longer the required time was to loosen the HMA core. This period was also sufficient to get a constant mass. It was found that the smaller the separated coated aggregate particles we got (without aggregate collapse) (**Figure 2a**), the easier the binder extraction was achieved due to a higher surface area with interfacial binder-solvent contact. The empty extraction bowl was weighed to the nearest 0.1 g, hence filled with the loose asphalt mix and evenly distributed (**Figure 2b**) in order to balance the centrifuge process. An amount of about a 1200-ml efficient petroleum solvent. Trichloroethylene (TCE) was used in this study as one of the most common binder-extraction solvents (*19*). The TCE was added to cover the mix surface (**Figure 2c**) for about 52.5 min (not

over 1 hour recommended in ASTM D2172), so the binder was about to be entirely dissolved in TCE. Additionally, the bowl was located down in the centrifuge extraction apparatus and covered with the lid to minimize the solvent volatilization. It was found that stirring the asphalt mix for about three times in approximately equal time intervals (15 min each), starting from the addition of the sample in the bowl, was remarkably efficient. The TCE would thoroughly permeate the coated aggregate. However, it was figured out not to stir the mix in the last 15–20 min, so minerals had enough time to settle down. Therefore, the mineral extraction with the binder-solvent solution would be minimized. This minimization was beneficial for two aspects: (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 during the ignition process. The preparation method of SACS is presented later.

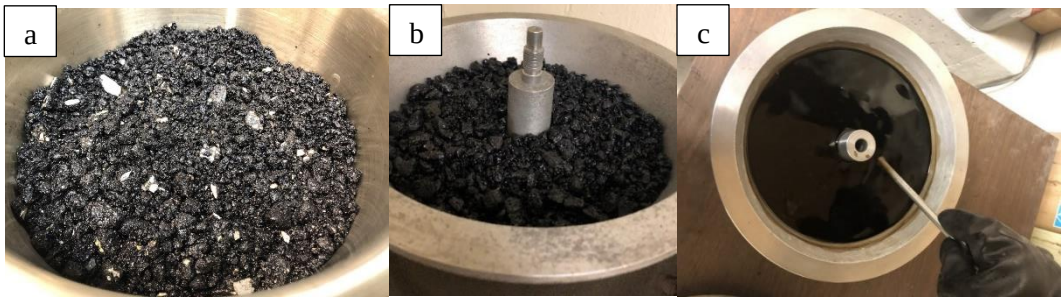

Figure 2. Loose asphalt mix exposed to the solvent for binder extraction process: (a) mix after being loosened, (b) even distribution of asphalt mix, and (c) asphalt mix soaked in TCE

The clean filter ring was weighed to the nearest 0.1 g after being heated in the oven at 110℃, hence put between the bowl and its lid with thoroughly tightening the setscrew. Tightening the setscrew, properly, would also minimize the minerals associated with the extracted solution. The centrifuge extraction apparatus was gradually rotated until reaching a sufficient speed to extract all asphalt-solvent solution (**Figure 3**). The speed mentioned in ASTM D2172, which was up to 3600 rpm, was found to be too high for the device capabilities, particularly with large samples. The authors applied several trials on two centrifuge devices with no stabilization, for any, at such a high rotation speed (3600 rpm). Such a non-stabilization could lead to a high vibration associated with excessive minerals extracted with the asphalt-solvent solution from the centrifuge extraction apparatus. Subsequently, it was recommended to use a rotation speed in a range of 1500–2000 rpm instead, sufficient to approximately extracting the entire solution. Likewise, it was found that beginning with a low rotation speed in order to centrifuge the solution into the beaker slowly was efficient as we gave a chance for TCE to remarkably wash the aggregate from the binder, then gradually increase the speed with last droplets. Such a process was repeated for the subsequent washes. In order to ensure no remaining binder in the aggregate, extra TCE (approx. 200 ml), was added from the lid holes with waiting 4 ± 1 min before centrifuge rotation to ensure an efficient role of the solvent washing process. In all cases, such an operation had to be repeated until reaching out to a light straw color of extracted TCE, indicating pure aggregate in the bowl. It was noticed that mainly three solvent additions could not be enough to thoroughly wash aggregate and extract close to 100% binder, mainly when dealing with old samples (in-service roads). The authors found that a range of 6–10 200-ml solvent washes could be sufficient with field core samples with an average of 8 washes concerning the 38 investigated samples.

2.1.2 Ashing Method

One of the most critical aspects of the ashing method is how to make the solution homogeneous and equally distributed in the 2000-ml longitudinal beaker as each aliquot only represents 100-ml TCE, at the very least, out of 1800 ml. Thus, if such a small sample was not representative, it would yield a significant error in the P_{be}%. After a lengthy investigation and several trials, it was found that the over stirring might take the minerals to the top portion of the beaker. Thus, a higher concentration of minerals

might be attributed to the aliquot than that of the remaining solution at the bottom. Such mineral distribution might end up with unreal ash representation, which leads to a very low P_{be}% compared to the P_{ba}%. That's why it was found that a moderate stirring using a thin agitator (e.g., glass) with no direct pour after the stirring process but waiting for about 5 min to ensure a homogeneous distribution could be efficient. In such a case, the results were found much better, as discussed in the results and discussion section. Since the used beaker was 2000-ml capacity, the solution might exceed such amount; thus, another beaker could be used for the remaining solution. However, all amount of solution had to be decanted through the two beakers multiple times (about 5 times) to ensure an even distribution of minerals in the two beakers. Simultaneously, two preheated ignition dishes at 110℃ were weighed empty and clean after putting it in a desiccator for 10 min. Such a time interval was desired to cool it down with getting a stabilized weight to the nearest 0.001 g. After the time interval mentioned above (5 min), two ~100-ml aliquots were poured in the two preheated ignition dishes. Since most of the time, the beaker has a 20-ml accuracy, it was found that using the sense to take the reading could be 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.

Figure 3. Centrifuge extraction apparatus: extraction of asphalt-solvent solution

Mineral matter extraction

ASTM D2172 (*17*) was followed for mineral extraction. Each ignition dish filled with the 100-ml aliquot was heated on a hot plate at an appropriate temperature (**Figure 4a**). Due to the TCE comprised in the solution, which could easily catch fire, this process had to be in a well-ventilated fume hood. This process remarkably minimized the solution boiling, and the TCE evaporated in a short period (probably 25% of the time if the hot plate was left in the air) in addition to avoiding the risk of explosion that may occur due to the TCE in the solution. In other words, the suction process gets quickly rid of the evaporated TCE; thus no opportunity for TCE to catch fire in the fume hood atmosphere with even a sever temperature exposed (275–300℃ on the hot plate surface, definitely much less in the dish). Almost pure liquid binder, in addition to minerals, would remain after such a process with almost no TCE. Subsequently, the dish, including the extracted liquid binder plus minerals, was exposed to a 600℃ ignition oven for a sufficient time (**Figure 4b**) to end up with the mineral ash. It was found that 75 min could be enough to make sure no remaining liquid binder involved. Due to the severely hot status of dishes, they were kept in room temperature for about 10 min to be easily handled, followed by an extra 10 min in the desiccator (**Figure 4c**) for no external moisture effect.

Saturated Ammonium Carbonate Solution Preparation

The requirements to prepare about 100 ml of the SACS are a 100-g ammonium carbonate and deionized water. Ammonium carbonate was well ground until acquiring a high fineness to increase the surface area as it facilitated its dissolution in water. The 100-g ground ammonium carbonate was poured in an empty, clean beaker, 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℃ (almost the same storing and testing temperature) and until reaching out a homogeneous solution with all particles dissolved in the deionized water to ensure a saturated solution of ammonium carbonate (*20*). The ammonium carbonate crystals were slowly dissolved in the deionized water. Thus, the desired solution would take a long time to be reached. Ultimately, it is no problem if some of the ammonium carbonate crystals settled down on the container's bottom. Such a settlement indicates reaching out to the saturation case.

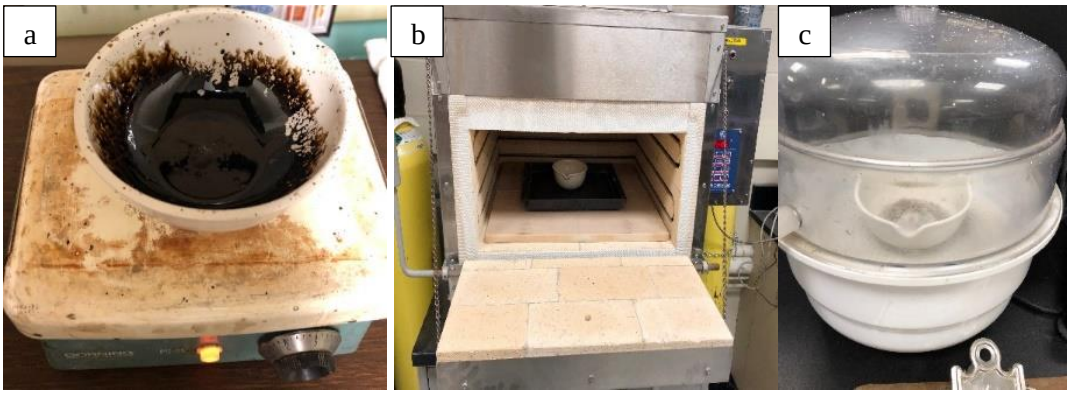

Figure 4. Mineral matter determination: (a) ignition dish with aliquot heated on a hot plate (minerals + binder + TCE); (b) ignition dish in an ignition oven after removing TCE (minerals + binder); (c) ignition dish in a desiccator to cool down after removing binder (mineral ash)

2.1.3 Centrifuge Method

The remaining binder-solvent solution went through the filterless centrifuge device (**Figure 5**). This device was extremely efficient to split the minerals from the binder-solvent solution. The 2000-ml beaker(s) should be washed using extra clean TCE via the filterless centrifuge funnel to make sure that all minerals were included in the filterless centrifuge cup. It was found that gradually decant the solution through the funnel not to block out. Ultimately, the product extracted from this process, which was a pure binder-solvent solution (no minerals involved), was ready for the binder recovery process (out of the scope of this study). According to ASTM D1856 (*21*) and AASHTO T170 (*22*), it is recommended to end up the recovery process within eight hours from blending asphalt mix with the solvent to minimize the undesired binder-solvent interaction. On the other hand, the filterless centrifuge cup involving the minerals was weighed to the nearest 0.001 g to get the net weight of minerals by subtracting the pre-known weight of such a metal cup. The weighing process was implemented after cooling down (about 10 min in the desiccator).

This process is based on the overall weight of minerals extracted with the extracted liquid binder; hence the measured P_b% error is minimized. However, the ashing method is only based on the ash corresponding to approximately 100-ml aliquot (maximum of 5% of all solution). So, if the taken sample is not representative, the error could be significant, ending up with a non-representative P_b%. The results and discussion compare the two methods in a quantitative analysis based on the statistical significance of difference using ANOVA and Tukey Post-Hoc statistical analyses (*18*).

Figure 5. Filterless centrifuge device

2.3 Extracted Aggregate

The extracted aggregate was air-dried under a ventilated hood for about 15 min to minimize the effect of TCE for safety precautions. Hence the bowl, including the aggregate and filter ring, was heated in a conventional oven for about 60 min at 110℃ to ensure no extra TCE included. The bowl, aggregate, and filter ring cooled down at room temperature before getting their weights. In order to get the weight of the filter ring to the nearest 0.1 g, it was brushed from residual minerals into the aggregate bowl. The cooled bowl, including the extracted aggregate, was weighed to the nearest 0.1 g.

3. RESULTS AND DISCUSSION

Table 1 involves the data illustrating the 38 pavement core samples, 13 core sets. The core code could be 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}% as well as P_{be}% by both ashing method (average of two) and centrifuge method.

3.1 ANOVA and Tukey Post-Hoc Statistical Analysis

One-way ANOVA was used to evaluate the significance of difference among the compared binder contents (actual vs. extracted by both ashing and centrifuge methods), as presented in **Table 2**. 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 **Table 3**. One could notice that the statistical significance did not rely on the status of binder (virgin or modified). For instance, the highest statistical significance was attributed to a virgin asphalt mix (US54-7 with F = 27), and one of the smallest ranked statistical significance was assigned to a virgin asphalt mix as well (US54-5 with F = 2.3). Additionally, the F statistic rank did not rely on the RAP/RAS concentrations. As can be seen in the recent four rows in **Table 2**, the US54-3, US54-4, US54-5, and US54-8 core samples provided the lowest significance (less than F critical) with no distinction of modification impact on the variation between the P_{be}% and P_{ba}%. This analysis demonstrates the efficiency of the used solvent (TCE) with the virgin asphalt cement, RAP, or RAS, including the used additives.

Table 1. HMA Core Samples Data and Extracted Binder Content by Ashing and Centrifuge Methods

Set	Sample Label*	Construction Year	Sample Weight	RAP%	RAS%	Virgin PG	Contract grade	Additive***	Virgin AC%	Total P_{ba}%	P_{be}%	
											Ashing (Avg)	Centrifuge
	MO13-1(1)		1228								5.3	5.3
1	MO13-1(2)	2016	1315	17	0	64-22H	70-22	A: 0.5%	4.4	5.7	5.2	5.5
	MO13-1(3)		1255								5.3	5.6
	MO52-1(1)		1677								4.9	5.0
2	MO52-1(2)	2010	1720	0	34	64-22	NA**	B: 1.5% C: 0.8%	3.7	4.8	4.9	5.2
	MO52-1(3)		1548								4.9	5.1
	US50-1(1)		1630								4.9	5.0
3	US50-1(2)	2011	1760	25	0	64-22	NA	B: 1.5% C: 1%	3.8	5	5.0	5.2
	US50-1(3)		1550								4.6	4.9
	US54-1 (1)		2066					D: 2.5%			5.1	5.3
4	US54-1(2)	2016	2098	0	33	58-28	NA	E: 3.5%	3.6	5.2	5.0	5.3
	US54-1(3)		1905					A: 1.5%			5.1	5.5
	US54-2(1)		1842								5.3	5.5
5	US54-2(2)	2016	1970	33	0	58-28	NA	A: 1%	3.6	5.3	5.1	5.4
	US54-2(3)		1973								5.1	5.3
	US54-3(1)		1867								4.8	5
6	US54-3(2)	2016	2223	18	15	58-28	NA	A: 1%	3.6	5.2	5.0	5.3
	US54-3(3)		2115								5.1	5.4
	US54-4 (1)		2186								4.8	5
7	US54-4(2)	2016	2205	35	0	64-22H	NA	E: 3% A: 1%	3.2	4.8	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
	US54-6(1)		1782								4.4	5
9	US54-6(2)	2016	1490	31	0	58-28	70-22	A: 1%	3.6	5.1	4.9	5.2
	US54-6(3)		1721								4.8	5.1
	US54-7(1)		1976								5.8	5.9
10	US54-7(2)	2003	1655	0	0	64-22	NA	F: 0.25%	6.2	6.2	5.8	6.0
	US54-7(3)		1698								5.7	5.8
	US54-8(1)		1576								5.2	5.4
11	US54-8(2)	2006	1695	9	0	70-22	NA	C: 0.5%	5.1	5.6	5.3	5.5
	US54-8(3)		1600								5.6	5.7
	US63-1(1)		1633								4.8	4.9
12	US63-1(2)	2016	1664	35	0	58-28	70-22	G: 0.5% H: 1.75%	3.4	5.1	4.5	4.7
	US63-1(3)		1607								4.6	4.8
	US63-2(1)		1433								4.6	5.4
13	US63-2(2)	2008	1707	20	10	64-22	NA	B: 1.5% C: 0.5%	4.1	5.6	4.9	5.0
	US63-2(3)		1399								5.1	5.3
Average			1755						4.1	5.3	5.0	5.3
Maximum			1228						3.2	4.8	4.4	4.7
Minimum			2223						6.2	6.2	5.8	6.0

*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).
**NA: Not Available.
***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).

Table 2. One-Way ANOVA Results for Actual and Extracted (Ashing and Centrifuge) Binder Contents

Sample	Asphalt Binder Replacement			F	P-value	F crit
	RAP%	RAS%	sum			
US54-7	0	0	0	27.0	0.001	5.1
MO52-1	0	34	34	19.9	0.002	5.1
MO13-1	17	0	17	16.8	0.003	5.1
US63-1	35	0	35	15.0	0.005	5.1
US54-1	0	33	33	12.2	0.008	5.1
US63-2	20	10	30	12.1	0.008	5.1
US54-6	31	0	31	6.2	0.035	5.1
US54-2	33	0	33	5.3	0.047	5.1
US54-3	18	15	33	2.9	0.135	5.1
US54-4	35	0	35	2.7	0.148	5.1
US54-5	0	0	0	2.3	0.251	9.6
US54-8	9	0	9	2.1	0.200	5.1
US50-1	25	0	25	1.9	0.228	5.1

The Tukey tests show the statistical significance of Pba% against Pbe% by ashing, Pba% against Pbe% by centrifuge, and Pbe% by ashing against Pbe% by centrifuge. Four cores resulted in a significant difference between Pba% and Pbe% by ashing, which were MO13-1 (qact = 8.198), US54-7 (qact = 10.205), US63-1 (qact = 7.660), and US63-2 (qact = 6.961) whereas qcrit = 4.339. However, only two cores resulted in a significant difference between Pba% and Pbe% by centrifuge, which were MO52-1 (qact = 8.647) and US54-7 (qact = 6.803). Such statistical analysis may illustrate much credibility towards the centrifuge method compared to the ashing method. On the other hand, the statistical significance between ashing and centrifuge extraction methods resulted in three core samples, which were MO52-1 (qact = 6.245), US54-1 (qact = 6.971), and US54-2 (qact = 4.556).

Table 3. Tukey Post-Hoc analysis results of compared actual and extracted binder contents

Core Sample		MO13-1*	MO52-1*	US50-1	US54-1*	US54-2*	US54-3*	US54-4*	US54-5**	US54-6*	US54-7*	US54-8*	US63-1*	US63-2*
i	j							q_{act}						
P_{ba}%	P_{be}%, Ashing	8.198	2.402	0.031	3.098	2.847	2.711	0.721	2.976	4.313	10.205	2.818	7.660	6.961
P_{ba}%	P_{be}%, Centrifuge	4.099	8.647	0.006	3.873	1.708	0.387	2.402	1.082	0.000	6.803	0.751	4.754	3.329
P_{be}%, Ashing	P_{be}%, Centrifuge	4.099	6.245	0.036	6.971	4.556	3.098	3.122	1.894	4.313	3.402	2.067	2.905	3.632

*q_{crit} = 4.339
**q_{crit} = 4.501

3.2 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}% could depict to what extent each extraction method outcomes were close to the P_{ba}% in a simple manner. **Figure 6** illustrates such a comparison using the 38 core sample size. The line of best fit of the P_{be}% by centrifuge was closer to the identity line, compared to the ashing P_{be}%. Such a comparison could indicate 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.

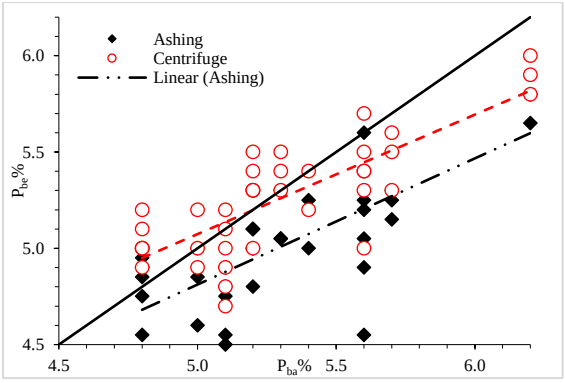

Figure 6. Linear least-squares regression: P_{be}% by ashing and by centrifuge compared to P_{ba}%

4. CONCLUSIONS

The authors in this research believe that Method A (Centrifuge Extraction) in ASTM D2172 is highly efficient, and almost 100% of the binder could be extracted. Additionally, it is the only method that could be followed by the extracted binder characterization. However, the problem of the P_{be}% calculations is 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 a remarkably hands-on experience to end up with consistent outcomes. Thus, the variation between results could be minimized when comparing the P_{be}% to the P_{ba}%, or even among the P_{be}% (by different extraction methods) from the same asphalt mix source. Although both ashing and centrifuge methods did not result in identical outcomes against P_{ba}%, the centrifuge method relatively provided better results compared to the ashing method by about 0.2–0.3%. Based on the provided 38 pavement core samples, the average P_{be}% by ashing was 5.0%, whereas it was 5.3% by centrifuge. Additionally, this later average was the same as the average P_{ba}% (5.3%). The ashing method provided such a significant variation with P_{ba}% despite enhancing its procedure by some modifications to ASTM D2172. The authors derived that 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 has a high potential to take place. On the other hand, the centrifuge method is based on the evaluation of the minerals in the overall binder-solvent solution. Thus, the calculation error is minimized to a high extent. Furthermore, the centrifuge method provides much saving in the experimental time (about 3.5 hours) compared to the ashing method (about 7 hours), if the sample is loosened and ready for the solvent addition. Nevertheless, it was found that the ashing outcomes (P_{be}% by ashing) were equal to the centrifuge outcomes (P_{be}% by centrifuge) in case of disregarding the addition of SACS. Thus, it could be recommended to reassess the SACS addition since it might excessively compensate for minerals that have not been lost by the ignition oven.

ACKNOWLEDGMENT

Special thanks go to Allison Talley (a Missouri Department of Transportation, MoDOT representative), who technically provided us with all support needed. Likewise, the authors would like to thank Steven Lusher, Ph.D. (Senior Research Specialist, Missouri S&T) for his assistance with providing extraction and recovery information based on his appreciated experience.

AUTHOR CONTRIBUTIONS

The authors confirm contribution to the paper as follows: study conception and design: Abdelrahman, Hemida; data collection and lab testing: Hemida and Deef-Allah; analysis and interpretation of results: Hemida, Abdelrahman; draft manuscript preparation: Hemida, Abdelrahman. All authors reviewed the findings and approved the final version of the manuscript.

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