QUALITY CONTROL ON BITUMEN DELIVERED TO ROAD CONSTRUCTION SITES

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ABSTRACT: There are concerns over poor quality bitumen on road pavements in Malaysia. This is manifested by premature failure of bituminous road surfacing which is common in this country. This paper identifies opportunities for improvement to the current bitumen quality framework, mainly the Malaysian Standards (MS 124) and with reference to laboratory test methods, Public Works Department Malaysia (PWD) specifications and road construction guidelines. It also looks into the current industry practice and identifies Quality Assurance/Quality Control (QA/QC) measures applicable throughout the entire bitumen supply chain. Bitumen samples from some on-going road construction project throughout the country were collected, tested and the results are presented in this paper. From this study, the PWD has introduced procedures to be used by the asphalt plants before accepting bitumen. These procedures include testing, documentation and observation. This is to ensure that the bitumen supplied is not of inferior quality that would affect the performance of asphalt on the road.

KEY WORDS: Quality, bitumen, pavements, failure, testing, documentation, observation, asphalt

1. CURRENT BITUMEN INDUSTRY PRACTICES IN MALAYSIA

There are several bitumen suppliers in Malaysia, which includes PETRONAS, Shell, Esso, Caltex and Kemaman Bitumen Company (KBC). The different bitumen suppliers have various origins of crude oil production, types and sources, which contribute to variation in the bitumen quality.

Crude petroleum varies in their compositions from source to source. They yield different amounts of hydrocarbon fractions, including bitumen. Not all crudes contain heavy hydrocarbon fractions, which is the essential component in making bitumen.

Depending on the crude type and refining process capabilities, there are several manufacturing routes available to produce bitumen. Straight run or reduction to grade is the most direct and common practice; however, the other options such as blending and air blowing to grade might be necessary. Blending of two or more crude feedstock or residues is quite a common practice now. Control blending proportions of bitumen residues of different viscosities, e.g. high and low components, will yield a blend meeting the desired specification requirements. However, the selection of suitable crude oil and bitumen feedstock is also important to ensure compliance.

The quality of bitumen produced is tested and certified by the refinery QC laboratory upon entering the storage tank. It is not very often that a refiner introduces a new type of crude oil or change manufacturing operations in the refinery. Hence knowing the component properties, the final blend output or finished product properties can be estimated prior to having the full test results of product quality and certificate.

This paper looks into the current industry practices and identifies QA/QC measures applicable throughout the entire bitumen supply chain, starting from bitumen production at the refinery, during distribution and transport process, at customer storage facilities at mixing plants and through to the final road paving applications. It also gives recommendations on how these could be further improved to ensure that the bitumen supplied is not of inferior quality.
2. RESULTS AND FINDINGS

2.1 Basic Properties: Penetration and softening point

The results as shown in Figure 1 indicates that the penetration from laboratory testing have minimum values of 67, maximum 90 and median 81 dmm. On the other hand, the Certificate of Quality (COQ) traced these samples reported minimum values of 83, maximum 93 and median 86 dmm.

The penetration of bitumen samples should be in the range 80 to 100 according to the Malaysian Standard [1]. From 59 samples collected from all over Malaysia, only 56% of them passed the Malaysian Standard [1] specification although 100% samples passed as stated in the COQ. The results show inconsistency penetration of bitumen that was delivered by the suppliers compared to the result that were stated in the COQ. This might be caused by many reasons and one of them was the bitumen properties that had changed or been disturbed during transportation. Because of the long journey during travelling from one stop to the other, the bitumen could have been overheated and this could change the properties of bitumen.

![Figure 1. Penetration results](attachment:image1.jpg)

![Figure 2. Softening point results](attachment:image2.jpg)
Softening point values in Figure 2 show a better correlation and less spread. The softening point of bitumen samples should be in the range 45 to 52 °C according to the Malaysian Standard [1]. From 59 of total samples collected, 20% or 12 samples failed to meet the minimum specification requirements although 100% samples were deemed pass as stated in the COQ.

Figure 2 also shows that the minimum softening point was 42 °C, maximum 48 °C and average 45.1 °C. COQ results traced to similar samples showed minimum value was 45.4 °C, maximum 47.6 °C and average 45.8 °C. A similar scenario existed whereby the bitumen softening point results were not consistent with COQ values as happen to penetration results. All the samples seem to pass the softening point requirements for 80/100 bitumen grade according to COQ but the test results show 79% passed the requirement of the Malaysian Standard [1] for 80/100 bitumen grade and 51% of them was below the minimum value. As what had been related to the transportation condition before, the different value between test results and COQ values also may be due to the improper equipment handling or non-skilled operator in carrying out both tests.

Figure 3 shows the data tabulation of, softening point against penetration. Only 41% of the points fall within the specification area and it generally shows that softening point of bitumen decreases as penetration increases.

2.2 Temperature Susceptibility: Viscosity and penetration index (PI)

The measurement of bitumen viscosities at different temperatures is not specified in the current MS124 specifications [1]. A total of 20 samples were selected, after passing the basic criteria and the samples show steady rate of viscosities change, which is typical of fresh neat bitumen. Referring to Figure 4, at 60 °C, the viscosities have wider range of results than that at 135 °C. Hence, at higher temperatures, the bitumen is showing a closer viscosities range, especially at bitumen-aggregate mixing, paving and compaction temperatures. This would seem easier for asphalt plant operator or paving contractor to monitor and control the operational temperatures, even if there is a change in bitumen source or batch.
Figure 4. Viscosity results

Referring to Figure 5, the PI calculated from the softening point and penetration values shows a much higher degree of variation. The highest PI calculated was -4 and the lowest were -2.2. This explains why the bitumen samples have shown similar viscosity at high temperatures, but different at low temperatures. With high PI, the rate of viscosity change to temperature is lower than bitumen with low PI.

Figure 5. PI estimated from penetration and softening point

2.3 Purity Criterion: Solubility, flash point and true boiling point
Solubility of bitumen in trichloroethylene was conducted according to ASTM D2042 - 09 [2]. A total of 41 samples were tested. Overall, the solubility results show good compliance to the MS 124 requirements [1] with minimum value 99.4%. The minimum requirement level is 99.0%. The mode was 99.9%, which shows good purity level.
Flash point according to ASTM D92 - 12 [3] was conducted on 25 samples. Fifteen samples were selected on the purity criteria after the basic properties tests while an additional ten samples were selected due to the unexpected “high loss of material” results from the durability criteria, i.e. Loss on Heating (LOH). Following the laboratory test flow scheme, the next requirement is to check these samples for purity criteria. Initial findings indicate that the flash point test shows somewhat a weak correlation with loss on heating. Bitumen with higher penetration drop value will show a lower flash point value.

Flash point is an important indication for safe handling of bitumen and may detect presence of low temperature flashing materials in the sample. The results of flash point test on the samples show compliance to the MS124 requirements [1] with the overall average of 322 °C and standard deviation value 14.7 °C. The initial findings based on the results from the 10 selected samples (after high loss of mass result of LOH) show a lower average of 310 °C. Hence, it may still be useful to establish whether flash point results are able to correlate and indicate samples with higher content of volatile materials than normal after going through the hardening test (LOH or TFOT).

Another type of test carried out on the purity criterion was the True Boiling Point (TBP) using Gas–Liquid–Chromatography technology according to ASTM D7169 - 11 [4]. The objective of the test is to find any significant high level of light petroleum or oil fractions in the bitumen sample. This will also indicate whether that there is any contamination of the bitumen components, other than the normal heavy fractions of the refining residue. The samples are first heated to 540 °C to assess if there is more than 10% of material boiling at this temperature. If there is more than 10%, the test will start from lower temperatures, the equipment will measure the percentage of material boiling at that temperature. The key here is to identify the percentage of bitumen material boiling at 450 °C and 500 °C. Normal limiting value used in this study is less than 4% at 450 °C and less than 10% at 500 °C.

The TBP test was conducted on 15 samples selected based on their results after the basic properties test criterion. Overall, there were eight samples having less than 10% material boiling at temperature of 540 °C, hence the test stopped there for these samples. They can be considered as pure and typical neat bituminous samples. Nonetheless, seven other samples required further evaluation at 450 °C and 500 °C. Even for these samples, the test results show that there were minimal amount of material that boils at the lower end of the temperature range. The overall average at 450 °C was 0.9%, while at 500 °C it was 3.5%. Hence, the TBP-CLC result is non-conclusive and not useful at this stage for further evaluation. More in depth study may be needed to determine any correlation to high values of LOH or hardening after Thin Film Oven Test (TFOT) to the TBP-GLC results.

3. ADDITIONAL SURVEY – BITUMEN CONTAMINATION

It is empirical to carry out validation tests on the quality of bitumen. It is estimated that each delivery of 35 tonnes of bitumen will produce about 650 tonnes of paving asphalt. Based on laboratory test result (refer to Figures 6, 7 and 8), some foreign material mixture as low as 2.5% by weight of the bitumen is sensitive to the penetration and softening point. However, the flash point test is not sensitive to contamination but formation of foam during heating the bitumen can be used as an indicator of bitumen contamination.

Each delivery of bitumen to the asphalt plant is usually given a COQ, which contains the results of bitumen tests conducted by the refinery plant, and the Certificate of Quantity.

From the survey conducted, only 24 of the 59 asphalt plants could produce the COQ. From 59 asphalt plants, only 13% had actually requested for a copy of COQ for each bitumen delivery, 8% requested only once a month, 4% requested every 6 months, 4% requested once a year but the majority of 71% rarely requested the COQ.

The asphalt plant also should examine the condition and serial number of security seals as the bitumen tanker arrived at the plant. According to a survey conducted, the majority of the asphalt plants (49) did examine the security seal before bitumen was transferred to the storage tank.
The worry is the security seal could be cut or changed and some bitumen stolen and replaced with recycled materials. This would affect the quality of bitumen.

![Graph 1: Softening point versus adulteration](image)

Figure 6. Softening point versus adulteration

![Graph 2: Penetration versus adulteration](image)

Figure 7. Penetration versus adulteration
4. CONCLUSIONS

Out of 59 samples, 44% pass both penetration and softening point tests as per Malaysian Standard requirements [1], whereas 17% fail on softening point, 36% fail on penetration and 3% fail both tests. The temperature susceptibility criterion points toward a more typical viscosities at high temperatures and have not shown any significant differences for the variation in bitumen sources. Preferably, the asphalt plant operators must retain sample of bitumen delivery for later testing.

The solubility test in trichloroethylene is found to be not a sensitive measure of bitumen purity and to consider excluding it from the MS124 requirements [1] or listing it as non-critical test. Many other national specifications also have abandoned this test as mandatory and/or replace it with a more environment-friendly solvent e.g. toluene.

The flash point value on the other hand can be used more effectively as basis to detect impurities or presence of light and volatile materials in the bitumen. The MS124 requirement [1] or minimum level of passing (230 °C) may need to be re-assessed as test results from all samples have an average of 322 °C to cover 95% of all bitumen samples tested. The flash point test should also be the first test carried out at the laboratory for safety reasons. This is because the test operator may expect a much higher flash point level and can be hazardous if the bitumen sample is over heated in the oven during test preparations. The test is relatively “quick and practical” with immediate result upon completion of testing.

TBP measure using the gas liquid chromatography technology is found to be not a sensitive test for detecting minor contamination. While, the TFOT and LOH were compared and the results show that the TFOT is a more severe aging test and can better show higher percentage on loss of mass. The TFOT test combined with measurement of drop in penetration and increase in softening point after heating will have better indication on the limit of hardening for the bitumen grade.

The current industry practice and operations required in ensuring quality in supply, distribution and storage of bitumen prior to being utilised in the road paving application has a lot to improve. The complete bitumen supply chain is an important part of the quality framework and stakeholders, including bitumen suppliers and users,
must protect it against potential quality degradation in any part of supply chain. A few measures worth consideration were suggested, such as implementing the bitumen transport vehicle monitoring system and sample retention at customer site on delivery, are some ways to improve the current practice.

5. RECOMMENDATIONS

The road authority and project owner are recommended to ensure that there is evidence of traceability of bitumen supply to the customer or asphalt plant through to the road contractor for application and are well documented. A quality system can be adopted into the bitumen quality framework to ensure that the bitumen suppliers, asphalt plant operators and road contractors follow the recommended procedures. Hence, bitumen quality, grade, type, and source can be quickly determined at every stage of the bitumen supply chain.

Bitumen samples should be retained for a designated period at the refinery source, distribution depot and at delivery locations, e.g. the asphalt plants. This is to ensure that there is a traceable bitumen sample of the delivery made in cases of quality problems at any of the supply chain stages.

In addition to using suitable security seal for each delivery load, bitumen transporters and road tankers of bulk bitumen should adopt the “vehicle-tracking-system” to reduce risks of product loss, contamination and to increase quality assurance level for bitumen during distribution and transit. Security seal must be of suitable design and material, such as steel, that is not easily tampered. Each asphalt plant should be equipped with at least three bitumen testing equipment i.e. penetration, softening point and flash point for checking the quality of bitumen delivered to the plant.

REFERENCES: