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Bitumen Extraction Techniques and Their Influence on Hot Mix Asphalt Properties

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ABSTRACT

The bitumen content plays a crucial role in ensuring the quality of hot mix asphalt (HMA). Precise measurement of bitumen levels in HMA is vital for assessing its performance and durability in pavement applications. This study employs two systematic methods for determining bitumen content using the volumetric properties of aggregates, asphalt in the HMA. The methods used are the Centrifuge Solvent Extraction Test (AASHTO T164), which quantitatively extracts bitumen from bituminous paving mixtures, and the Ignition Furnace Test (AASHTO T308), which measures the bitumen content in bituminous mixtures. Laboratory data were collected from two mixes of asphalt concrete wearing course (14 mm and 20 mm). Designed with the Marshall mix design system according to the AASHTO standard. Aggregate from a quarry at Ras Al-lafia and bitumen of 60/70 PEN. The optimum bitumen content for each mix was 5.0% and 5.3%, respectively. Samples were extracted using the centrifuge method at a speed of 3600 rpm and the ignition method at a temperature of 538°C. Testing revealed a significant difference in bitumen content between the two methods. Additionally, sieve analysis results showed that high temperature and solvent exposure affect aggregate gradation, leading to noticeable differences.

Keywords: hot mix asphalt, bitumen content, AC14, AC20, centrifuge solvent extraction, ignition method.

تقنيات استخراج البيتومين وتأثيرها على خصائص الخلطة الإسفلتية

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ملخـــــــص البحـــــــث

يعتبر محتوى البيتومين عاملاً حاسماً في ضمان جودة الخلطة الإسفلتية الساخنة (HMA) يعد القياس الدقيق لمستويات البيتومين فيها أمرًا بالغ الأهمية لتقييم أدائها ومتانتها في تطبيقات الرصف. تستخدم هذه الدراسة طريقتين منهجيتين لتحديد محتوى البيتومين باستخدام الخصائص الحجمية للركام والإسفلت في الخلطة الإسفلتية الساخنة. الطرق المستخدمة هياختبار الاستخلاص بالمذيبات بالطرد المركزي (AASHTO T164) الذي يستخلص البيتومين كمياً من الخلطات الإسفلتية، واختبار الفرن بالاحتراق (AASHTO T 308) الذي يقيس محتوى البيتومين في الخلطات الإسفلتية . تم جمع بيانات

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المختبر من خليطين الطبقة السطحية للخرسانة الإسفلتية (14 مم و20 مم) بيتومين 60/70 والركام من كسارات راس اللفع. كان المحتوى الأمثل للبيتومين لكل خليط 5.0% و5.3%، على التوالي. تم استخلاص العينات باستخدام طريقة الطرد المركزي بسرعة 3600 دورة في الدقيقة وطريقة فرن الاحتراق عند درجة حرارة 538°C أظهرت الاختبارات فرقًا واضحا في محتوى البيتومين بين الطريقتين أضافة إلى ذلك، كشفت نتائج التحليل المنخلي أن التعرض لدرجات الحرارة المرتفعة والمذيبات يؤثر على تدرج الركام، مما يؤدي إلى اختلافات ملحوظة.

الكلمات الدالة: طريقة الاحتراق. طريقة الطرد المركزي، AC14, AC20 الخلطة الاسفلتية الساخنة، محتوي البيتومين.

1.0 Introduction

Asphalt has been utilized by humans for millennia. In the hot mix asphalt (HMA) paving industry, bitumen serves as a crucial binder, securing aggregate particles. The bitumen content in asphalt mixtures is a key physical property that significantly impacts the durability and longevity of bituminous pavements. Too much bitumen results in mixture stability problems, while too little bitumen compromises durability [1].

For the accurate determination of bitumen content in paving mixtures, the centrifuge extraction method has been widely utilized due to its efficiency. This method involves weighing the sample, slightly heating it until it crumbles, cooling it, placing it in a rotor bowl, and adding a solvent for extraction. Designed in accordance with AASHTO T58, T164 & ASTM D2172 standards, the centrifuge extractor ensures reliable bitumen percentage determination. Historically, trichloroethane (TCA) and trichloroethylene (TCE) were commonly used solvents [2]. However, with biodegradable solvents failing to fully replace chlorinated solvents, alternative testing methods are being developed to enhance accuracy and safety.

One such method is ignition testing, developed at the National Centre for Asphalt Technology (NCAT) for determining asphalt content in HMA mixtures. This approach eliminates the need for solvents, using an ignition furnace that meets AASHTO T308 Method A and ASTM D6307 Method A standards. Bitumen content is calculated by measuring the mass difference between the initial HMA sample and the residual aggregate after ignition [4].

Studies have shown that ignition testing outcomes closely match centrifuge extraction results when ammonium carbonate is excluded. This suggests the need to reassess its role, as it may excessively compensate for minerals unaffected by ignition [3].

However, the research suggests that the ignition oven method is more environmentally friendly and provides quicker results compared to the centrifuge extraction method. Its accuracy can be affected by the aggregate properties and binder characteristics [4]. Additionally, ignition testing has demonstrated a tendency to yield slightly higher binder content measurements compared to solvent-based extractions, emphasizing the importance of mineral separation in centrifuge testing. Precision studies examining repeatability and reproducibility confirm the reliability of both techniques [5].

Further validation was achieved through testing 80 HMA samples comprising limestone and gravel aggregates. Findings suggest that aggregate type, gradation, and bitumen content influence measurement accuracy, affecting deviations between true and measured binder content [6].

Comparative analysis involving multiple laboratories confirmed that nuclear gauge measurements provide higher precision and accuracy than centrifuge solvent extraction [7]. Additionally, research evaluating normal propyl bromide (nPB) solvents as potential replacements for chlorinated solvents demonstrated their feasibility for use in HMA extraction and recovery processes [8].

In addition, the research explores the interaction between Reclaimed asphalt pavement RAP and virgin binders, emphasizing that assuming full blending can lead to insufficient binder content and reduced pavement durability. A modified blending chart and partial blending method are proposed to determine the appropriate virgin binder grade and content. Findings suggest that considering blending ratios improves cracking resistance while maintaining rutting performance [9].

1.1 Problem Statement

The Centrifuge Extraction Method and the Ignition Furnace are two commonly used techniques for measuring asphalt bitumen content. The Centrifuge Method involves dissolving bitumen using petroleum and chemical solvents. However, with increasing petroleum costs and safety concerns, the Ignition Furnace method has gained preference as an alternative. Despite its advantages, the Ignition Furnace is costlier compared to the centrifuge machine. This study seeks to determine which method is most suitable for meeting design requirements across various mix types.

1.2 Objective

This study aims to comprehensively evaluate two distinct methods for determining bitumen content in hot mix asphalt (HMA) mixtures while examining their influence on aggregate breakdown.

2 Methodology:

A test plan was developed to achieve the study's objectives. The Marshall mix designs were adopted from two previous studies based on AASHTO standards, resulting in the production of two asphalt concrete wearing course (ACW) mixture types 14 mm and 20 mm. The aggregate was sourced from a quarry at Ras Al-Lafia, and 60/70 PEN bitumen was used. The optimum bitumen content for each mix was determined to be 5.0% and 5.3%, respectively. Six specimens were prepared for each mix, with three tested using the Centrifuge Extraction Method (AASHTO T164) and three using the Ignition Furnace Method (AASHTO T308) [10, 11]. The mixtures were extracted using the centrifuge method, and the bitumen content was calculated by subtracting the mass of the extracted aggregate, moisture content, and mineral matter from the total mass **at** Alalia Geotechnical Consulting Company laboratories in Tripoli, Libya. For the ignition furnace method, the bitumen content was determined by the difference in mass between the residual aggregate and the moisture content. Sieve analysis was conducted to assess the potential aggregate breakdown resulting from both methods.

2.1 Calculations:

Calculate the asphalt content by Centrifuge Extraction.

$$\text{Asphalt Content \%} = (W_1 - (W_2+W_3))/W_1 \times 100 \qquad (1)$$

where W_1 is the weight of the test sample, [g], W_2 is the weight of extracted aggregate, [g], and W_3 is the weight of fines in extracted solvent, g.

Calculation of the asphalt content by Ignition Furnace.

$$\text{Asphalt Content \%} = \frac{Mi - Mf}{Mi} \times 100 \ \text{- Mf - Cf} \qquad (2)$$

where Pb is the corrected asphalt binder content as a percent by mass of the HMA sample, Mf is the final mass of aggregate remaining after ignition, Mi is the initial mass of the HMA sample before ignition, Cf is the Asphalt binder correction factor as a percent by mass of the HMA sample and M is the percent moisture content.

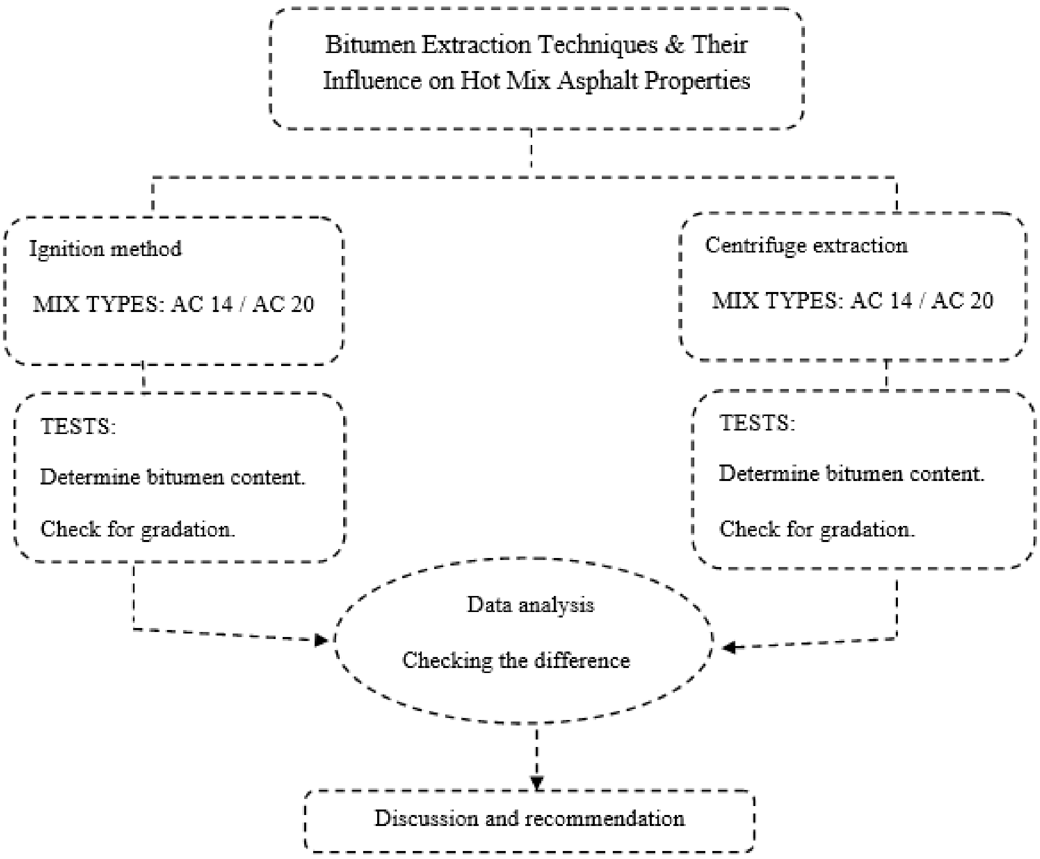

Figure 1. Procedure outline

Figure 2. Centrifuge Extraction

Figure 3. Ignition Furnace

3 Results and Discussion

3.1 Ignition Aggregate gradation:

In general, both of the mix types experience some weight loss when tested at high temperatures. The weight loss is measured as a percent of the initial aggregate weight. To evaluate only the effects of the ignition method on aggregate gradation, the changes in gradation due to mixing must first be quantified. The average percent passing of the recovered aggregate for each mix type was calculated for each sieve size. The next step was to determine the bias from the original value for two types of mixes, and the results ranged from about (-2.81 to 1.17), depending on the sieve size. The respective difference between the actual and the measured percent passing for each sieve is relatively high. Tables 1 and 2 show the difference between the measured percent passing and the actual one, and Figures 4 and 5 outline the gradation limit of the measured percent passing of each.

Table 1. The aggregate gradation of AC14 after being tested by Ignition.

Sieve size mm	^0.45	Lower	Upper	True Passing %	Avg passing %	Bias %
20.0	3.85	100	100	100	100	0
14.0	3.279	90	100	95	95.19	0.19
10.0	2.818	76	86	81	82.17	1.17
5.0	2.063	50	62	56	56.34	0.34
3.35	1.723	40	54	47	46.95	-0.04
1.18	1.077	18	34	26	25.43	-0.56
0.425	0.68	12	24	18	16.22	-1.77
0.150	0.426	6	14	10	8.05	-1.94
0.075	0.312	4	8	6	3.68	-2.31

Figure 4. displays the gradation limit of the passing% AC14 Ignition

Table 2. The gradation of AC20 after being tested by ignition

Sieve size mm	^0.45	Lower	Upper	True Passing %	Avg passing %	Bias %
28.0	4.479	100	100	100	100	0
20.0	3.85	76	100	88	88.84	0.84
14.0	3.279	64	89	76.5	76.08	-0.41
10.0	2.818	56	81	68.5	67.46	-1.03
5.0	2.063	46	71	58.5	57.27	-1.22
3.35	1.723	32	58	45	44.77	-0.22
1.18	1.077	20	42	31	29.60	-1.39
0.425	0.68	12	28	20	17.96	-2.03
0.150	0.426	6	16	11	8.36	-2.63
0.075	0.312	4	8	6	3.18	-2.81

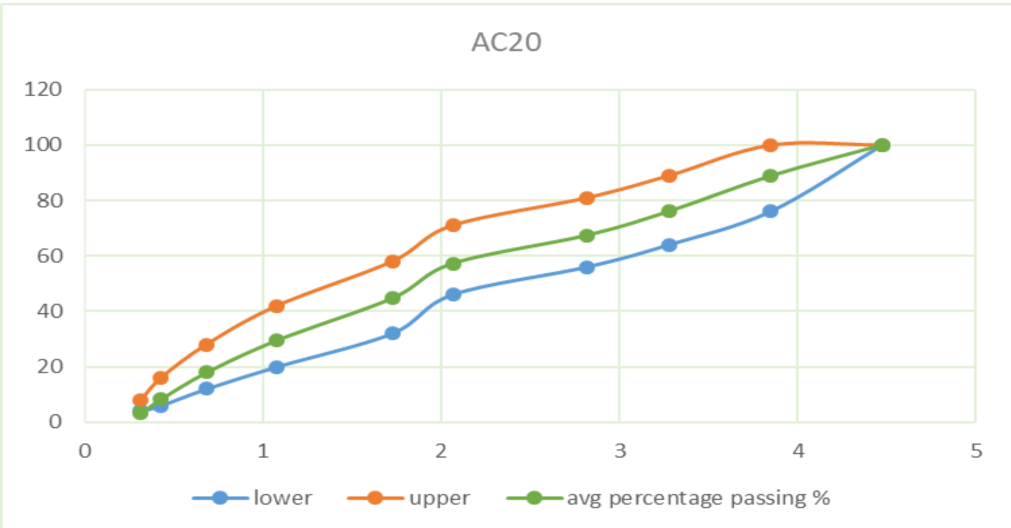

Figure 5. The gradation limit of the passing % AC20 Ignition

3.2 Centrifuge Aggregate Gradation

Due to the solvent and speed 3600 rpm resulting in aggregate weight loss, some of the dust goes out with the effluent and some hangs on the filter that causing dust loss. The deviation of the measured percent passing from the true percent passing for the two mixes is shown in the Tables below. The bias ranges from (-2.29 to 0.99), (-2.64 to 2.64) percent for (AC14 and AC20), respectively. Since the difference between the true percent passing and the measured percent passing was high, the percent passing can be determined with a low degree of accuracy. Tables 3 and 4 show the difference between the measured percent passing and the actual. Figures 6 and 7 show the gradation limit of the measured percent passing of each.

Table 3. The gradation of AC14 after being tested by the centrifuge.

Sieve size mm	^0.45	Lower	Upper	True Passing %	Avg passing %	Bias%
20.0	3.85	100	100	100	100	0
14.0	3.279	90	100	95	94.71	-0.28
10.0	2.818	76	86	81	81.99	0.99
5.0	2.063	50	62	56	55.11	-0.88
3.35	1.723	40	54	47	46.21	-0.78
1.18	1.077	18	34	26	24.52	-1.47
0.425	0.68	12	24	18	15.95	-2.04
0.150	0.426	6	14	10	7.99	-2.00
0.075	0.312	4	8	6	3.70	-2.29

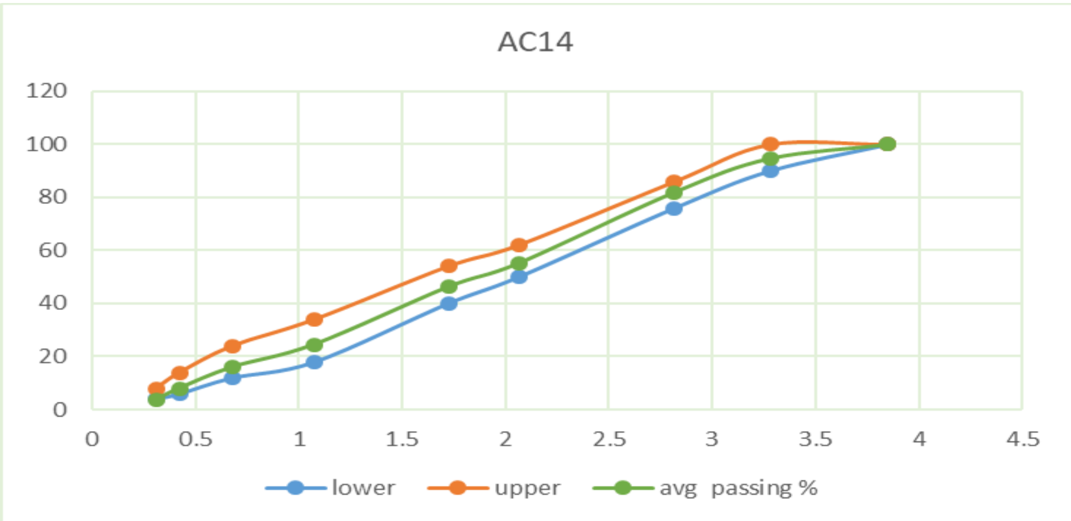

Figure 6. displays the gradation limit of the passing % AC14 Centrifuge

It can be noticed that there is not much difference among the aggregate gradations of each method. According to the tables, the average percentage passing fits the range of the upper-lower limit at every sieve except for sieve 75 µm, which obviously can be seen in the figures because of dust loss.

Table 4. The gradation of AC20 after being tested by the Centrifuge.

Sieve size mm	^0.45	Lower	Upper	True passing %	Avg passing %	Bias%
28.0	4.479	100	100	100	100	0
20.0	3.85	76	100	88	90.64	2.64
14.0	3.279	64	89	76.5	76.38	-0.11
10.0	2.818	56	81	68.5	67.51	-0.98
5.0	2.063	46	71	58.5	57.16	-1.33
3.35	1.723	32	58	45	44.36	-0.63
1.18	1.077	20	42	31	29.49	-1.50
0.425	0.68	12	28	20	18.36	-1.63
0.150	0.426	6	16	11	8.67	-2.32
0.075	0.312	4	8	6	3.35	-2.64

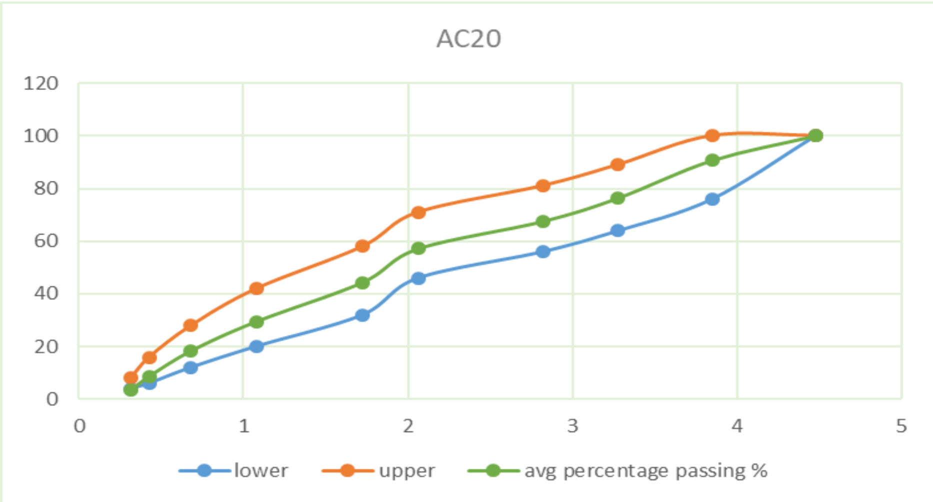

Figure 7. displays the gradation limit of the passing% AC20 Centrifuge.

3.3 Centrifuge and Ignition:

The asphalt content is determined by comparing the weight of the aggregate before and after testing. The results of the T-test on Microsoft Excel indicate that the two-tailed p-value for AC14 exceeds 0.05, meaning there is no statistically significant difference in performance. For a difference to be considered significant, the t statistic must be equal to or greater than the t critical value. However, at AC20, the p-value is below 0.05, and the t-statistic surpasses the t-critical value, confirming a significant difference, as illustrated in Table 5.

Table 5. The result of the t-test paired two-sample for means

Mix	t stat	t-critical two-tailed	P(T<=t) two-tail
AC20	8.597	4.302	0.01325
AC14	0.581	4.302	0.61971

Each mix has three samples for Ignition and three replicates samples for Centrifuge method. Table (7) shows the different between the true AC% and the average AC% of three specimens for the two mixes. For AC14 the difference between true AC% (design) & measured (Ignition) ranged from 0.22 to 0.33

percent, AC% (design) & measured (Centrifuge) ranged from 0.08 to 0.15 percent and the difference between Ignition & Centrifuge ranged from 0.07 to 0.41 percent.

Table 6. The Average AC% measured by Ignition and Centrifuge

Mix	*AC%*	*Ignition AC%*	*Centrifuge AC%*
AC14	5.0%	5.15	5.3
		5.21	5.1
		5.31	5.07
Avg		5.22	5.15
AC20	5.30%	5.5	5.06
		5.8	5.32
		5.6	5.28
Avg		5.63	5.22

Table 7. Range difference for the two mixes.

Mix	**Diff (Ignition &Design)**	**Diff (Ignition &Centrifuge)**	**Diff (Centrifuge &Design)**
AC 20	0.33	0.41	0.08
AC 14	0.22	0.07	0.15
Avg	0.275	0.24	0.115

4 Conclusions:

Based on the results presented in this study, several key conclusions can be drawn. Statistical analysis indicates that for AC14, there is no significant difference in bitumen content when comparing the Centrifuge Solvent Extraction Test and the Ignition Furnace Test. However, for AC20, a notable variation in performance between the two methods was observed, suggesting that the selection of the extraction method plays a crucial role in determining bitumen content accuracy. Additionally, sieve analysis highlights the influence of heat and solvent exposure on aggregate gradation. From the percentage passing results, it can be observed that the ignition test yields values nearly identical to those obtained through the centrifuge method. Nevertheless, the gradation remains near the specified limits for most sieve sizes tested using either method, except for the 75µm sieve, which consistently falls outside acceptable thresholds for both AC14 and AC20. Furthermore, the effects of solvent exposure and centrifuge speed (3600 rpm) contribute to aggregate weight loss. Some fine particles are carried away with the effluent during the extraction process, while others adhere to the filter, resulting in dust loss. These findings underscore the importance of carefully considering the extraction method to ensure reliable bitumen content measurements and maintain aggregate integrity in hot mix asphalt applications.

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