# Design of Warm Mix Asphalt (WMA) Using an Amino-Based Resin

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**Abstract:** Rising energy prices, global warming and environmental awareness, in production of traditional asphalt (Hot Mix Asphalt) have resulted in the innovation of a production technology named Warm Mix Asphalt (WMA) as a mean to decrease the energy consumption and emissions. In this technology, additives work in different ways to either reduce the viscosity of the bitumen or to allow better workability of the mix at lower temperatures. In recent years widespread researches have been done to develop new and economical technologies of WMA production, however, study of Iranian technologies to produce WMA are significantly lacking for using WMA in Iran.

The Objective of this study is to analysis using an amino-based resin in order to produce of WMA. This resin, which is thermoset and produce in Iran, was used as an additive to decrease the viscosity of bitumen. This resin was used amount of 5%, 10% and 15% the weight of bitumen. The materials included one type of bitumen (AC 60-70) and one type of aggregate (Lime stone). Tests included dynamic/shear rheometer, bending beam rheometer, Marshall Test, moisture sensitivity, indirect tensile strength and resilient modulus tests. The results of the experiments indicated that the addition of amino-based resin can decrease the viscosity of bitumen by producing foam. This reduction of viscosity leads to reduce the temperature mix asphalt production as much as 15°C in application of 10% resin. It is also concluded that WMA had acceptable engineering properties.

**Keywords:** Warm mix asphalt, amino based resin, energy consumption.

## 1. INTRODUCTION

Now a day global warming and greenhouse effect become significant issue through environmental and scientific societies all over the world. One of the most important challenges facing societies is the efficient and economic use of energy, with the corresponding reduction in the emission of greenhouse gases. Large amount of fuel and energy is consumed by Process of producing hot mix asphalt (HMA); which is lead to emission of greenhouse gases specially $CO_2$. Due to sustainable development in asphalt industry and control air pollution and resource consumption, warm mix asphalt (WMA) technology is designed by innovative idea [1-8].

WMA technology is based on producing and paving asphalt at lower temperature than traditional asphalt (HMA) with same quality of pavement. In this technology, additives work in different ways to either reduce the viscosity of the bitumen or to allow better workability of the mix at lower temperatures. Figure **1** shows a classification of various application temperatures for asphalt concrete, from cold mix to hot mix. As shown in Figure **1**, the mix temperature of WMA is between 100 to 140°C in comparison with the mix temperature of HMA that is between 140 to 170°C [2, 8].

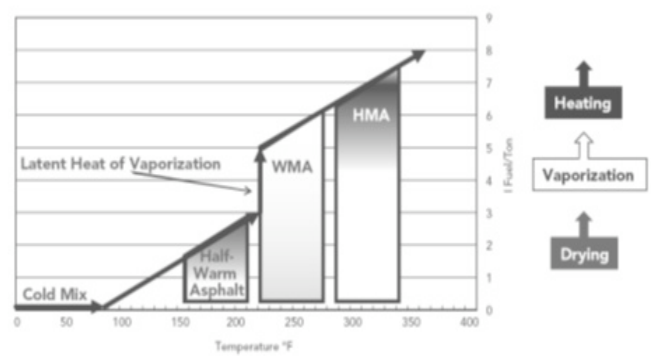


**Figure 1:** Classification of asphalt technology based on temperature range [2].

The technology of WMA is birthed by the Kyoto protocol and its primary activity began in Europe. Since then, it gradually gained reputation all over the world and several processes and considerable research programs on WMA have been developed and conducted. Former studies have indicated that the advantages of WMA are briefly summarized as follow [3, 8]:

- Lower plant emission and fumes.
- Reducing energy consumption and saving costs.

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E-ISSN: 2311-8717/15 

- Improve workability and compaction efficiency.
- Quickening turnover to traffic due to reduced cooling time.

WMA technologies can be classified mainly based of additive to four groups: organic additives, chemical additives, water-bearing additives and water-based processes. These technologies are based on reducing the viscosity of bitumen [1-13].

The objective of this study are to evaluate using a new additive made of an amino-based Resin in order to produce WMA *via* reduction of the bitumen viscosity. The resin was water–base thermoset resin. The following steps show experimental design used in this study:

- Step 1: Determining properties of materials (*via* initial tests),
- Step 2: Selecting optimum percentage of bitumen in mix asphalt (*via* Marshall Test).
- Step 3: Analysing effect of temperature of mixing and percentage of resin (3 temperatures: 110, 125,
- 140°C/four percentage of resin: 0, 5, 10 and 15%). Selecting optimum temperature of mixing and percentage of resin (*via* Marshall Test),
- Step 4: More Analysis on effect of temperature of mixing and percentage of resin (optimum temperature / four percentage of resin: 0, 5, 10, 15%), (*via* ITS, Resilient modulus, Moisture Sensitivity, DSR and BBR test).

## 2. MATERIALS AND METHODS

### 2.1. Aggregate

One types of aggregate have been used in this study that is limestone aggregate. The engineering properties of aggregates is shown in Table **1**.

**Table 1: Engineering Properties of Aggregates used in this Study**

| Abrasion Lost (Los Angeles) (%) | Compressive Strength (kg/cm$^3$) | Absorption of Water (%) | Specific Gravity (g/cm$^3$) |
|---|---|---|---|
| 23 | 400 | 0.96 | 2.61 |

The gradation of the test specimens was performed in accordance with ASTM D3515. The gradation curve in ASTM D3515 standard is based on minimizing of void in mixture Figure **2**. Illustrates the gradation of aggregates and the results of sieve analysis of materials for making asphalt mixtures are given in Table **2**.

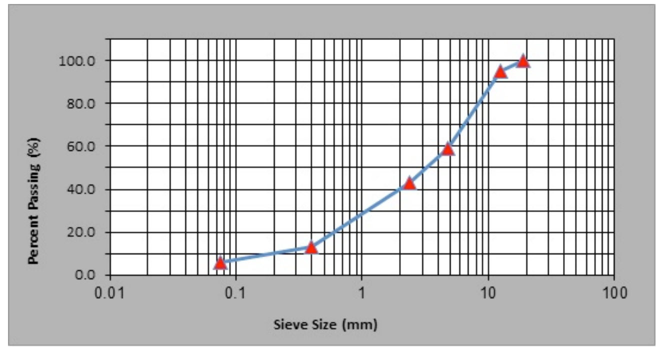


**Figure 2:** Aggregate gradation curves.

**Table 2: Sieve Analysis for Making Asphalt Mixtures on the Basis of Two Types of Gradations**

| Sieve size (mm) | 19 | 12.50 | 4.75 | 2.36 | 0.3 | 0.075 | ≤0.75 |
|---|---|---|---|---|---|---|---|
| Percent remain (%) | 0 | 05 | 36 | 16 | 30 | 07 | 06 |
| Weight (g) | 0 | 60 | 432 | 192 | 360 | 84 | 72 |

### 2.2. Bitumen

The bitumen used in this study was prepared from Isfahan oil Refinery, Isfahan, Iran. A series of bitumen tests was done to determine properties of beat bitumen (base bitumen and bitumen that were modified by resin). The percentage of resin that were mixed with bitumen was different (5%, 10% and 15% the weight of bitumen).

#### *2.2.1. Ductility*

The sample preparation and the tests were performed in accordance with ASTM D113-79. Samples contained resin were prepared in 3 blending temperatures (110°C, 125°C and 140°C) and after that they were experimented in test temperature.

#### *2.2.2. Penetration*

Penetration Test (ASTM D5-73) evaluates the hardness or softness of bitumen by measuring the depth in tenths of a millimeter to which a standard loaded needle, with a total weight of 100 g, will penetrate vertically in 5 seconds. All tests were conducted at the temperature of 25°C.

#### *2.2.3. Softening Point*

Softening point test was conducted using Ring and Ball apparatus in accordance with ASTM D36-76.

#### *2.2.4. Flash Point*

BIS 4689 defines the flash point as the temperature at which the vapor of bitumen momentarily catches fire in the form of ash under specified test conditions. This test was carried out according to ASTM D 92 for all treatments.

#### *2.2.5. Dynamic/Shear Rheometer (DSR)*

In order to determine the high temperature rheological properties of the bitumen due to the addition of the resin, the base bitumen and modified resin were tested in a Dynamic Shear Rheometer (DSR) according to AASHTO T 315. In this study, Measurements were carried out in a stress controlled rotational DSR with parallel plate sample geometries of 25mm diameter and 1mm gap (with manual gap compensation at each test temperature). Each bitumen was measured in terms of the complex shear modulus (G*) and phase angle (δ) values; starting from 50°C until failure in accordance with Superpave mix design specifications.

#### *2.2.6. Bending Beam Rheometer (BBR)*

The Bending Beam Rheometer (BBR) test provides a measure of bitumen low temperature PG grade and these parameters give an indication of an asphalt bitumen ability to resist low temperature cracking. In this study, this test was carried out according to AASHTO PP 42.

### 2.3. Resin

The liquid resin used in this study was prepared from Fars Chemical Industries (FCIC, Shiraz, Iran). The resin is a water-base thermoset resin and belongs to a class of thermosetting resin that is the reaction product of a primary or a kind amine in which having at least two functional amines or amid groups and an aldehyde. This class of macromolecules with terminal amine or hydroxyl functional group with structure of R-NH-$(CH2O)_n$-NH-R or R2N-$(CH2O)_n$-NR2 is the best replacement for resins such as epoxies and vinyl esters.Compared to other polymers, this modified amine-based resin possesses some advantages, including fast curing without addition of cross linking agent, lower dosages of polymer to achieve reasonable result, good solubility in bitumen and lower price than the other bitumen additives.

It was analyzed by 13C-NMR analysis. The liquid 13C-NMR spectrum of the resin was obtained on a Bruker MSL 400 FT-NMR spectrometer. Figure **3** shows the 13C-NMR spectra of the amine-based resin. Furthermore, Chemical Properties such as viscosity determined by several tests. The nonvolatile solid content was determined by the measurement of the weight of the resin before and after drying. The pH of

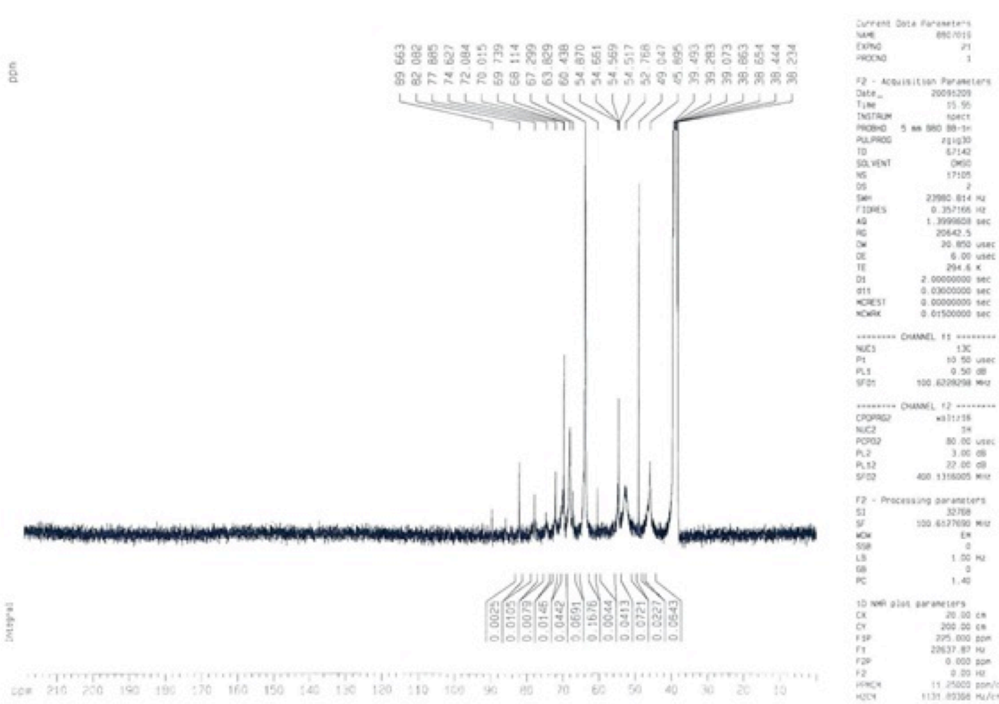


**Figure 3:** 13C-NMR Spectrum of Amino-based Resin.

the resin after synthesis was measured by an electronic pH meter at 25°C. The viscosity of the resin at 25°C was measured by a cone plate viscometer (DV-II1, Brookfield, Middleboro, MA) with a No. 2 spindle at 60rpm. The results of the tests have been presented in Table **3**.

**Table 3: Chemical Properties of the Amino-Based Resin**

| White Liquid | Appearance |
|---|---|
| 50~55 | Solid Content /wt. % |
| 1.100-1.060 | Specific gravity; 20°C |
| 8~9 | PH; 25°C |
| 140~200 | Viscosity (cp); 25°C |

### 2.4. Mixture Asphalt

#### *2.4.1. Marshall Test*

In this study, the Marshall method was used to determine optimum bitumen content, and to compare different samples, as well. Marshall Specimens were made and tested according to ASTM D1559.

For making Marshall Specimens, first, the aggregates were kept in a heater for 24 hours at mixing temperature (110°C, 125°C and 140°C). Then, the bitumen was heated to mixing temperature and finally, aggregates, bitumen and resin were blended with each other. It was observed that as the resin was added to pre-heated bitumen, the mixture viscosity would immediately drop off that increased the specific surface of the bitumen. This phenomenon allows intimate coating of aggregates. A mixer mixed bitumen and aggregates until whole aggregates seemed to be impregnated with bitumen and a homogeneous mixture appeared. According to each treatment specification, the temperature was controlled during this process. Then, the mixture was placed in a Marshall mold and compacted by applying 75 blows on each side of the specimen. The standard dimensions of the samples were 63.5mm in height and 101.5mm in diameter.

After making them cool at the room temperature for one day, Specific gravity test was carried out in accordance with the ASTM D 1188. Consequently, the weights in the air and in water were obtained for each sample and then according to Archimedes formula the specific gravity was calculated through:

$$SG_{AC} = W_D / (W_D – W_W) \quad (1)$$

$W_D$ and $W_W$ are weights of AC in the air and in water, respectively. In the final stage, they were left in water at 60°C for 30min. Consequently they were tested with Marshall Test apparatus. Three specimens were carried out for each treatment.

#### *2.4.2. Indirect Tensile Strength (ITS) Test*

The indirect tensile strength test (ITS) is performed at loading rate of 51mm/min by using the Marshall apparatus. The ITS test involves loading a cylindrical specimen with compressive loads that act parallel and loading the vertical diametrical plane. The ITS test is carried out to define the tensile characteristics of the asphalt concrete which can be further related to the cracking properties of the pavement. In order to compute the ITS, the following equation is used:

$$ITS = 2P_{max}/\pi td \quad (2)$$

$P_{max}$ is the maximum applied load (kN), t is thickness of the specimen (mm) and d is diameter of the specismen(mm).

#### *2.4.3. Resilient Modulus Test*

Resilient modulus of asphalt mixtures, measured in the indirect tensile mode (ASTM D 4123), is the most popular form of stress-strain measurement used to evaluate elastic properties. The resilient modulus along with other information is then used as input to the elastic theories of model to generate an optimum thickness design. Three laboratory fabricated specimens are tested in order to determine the resilient modulus of that asphalt mix. Each of the specimens is tested twice (the orientation of the specimen of the second test is 90° from the first test) producing a total of six measured resilient modulus values.

In this study, the resilient modulus of mixtures has been measured at 25°C and loading frequency of 1 Hz. Moreover, it has been assumed that the Poisson's ratio is 0.35. The resilient modulus of elasticity, E, is calculated by the following equation:

$$E = P(\upsilon + 0.27)/t\Delta H \quad (3)$$

P is repeated load (N), v is the Poisson's ratio (that has been supposed 0.35 in this study), ΔH is horizontal deformation (mm) and t is thickness of specimen (mm).

#### *2.4.4. Moisture Sensitivity Test*

Moisture sensitivity test were tested according to ASTM D1075-49. Two groups of compacted specimens were used in this test. One group was submerged in water at 60°C for 24 hour for conditioning, and the other group was maintained dry. Compressive strength was measured on specimens of both groups at 25°C at

**Table 4: Ductility of Mixtures of bitumen and Resin**

| 140 | | | 125 | | | 110 | | | Base | Blending Temperature (°C) |
|---|---|---|---|---|---|---|---|---|---|---|
| 15 | 10 | 5 | 15 | 10 | 5 | 15 | 10 | 5 | 0 | Percentage of Resin |
| 65 | 77 | 88 | 56 | 60 | 100< | 100< | 100< | 100< | 100< | Ductility (cm) |

a deformation rate of 0.05 in/min per inch of height. For a 4-in.-tall specimen, the rate would be 0.2 in/min. The average strength of conditioned specimens divided by that of dry specimens is used as a measure of moisture sensitivity of the mix. Most agencies have used a 70% ratio as a passing limit.

## 3. RESULTS AND DISCUSSION

### 3.1. Bitumen

#### *3.1.1. Ductility*

As it was stated, this test was done on base bitumen and Samples containing 5%, 10% and 15% resin were prepared in 3 blending temperatures (110°C, 125°C and 140°C) and after that were experimented in test temperature. Table **4** shows the ductility values for all samples.

From Table **4**, it can be found that by increasing the resin content, the ductility of bitumen will be decreased. The possible reason is due to thermoset properties of the resin that cures after a short time. In addition, by increasing the temperature, the ductility will decrease because of increasing curing-time of the resin.

#### *3.1.2. Penetration*

Samples of this test were prepared similar to ductility test and then tests were conducted at the temperature of 25°C. The experimental results are presented in Figure **4**.

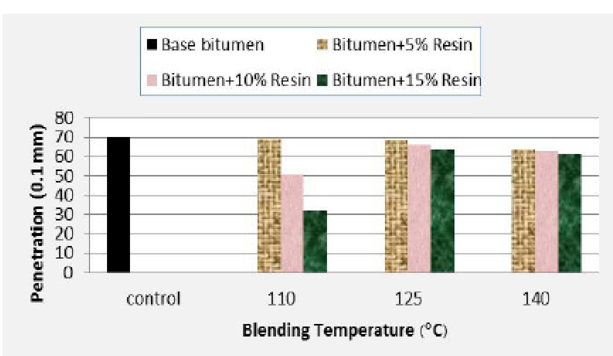


**Figure 4:** Penetration values of bitumen.

Figure **4** shows that by increasing mixing temperature and resin content, the penetration of the modified bitumen generally will decrease. This may be due to increase in bitumen stiffness by increasing temperature and resin content. This reduction of penetration is more sensible in 110°C. The possible reason is that the resin impression is more drastic in lower temperatures which bitumen has more viscosity and resin impression is lesser in higher temperature which bitumen viscosity is less.

#### *3.1.3. Softening Point*

Similar by 2 previous tests, samples were prepared and test results are shown in Figure **5**.

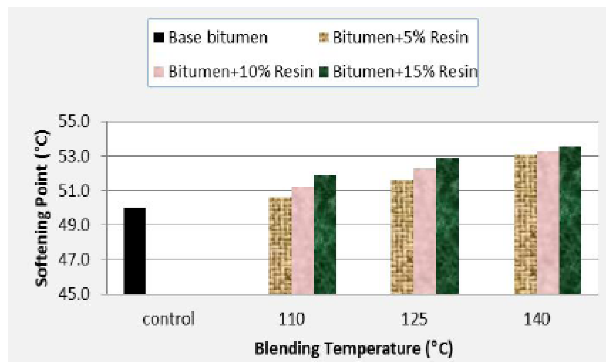


**Figure 5:** Softening Point values of bitumen.

The same result by penetration test can be obtained from Figure **4** in the case of softening point.

#### *3.1.4. Flash Point*

This test was also done on base sample and samples were prepared in 3 blending temperatures (110°C, 125°C and 140°C) and three different percentage of resin (5, 10 and 15 percent). The results of the experiments are illustrated in Figure **6**.

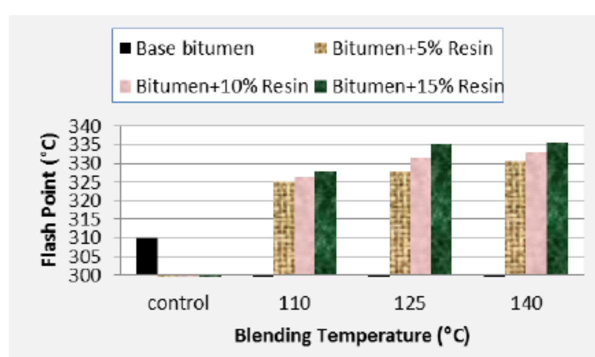


**Figure 6:** Flash Point values of bitumen.

Figure **6** implies that flash point can be improved if resin content and blending temperature increase. This may be due to increase of molecular weight of resin modified samples.

### *3.1.5. Dynamic/Shear Rheometer (DSR)*

The grade determination feature of the DSR was used to determine the failure temperature in the original unaged state. Samples which were tested were base bitumen and bitumen was mixed with resin (5%, 10% and 15% resin) in 125°C (minimum acceptable temperature to produce WMA). This procedure tests the sample at a starting temperature (50 °C) and if the G*/sin **δ** value is greater than the 1.000kPa (value required by AASHTO M320) temperature increases the next PG grade (64 and 70°C). After recording all of the data, the failure temperature is determined through interpolation of temperature at which the G*/sin **δ** value is less than the required value. Three replicates were tested for each aging condition of each bitumen. Figure **7** shows that the addition of resin results in an increase in failure temperature. The most increase belongs to when 10% resin was added to the bitumens and after that although adding resin (15%) decreases failure temperature, this temperature is more than temperature of base bitumen. In addition, based on the complex modulus (G*) and phase angle (**δ**) values; Figure **8** indicates that regardless of the starting temperature of 50°C or higher test temperatures, the bitumen containing 5 and 10% resin have the higher G*/sin **δ** value than samples. When amount of resin increases to 15% resin, G*/sin **δ** value drops and shows lowest value. Furthermore, both neat bitumen and the bitumen content resin have the G*/sin **δ** value less than 1.000kPa at 64°C. As a result, the addition of resin does not have much influence on PG grade of bitumen and contributes to a slight improvement of rutting resistance at a high performance temperature.

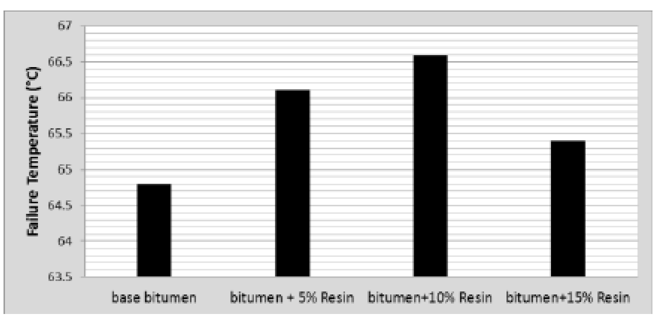


**Figure 7:** Failure temperature of bitumen.

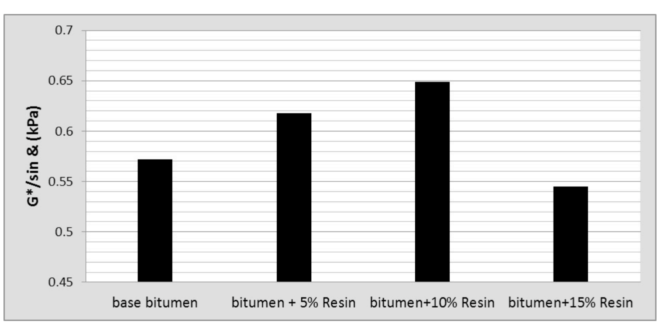


**Figure 8:** G*/sinδ of bitumen.

### *3.1.6. Bending Beam Rheometer (BBR)*

As it were mentioned, results of Bending Beam Rheometer tests give an indication of an asphalt bitumen's ability to resist low temperature cracking and so it determines low performance grade. Samples of this test were prepared similar to DSR test and results are shown in Table **5**. Although Table **5** illustrates that the addition of 05 and 10 percent Resin additives does not have influence on bitumen's low temperature PG grade, addition of more than 10 percent decreases ability to resist low temperature cracking.

**Table 5: Low Temperature PG Grade**

| Mix Type | Temperature (°C) |
|---|---|
| Base bitumen (without Resin) | -22 |
| Bitumen + 05% Resin | -22 |
| Bitumen + 10% Resin | -22 |
| Bitumen + 15% Resin | -16 |

## 3.2. Mix Asphalt

### *3.2.1. Determine Optimum Bitumen Content*

To determine optimum bitumen content, Marshall Specimens including various percentages of bitumen (4, 4.5, 5, 5.5, 6 and 6.5 percent) were prepared by the total mass of asphalt mixture and then this content was determined based on the Marshall Stability, flow and specific gravity. Marshall Test results are shown in Figures **9-11**.

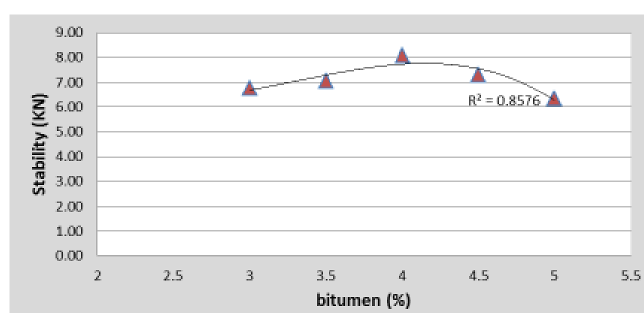


**Figure 9:** Marshall Stability for various percentage of bitumen.

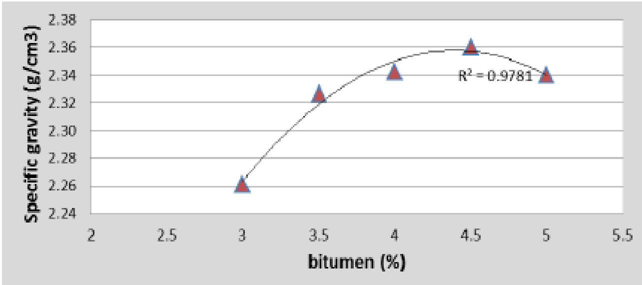


**Figure 10:** Specific gravity values for various percentage of bitumen.

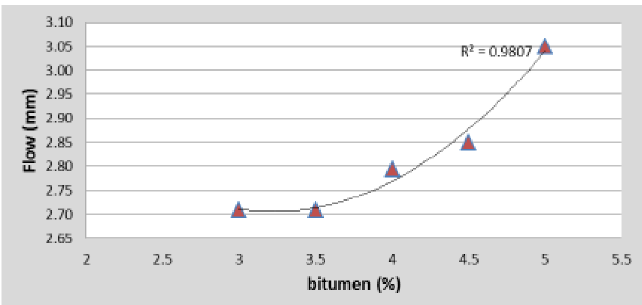


**Figure 11:** Flow values for various percentage of bitumen.

So, it can be concluded that Marshall Specimen four percent asphalt content may be the optimum asphalt content.

### *3.2.2. Analysis of the Effect of Mixing Temperature and Percentage of Resin*

After that the optimum percentage of bitumen was obtained (4.0%), two tests were conducted to determine the effects of percentage of resin and temperature on asphalt-concrete (AC) samples characterizations. In order to do it, the experimental variables were designed for three percentages of resin (5%, 10% and 15%) and 3 mixing temperatures of 110°C, 125°C and 140°C. As mentioned, each treatment was repeated three times and each result shows the average of three test specimens. Figures **12-14** shows the Stability, flow and specific gravity results for all samples, respectively.

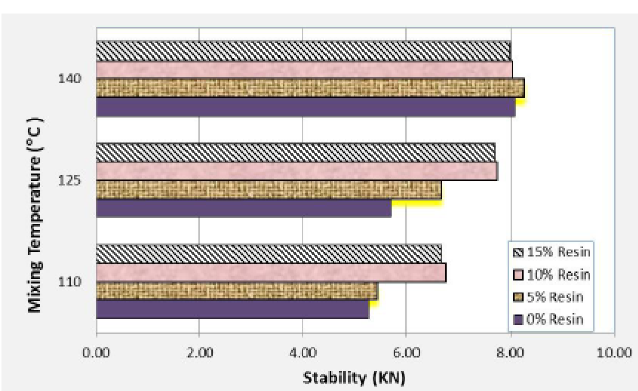


**Figure 12:** Marshall stability values.

Figure **12** illustrate that decreasing mixing temperature reduce stability but when resin was added to the mix, the stability, as expected, was improved. This impression of resin is watched in 110°C and 125°C, while stability results in 140°C are almost similar in the different percentages of resin. This may be due to water in resin that produces foam bitumen in time of mixing, so decrease viscosity of bitumen and as a result increases coating of aggregates in asphalt mix, but in 140°C, since bitumen has favorite viscosity itself, impression of resin is not very tangible. In addition, samples that are made in 125°C, have stability near to minimum Marshall stability, i.e. 8KN according to Iranian Highway Standards.

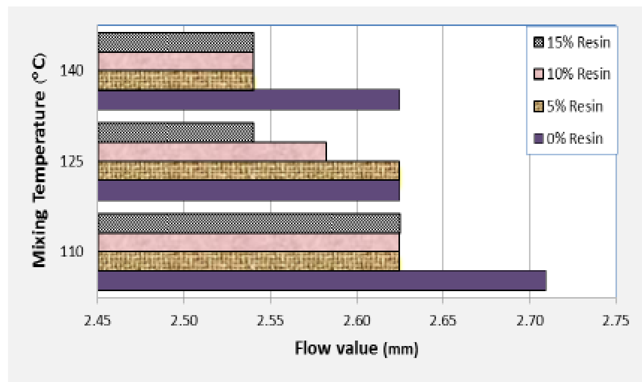


**Figure 13:** Flow values.

Flow values; see Figure **13**, show reduction of samples flow with rise of mixing temperature. In similar temperature, samples flow that is made with resin is less than neat samples (without resin) but this reduction is not very considerable. This may be due to thermoset property of resin. The other point that could be gotten from Figure **13** is that all samples have flow within standard limits, i.e. 2-3.5 according to Iranian Highway Standards.

Figure **14** shows that there is correlation between stability Marshall and specific gravity.

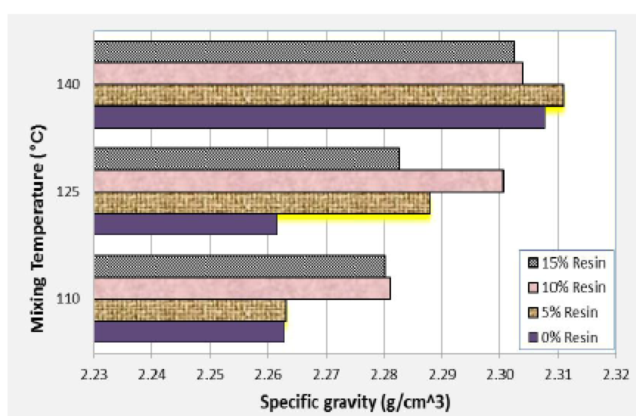


**Figure 14:** Specific gravity values.

As it was mentioned, resin which was used in this study has thermoset property and will stiff after any time. It seems that this property will cause cohesion between bitumen and aggregates to increase. For this aim, in later step, Marshall Test was done for some samples which were prepared in 125°C after one week from time of production of samples. The reason for this time span is to complete curing process. So resin will be stiffed after this process and it won't return to previous. Results are shown in Figure **15**.

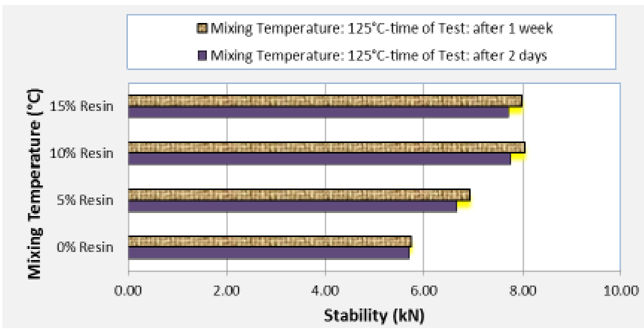


**Figure 15:** Marshall Stability values for samples prepared in 125°C (analysis of the effect of thermoset property).

From Figure **15** it can be found that stability of resin modified samples will increase when test is done after a more time span from time of production of samples. This increase in resin modified samples is between 3.6% to 4% while in sample without resin is less than 0.08%.

For more analyses of the effect of mixing temperature and percentage of resin, the others properties of mix asphalt were determined. These properties were indirect tensile Strength, resilient modulus and moisture sensitivity.

ITS test was done for samples prepared in 125°C and the control sample (without resin and mixing temperature of 140°C). ITS results are shown in Figure **16**.

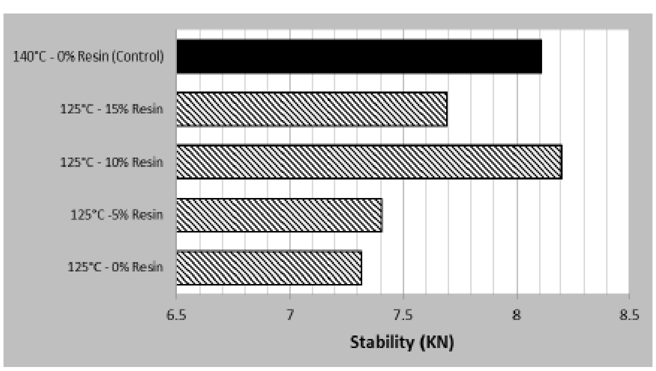


**Figure 16:** ITS values (Stability).

Figure **16** illustrate, as expected, indirect tensile strength drops when mixing temperature is decreased from 140°C to 125°C. This strength increased when resin was added to mixture. In addition, sample modified by 10% resin has the highest value of strength in comparison with other samples.

Resilient modulus test similar to ITS test was done for samples prepared at 125°C and the neat sample (without resin and mixing temperature of 140°C). Results are shown in Figure **17**.

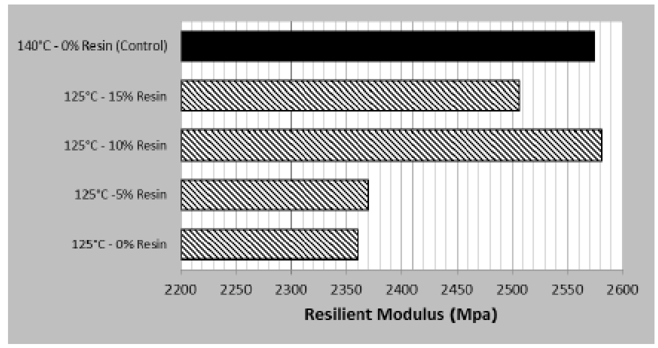


**Figure 17:** Resilient modulus values.

Figure **17** shows that the resilient modulus will decline at 125°C, It seems that the lack of flow ability of bitumen at 125°C leads to this phenomenon. Resilient modulus of the sample with 10% resin offers more amount than that of HMA prepared at 140°C (Control sample) while sample with 5 and 15% resin offers less amount than that of control mixing. Base on these results, the best stability value belongs to sample modified by 10% resin.

Moisture Sensitivity was compared among samples prepared at 125°C and whit 10% resin and without resin. Results are shown in Table **6**.

**Table 6: Moisture Sensitivity Results**

| Scale of Moisture Sensitivity (Stability(Wet)/Stability(Dry)) | Stability (KN) | Sample |
|---|---|---|
| 0.85 | 5.70 | 125°C - 0% Resin (Dry) |
| | 4.86 | 125°C - 0% Resin (Wet) |
| 0.96 | 7.75 | 125°C - 10% Resin (Dry) |
| | 7.46 | 125°C - 10% Resin (Wet) |

Results show that prepared mixing asphalt has enough resistance against moisture because their Scale of moisture sensitivity is more than its minimum limit, i.e. 0.75 according to Standard. In addition, it can be found that moisture resistance of sample by resin offers more value than sample without resin which may be because of thermoset property of resin. In fact, thermoset resin isn't returnable by moisture and heat after curing.

## 4. CONCLUSION

The following conclusions can be drawn from this limit study based on the experimental data and on their analysis:

- Using of an amino-based resin results in a slight increase in failure temperature, and has a slight effect on PG grade of bitumens and contributes to slight improvement of rutting resistance at a high performance temperature. In addition, although the thermoset property of resin changed some properties of bitumen such as decreasing of penetration point, bitumen modified by this resin have often similar treat like treat in low temperature grade when percentage of resin is less than 15%.

- This particular additive significantly reduced the viscosity of the bitumen and so decrease production temperature of asphal about 15°C. In addition, best percentage of resin to add mixture is 10% in base of tests.

- However both of samples with resin and without resin have enough resistance against moisture, sample which is made by this additive has better performance.

- Use 10% of this additive increases the Resilient Modules, indirect tensile Strength of the WMA in comparison with the HMA.

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Received on 04-06-2015 Accepted on 02-07-2015 Published on 31-07-2015

DOI: http://dx.doi.org/10.12974/2311-8717.2015.03.01.2