The Influence of Curing Methods on Marshall Stability and Flow

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Abstract. The Marshall Mix design is one of the most widely used methods for designing and evaluating hot asphalt mixes globally, and the main Marshall Test focuses are stability and flow. Two standard curing methods are normally followed to elevate the temperature of testing samples; these involve immersing the samples in 60°C water or placing the samples in an oven at 60°C. These standard curing methods may not simulate the actual state of heating of asphalt pavement in the field, however. In this research, a new curing method that includes insulating the samples before immersion in hot water is thus introduced and compared with the two standard curing methods. During immersion, the water temperature is increased to 60°C and the core temperature of the insulated samples determined. Three sets of Marshall Samples were prepared and cured using the outlined methods; each set consisted of 18 specimens of 101 mm diameter and 63.5 mm height. All specimens were tested using the Marshall Test for stability, flow, and Marshall Stiffness. The set mean results showed that the oven cured samples demonstrated the highest stability values, followed by the standard water cured samples, while the insulated samples exhibited the lowest stability values. The oven cured samples also exhibited higher Marshall Stiffness Index (MSI) values than the other curing methods. However, one-way ANOVA (single factor) testing demonstrated that these differences were not statistically significant.

Keywords: Asphalt concrete, Curing methods, Insulated samples, Marshall test, and Marshall stiffness

1. Introduction

The Marshall Mix design concept was developed initially by a highway engineer in the Mississippi Highway department named Bruce Marshall in 1939; it was then modified by the USA Corps of Engineers in 1948 and standardized by the American Society for Testing and Materials (ASTM) under the designation ASTM D1559 in 1958 (Preston, 1991, Brown et al., 2001). The main standard test was later divided into two standard tests, ASTM D6926, used to prepare the moulds for asphalt concrete, and ASTM D6927, which determines the stability and flow of bituminous mixtures using Marshall apparatus. The main outputs from Marshall apparatus testing are stability ratings, which indicate the maximum resistance of an asphalt concrete sample to an external diametrical load applied at a rate of 50.8 mm/min (2 in/min), and flow, recorded in 0.25 mm (0.01 in) increments, which represents the plastic deformation of the sample at the maximum applied load (Institute, 2007).

The Marshall Method has acquired great significance as a method for design and evaluation of the Hot Mix Asphalt (HMA) used in airports and highways. Alongside its suitability for different
types of gradations such as dense graded and stone matrix asphalt, its applicability in both laboratory mix design and field control processes is also relevant (Roberts et al., 2004). Due to its application breadth, the Marshall Method, despite some shortcomings, is the most widely used mix design method in the world (NHI, 2014, Sadid et al., 2010).

In order to determine the stability and flow of an asphalt concrete sample, whether prepared in lab or extracted from the field, the temperature of sample needs to be elevated to a standard value of 60°C, which is intended to represent the maximum pavement temperature that may be reached in summer (Roberts et al., 2004). For this purpose, two standard curing methods (where curing refers to elevating the asphalt concrete samples to standard testing temperature of 60±1 °C) are generally recommended. The first, which is the prevalent standard method (PSM) used worldwide, involves the specimen being immersed in a water bath with a constant temperature of 60±1 °C (140±2 °F) for 30 to 40 minutes. In the second method, the samples are cured in the oven with a standard temperature of 60±1 °C (140±2 °F) for 120 to 130 minutes (ASTM D6927, 2015).

The influence of water presence in asphalt concrete samples (lab conditions) or in asphalt pavement (field conditions) differs. In the laboratory, the tested samples are immersed in hot water of 60 °C for 30 to 40 minutes and then directly loaded at a continuous constant rate up to failure. During the immersion time, some of the hot water naturally intrudes inside the tested specimens, which may affect the results of Marshall Stability testing. The effect of water in the field, however, basically depends on the reaction of the water to cyclic loading. When an asphalt concrete pavement is saturated, it is subjected to an increasing and decreasing in pore pressure due to the cyclic loading and unloading from the motion of vehicles (Islam clic and Tarefder, 2014). In asphalt concrete pavement, some of the pores are interconnected, permitting the water to move through the pavement. Dynamic traffic loads can thus cause high water pressure within all pores that are filled by water, as shown in Figure 1 (Carl Thodesen and Hoff, 2010).

Figure:1. Pore pressure development in asphalt concrete pavement due to vehicle motion (Varveri et al., 2014)
The heating process for asphalt pavement in the field in summer is mainly due to direct sun heat on the surface layer of the pavement, while the parts below the surface are heated by conductivity without direct contact with the hot air. In the lab, heating is conducted by immersing the tested samples in hot water for 30 to 40 minutes prior to commencing loading. Both the heating methods and loading intensity are thus different from site to lab, so the idea of heating the insulated samples in the lab without allowing direct contact with hot water to better simulate the real conditions in the field by allowing all the parts of the sample to be heated by conductivity seems to offer a reasonable alternative to the two standard curing methods, in which the tested samples suffer from direct contact of hot water or hot air.

In this research, three methods of curing were thus used to elevate the temperature of samples to 60 °C (standard test temperature):

1. Immersing the samples in water for 30 to 40 minutes (Standard Marshall Procedure, also known as the Prevalent Standard Method (PSM)).
2. Putting the samples directly in the oven for 120 to 130 minutes (Alternative standard procedure).
3. Immersing insulated samples (IS) in water for 45 minutes (Proposed procedure).

2. Aim of the Study

The aim of this research is to compare Marshall Test results to determine the influence of the three curing methods on the Marshall stability and flow values of tested samples.

3. Materials and Experimental Work

3.1 Materials

The materials used in this research include aggregate with different particle sizes, filler, and asphalt. The source of the aggregate was the Ifraz query in Erbil, Iraq. The physical properties of the aggregates used in this research are listed in Table 1. Limestone dust was used as a filler in this work, as this is commonly used for HMA production in Erbil; its properties are shown in Table 2. The asphalt cement used, which has a penetration of 40 to 50, is from the Dora refinery, Baghdad; its physical properties are tabulated in Table 3.
Table: 1. Physical properties of the used aggregate

| Test                                | ISSRB* 2003 | Coarse Agg. (12-19) mm | Coarse Agg. (5-12) mm | Crushed Fine Agg. < Sieve No.4 | Natural Sand |
|-------------------------------------|-------------|------------------------|-----------------------|-------------------------------|--------------|
| Los Angles Abrasion value %         | < 30        | 14.6                   | 19.5                  | ----                          | -----        |
| Bulk Specific gravity               |             | 2.65                   | 2.635                 | 2.668                         | 2.455        |
| Degree of crushing %                | > 90        | 96                     | 97.2                  | ----                          | ----         |

*Iraqi Standard Specifications for Roads and Bridges

Table: 2. Properties of the used filler

| Sieve Size mm | Test Method    | Passing Percentage | ISSRB* 2003 |
|---------------|----------------|--------------------|-------------|
| 0.600 [No.030]| ASTM D546-05   | 100                | 100         |
| 0.300 [No.050]|                | 96                 | 95 – 100    |
| 0.075 [No.200]|                | 75                 | 70 – 100    |

Other Engineering properties

| Specific gravity | ASTM D854 | 2.761 |
| Plasticity Index | ASTM D4318-05 | 1.2 | < 4 |

*Iraqi Standard Specifications for Roads and Bridges

Table: 3. Properties of asphalt cement

| No. | Test Properties               | Test designation | Units | Result | Specification ISSRB *(2003) |
|-----|-------------------------------|------------------|-------|--------|-----------------------------|
| 1   | Penetration, 25 °C,100 g,5 sec| ASTM D5-05a      | 0.1mm | 49     | 40/50                        |
| 2   | Softening Point (R&B)         | ASTM D36-06      | °C    | 52     | ---                          |
| 3   | Flash Point                   | ASTM D92-05a     | °C    | 275    | > 232                        |
| 5   | Ductility at 25 °C, 5 cm/min  | ASTM D113-99     | Cm    | > 100  | > 100                        |
| 6   | Specific gravity              | ASTM D70-03      |       | 1.03   |                              |

*Iraqi Standard Specifications for Roads and Bridges

3.2 Experimental Work

A Job Mix Formula (JMF) was prepared from the materials noted above as shown in Table 4. Aggregate gradation was determined according to the midpoint of gradation in ASTM D3515-01(D-5), being the same gradation of the surface course Type IIIA of Iraqi Standard Specifications
for Roads and Bridges (ISSRB) (SORB, 2003). Figure 2 shows the gradation used in this research. The steps of the experimental procedure are exhibited in the flow chart shown in Figure 3.

Table: 4. Job Mix Formula Properties

| Property                        | Results | AASHTO Criteria & SORB2003 |
|---------------------------------|---------|----------------------------|
| Marshall stability (KN)         | 10.8    | Greater than 8             |
| Marshall Flow (mm)              | 3.90    | 2 to 4                     |
| Air Void (%)                    | 4.23    | 3 to 5                     |
| Bulk density (gm/cm^3)          | 2.36    | -                          |
| Optimum Asphalt Content (% of total mix) | 5.2 | 4 to 6                     |

Figure: 2. Gradation of job mix formula (ASTM D3515-01(D5) & SORB, 2003)
Figure: 3. Flow Chart of Experimental Procedure Steps

Start

Collecting and Testing Materials for HMA

Materials within specifications

Yes

Designing Job Mix Formula and Finding OAC for used Gradation

Compacting Marshall Specimens and testing to find the time needed to elevate the Core of Isolated samples to 60 °C

Compacting 18 specimens and curing it by standard method at 60 °C for (30-40 minutes)

Compacting 18 specimens and curing it by Oven method at 60 °C for (120-130 minutes)

Compacting 18 specimens isolated and cured by immersing in water at 60 °C for (40-50 minutes)

Applying Marshall Test Machine to find Stability and Flow of samples

Results and Analysis

End
3.2.1 Preparation and Testing of the Standard Marshall Samples

Three sets of asphalt concrete samples were prepared according to ASTM D6926 - 10. Each set consisted of 18 specimens of 101 mm in diameter and 63.5 mm in height (4 in. diameter and 2.5 in. height) compacted by using 75-blow/face compaction. Every set was divided into two groups: Group No.1 (G1) was comprised of 12 samples prepared only from crushed aggregate, in order to understand the effect of changing any fractions of crushed aggregate on the results of stability and flow, while group No.2 (G2) consisted of six samples prepared by replacing crushed materials with those passing 2.38 mm (No. 8) and 0.3 mm (No. 50) sieves with natural sand. The bulk specific gravity for the specimens was determined according to ASTM D 2726 – 10.

Finally, each sample in each set was cured using one of the three different curing methods mentioned above and all specimens were subjected to the Marshall testing machine to determine their Marshall Stability and flow according to ASTM D 1559 – 82. The air voids of specimens were calculated by applying the Rice method or theoretical maximum specific gravity as per ASTM D2041 – 03 procedures.

3.2.2 Insulated Samples (IS)

In order to discover how much time was sufficient to elevate the temperature of the core of tested samples to standard test temperature (60°C), an additional nine asphalt concrete samples were prepared according to JMF specifications. The centres of these samples were drilled with holes of 4mm diameter up to the mid distance of the total sample height, thus reaching the core. Thermo-couple probes were embedded inside the hole touching the bed of the hole (at depth of 32mm from the surface) and then the hole was refilled with molten asphalt. These samples which wrapped in cling film to create an insulated sample (IS), including a thermo-couple probes, before being immersed in 60°C water. The temperature with respect to time was recorded each 5 minutes using a digital thermometer, as shown in Figure 4.
Figure: 4. Drilling and embedding thermo-couple in the core of insulated asphalt concrete sample prior to wrapping.

The average recorded temperature rise versus time for all IS are shown in Figure 5. From this figure, it can be determined that for the inside temperature for the IS to reach 60°C, 40 to 45 minutes of immersion is required. This duration was thus adopted for curing all IS in 60°C water baths in this research. Once this time is was determined, 18 asphalt concrete samples were compacted and wrapped in cling film to ensure the complete isolation of the samples from the heating water (Fig.6). Before conducting the Marshall test, the insulation film was removed from the insulated and heated samples.

Figure: 5. Temperature rise via time relationship for the core of insulated asphalt concrete samples
3.2.3 Oven cured samples

Following the second standard test method, an oven is used to elevate the sample temperature to 60°C in a dry condition, which creates some difficulties. The main difficulty is adjusting the temperature inside the lab oven to a constant 60°C, as any opening of the oven door causes a dramatic drop of the temperature inside the oven and the available lab ovens were insufficiently accurate to ensure temperature maintenance. Another difficulty was the long time (120 to 130 min) needed to elevate the sample temperatures to the standard value. To overcome some of these difficulties, a standard manufactured instrument as used for rutting tests, shown in Fig.7 and made by the MATEST Company, was used for this purpose prior to conducting the Marshall Test.

Figure:7. MATEST Rutting test instrument used as an oven
4. Results and Analysis

4.1 Stability and flow

Figures 8 and 9 show the mean of stability results for specimens in G1 and G2, respectively, including the error bars of the standard deviation. Table 5 gives the average results for Marshall Stability and flow for each group.

Figure: 8. Average stability in kN for G1 samples subjected to different curing methods

Figure: 9. Average stability in kN for G2 samples subjected to different curing methods
Table:5. Means of Test results for tested groups subjected to three curing methods

| Properties   | Oven Cured (in air) | Insulated water cured | (PSM) Standard curing (in water) |
|--------------|---------------------|-----------------------|----------------------------------|
| Group        | G1                  | G2                    | G1                               | G2         |
| Stability(kN)| 12.88               | 10.6                  | 11.90                            | 9.44       |
| Flow(mm)     | 3.33                | 2.77                  | 3.38                             | 2.81       |
| MSI (kN/mm)  | 3.86                | 3.63                  | 3.52                             | 3.36       |

The stability results of specimens in group G1, for specimens cured by oven, PSM, and insulated curing methods, respectively, were 12.88, 12.49, and 11.9 kN, while, for group G2, the specimens cured by oven, PSM, and IS produced stability values of 10.06, 9.78, and 9.44 kN, respectively. The stability results of G1 were higher than those of G2; the main reason for this variation is due to the use of crushed aggregates in preparing G1 specimens, while in G2 specimens, crushed sand passing 2.36mm and 0.3mm sieves was replaced by natural sand. It is well known that crushed aggregates with angular ends and rough textures lead to higher interlocking between aggregate particles than the rounded ends of natural sand, hence giving lower stability results.

The slightly higher Marshall stability results for oven cured specimens, as compared to PSM, are due to the direct exposure to hot air inside the oven over a long time (120 to 130 minutes), which results in the evaporation of some of the volatile materials from the asphalt component, leading to some aging of the specimens so that they become more stiff and give higher stability results.

The PSM cured samples, immersed in hot water of 60 °C for 30 minutes prior to the test, showed quite high stability results as compared to IS. The presence of additional hot water inside some voids of the PSM samples during Marshall loading may create extra pore pressure, unlike IS samples whose voids are free from any water, leading to slight increases in the Marshall Stability values. It should be mentioned that the loading rate for the Marshall machine is 50.8 mm/min, which is high enough that the trapped water inside a sample cannot be drained quickly.
and easily during load application. Moreover, the amount of hot water absorbed during the curing process was 6.59 grams, compared to 4.6 grams of water at 25 °C, as shown in Figure 10.

![Figure 10. Comparison between absorption of hot and 25°C water by prepared samples](image)

Another empirical concept determined from the Marshall Test, which is sometimes used to distinguish and evaluate asphalt mixtures, is known as the Marshall Stiffness Index (MSI) or Marshall Quotient (MQ) (Chowdhury and Button, 2002; Roberts et al., 2004). MSI is defined as the ratio of Marshall Stability in kN to the sample’s flow in mm. Higher MSI values indicate stiffer mixtures, offering higher resistance to permanent deformation (Zhang et al., 2004). This concept is well known and used by some European engineers and is also used as a standard specification by many transportation agencies including the Ministry of Malaysian Works and the South African National Roads Agency, which have adopted values of 2000 N/mm and 2500 N/mm as a minimum allowable MSI of asphalt concrete mixture in their specifications, respectively (Malaysia, 2008, South African Agency, 2013). Lees (1987) considered that the value of MSI should not be less than 2.1kN/mm, and many researchers have thus adopted this value in their research (Rasel et al., 2011; Sobhan et al., 2011). Fatih Hattatoglu et al. (2015) further used an Artificial Neural Network tool to model the MQ of hot mixes.
The MSI values for the specimens in this test are shown in Figure 11. Here, the MSI values are more than 2.1kN/mm and thus within the specification of Ministry of Works Malaysia (Malaysia, 2008) and as recommended by Lees (Lees, 1987). The MSI results indicate that the samples have appropriate resistance to permanent deformation. However, the oven cured samples showed the highest MSI values among all curing conditions followed by PSM cured samples, while the insulated samples had the lowest MSI values.

![Marshall Stiffness Index (kN/mm)](image)

Figure:11. Marshall stiffness index values for G1 and G2 samples

4.2 Analysis of variance (ANOVA)

In order to verify that the differences observed between the means of the main outputs of the Marshall Test were actually due to the use of different curing conditions to elevate the temperature of samples, and not laboratory artefacts or sample preparation, statistical analysis of variance, ANOVA was conducted using Microsoft Excel with a 95% confidence interval.

Two hypotheses for the two-tailed test were set:
- H1: the curing condition has a significant effect on the output of the Marshall Test
- H0: the different curing conditions produce similar results.

The proposed hypothesis would be accepted if the $P$-value were smaller than the significance level ($\alpha$), which in this study is 0.05 as long as the F-statistic is greater than the F-critical value. Otherwise, the null hypothesis is accepted. Tables 6 to 9 present the variance analyses of the
stability and flow of different groups based on curing condition. It can be seen from those tables that the $P$-values are greater that the significance level (0.05) and that the F-stat values are smaller than F-crit. Thus, the proposed hypothesis must be rejected, and the different curing conditions used to elevate the sample temperatures assumed to not have a significant effect on the output of the Marshall Test.

Table: 6. ANOVA single factor between the different curing conditions and stability values of G1.

| Source of Variation | SS     | df | MS          | F-stat | P-value | F crit |
|---------------------|--------|----|-------------|--------|---------|--------|
| Between Groups      | 5.38197| 4  | 2.690986973 | 1.92324925 | 0.163724 | 3.31583 |
| Within Groups       | 41.9756| 4  | 1.399187838 |         |         |        |
| Total               | 47.3576| 1  | 32          |         |         |        |

Table: 7. ANOVA single factor between the different curing conditions and stability values of G2.

| Source of Variation | SS     | df | MS          | F-stat | P-value | F crit |
|---------------------|--------|----|-------------|--------|---------|--------|
| Between Groups      | 1.12976| 6  | 0.564883239 | 0.71160489 | 0.506698 | 3.68232 |
| Within Groups       | 11.9072| 4  | 0.79381584  |         |         |        |
| Total               | 13.037 | 17 |             |         |         |        |

Table: 8. ANOVA single factor between the different curing conditions and flow values of G1.

| Source of Variation | SS     | df | MS          | F-stat | P-value | F crit |
|---------------------|--------|----|-------------|--------|---------|--------|
| Between Groups      | 0.0530242 | 2  | 0.0265121   | 0.066477722 | 0.93582 | 3.31582 |
| Within Groups       | 11.964363 | 30 | 0.3988121   |         |         |        |
| Total               | 12.0173878 | 8  | 32          |         |         |        |

Table: 9. ANOVA single factor between the different curing conditions and flow values of G2.

| Source of Variation | SS     | df | MS          | F-stat | P-value | F crit |
|---------------------|--------|----|-------------|--------|---------|--------|
| Between Groups      | 0.03694444 | 4  | 0.018472222 | 0.042114836 | 0.958873 | 3.68232034 |
| Within Groups       | 6.57923333 | 15 | 0.438615556 |         |         |        |
However, replacing the crushed stone with natural sand leads to a significant effect on the stability and flow values as measured in the Marshall test, as can be seen from the $P$-values in Tables 10 and 11. The $P$-values in tables 10 and 11 are smaller than the significance level (0.05), and the F-stat values are greater than F-crit.

Table: 10. ANOVA single factor between the crushed stone and natural sand on stability values.

| Source of Variation | SS       | df | MS    | F-stat               | P-value   | F crit |
|---------------------|----------|----|-------|----------------------|-----------|--------|
| Between Groups      | 83.0896537 | 8  | 83.09 | 67.413182            | 9.12E-11  | 4      |
| Within Groups       | 60.3946131 | 6  | 1.233 |                      |           |        |
| Total               | 143.484266 | 9  | 50    |                      |           |        |

Table: 11. ANOVA single factor between the crushed stone and natural sand on flow values.

| Source of Variation | SS       | df | MS    | F        | P-value   | F crit |
|---------------------|----------|----|-------|----------|-----------|--------|
| Between Groups      | 3.08969709 | 1  | 3.08969 | 8.124862 | 0.00637   | 4.03839 |
| Within Groups       | 18.6335657 | 49 | 0.38027 | 7        |           |        |
| Total               | 21.7232627 | 50 |       |          |           |        |

5. Conclusions

The following conclusions can be derived from this research:

1- Marshall Test results indicated that oven cured samples show the highest stability values, followed by PSM cured samples, while the insulated samples exhibited the lowest stability values. However, ANOVA testing showed that these differences were not statistically significant.

2- The intrusion of hot water into PSM samples increased the stability values as compared with insulated samples.
3- The oven cured samples become stiffer due to the evaporation of volatile materials, which gave higher stability values and lower flow as compared with samples cured by other two methods, resulting in higher MSI values.

4- The suggested curing method, using insulated specimens, seems to simulate real conditions in the field by eliminating hot water absorption (in PSM) and hot air effects as in the second standard curing method.

5- The Marshall Stability values gained by using the prevalent standard curing method PSM are higher than those of actual state (insulated) samples by approximately 5%, which is a considerable percentage considering highway agency requirements.

6. **Recommendations:**

The main recommendations from this research are thus

1- Further research regarding the effect of curing methods on Marshall stability and Stiffness Index is highly recommended to consider different types of asphalt mixture parts such as binder and base course mixes.

2- Using insulation in curing the asphalt concrete specimens in the lab offers more reliable Marshall Stability results than the other curing methods.

3- If the PSM is used, in order to account for the reduction in stability, it is recommended to increase the minimum stability values by 1 KN over specification requirements.

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