



Investigating the Engineering Properties of Half-Warm Asphalt Mixes Using Chemical Additives

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ABSTRACT

Most of road networks are constructed using hot mix asphalt (HMA). HMA is used for roads construction and maintenance at high temperature (150-170°C) requiring high fuel consumption. In addition, gases emissions generated from HMA are issues of concern among the pavement researchers. Therefore, reducing gases emission and mixing temperature is an objective toward sustainable asphalt pavement industry. Thus, using warm or half-warm asphalt mixes as a modern technique are considered suitable alternatives to HMA. This study aims to reduce gasses emissions and manufacturing cost of HMA using half-warm mixes asphalt (HWMA). Chemical additives are used in order to produce HWMA at low temperature. In this study, Ethoxylated Nonyl Phenol (NP9) was used to enhance the performance of asphalt mixes at low temperature. The tried percentages of NP9 were 10, 15, 20, and 25% (by weight of bitumen). The tested mixes include control mix (mixed at 155°C) and modified mixes (mixed at 90°C, HWMA modified by NP9). Rolling thin film oven (RTFO), Marshall, moisture susceptibility, indirect tensile strength (ITS), wheel tracking, scanning electron microscopic (SEM) and gases measurements tests were carried out for control and modified mixes. The results indicated that, significant changes were occurred in modified mixes compared to the control mixes. Bitumen viscosity decreased by the increase of NP9 percentages. The optimum percentage of NP9 was 10% by weight of bitumen. The retained penetration of modified specimens by 10% NP9 was higher than conventional specimens by 40.26%. Also, the results showed that Marshall stability and Marshall Quotient (MQ) of HWMA enhanced by 15.22% and 11.58% respectively. Producing HWMA reduced the loss of stability by 18.10% and changed tensile strength ratio from 91% to 96% by an enhancement of 5.50% compared to the conventional mixes. In addition, the resistance of HWMA specimen against rutting was higher than or as same as the value of control specimen during the test period. From SEM test results, a significant change was observed for all tested specimens, the structure of control specimen under SEM was differ than the shape of modified specimens. Finally, the chemical analysis showed that the gases emissions reduced by manufacturing HWMA at 90°C.

1. Introduction

Due to the high extension in road networks over the world, therefore, the main concerns in producing asphalt mixes are quality and cost. The modern

techniques focuses on manufacturing asphalt pavement with low impact on the environment along with savings in energy [1,2]. Also, the main advantages of the modern techniques are energy conservation, saving costs, enhancing engineering

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properties and reducing the harmful environmental impacts. Flexible pavement production has a bad effect on the environment. Previous studies introduced other technologies like warm mix asphalt (WMA) to reduce the mixing and compaction temperatures of asphalt mixes [3]. WMA can produce asphalt mixes with strength and durability as same as or better than HMA [4]. WMA are produced at lower temperatures using chemical additives which enhance the workability of the mixture by changing the binder viscosity, which leads to increase the volume of the binder. Adding these additives reduces the binder viscosity and increases the bitumen flow which help to the complete coating of aggregates with bitumen at lower temperatures [5-7]. In addition, it should be mentioned that the amount of the used additives depends upon its type and impact on mix performance [8-11]. The previous studies listed various types of additives used in manufacturing WMA; the common types of these additives are aspha-min and Sasobit. Sasobit is a type of paraffin, which has a long chain aliphatic hydrocarbon with chain lengths of 40 to 115 carbon atoms, melts in the asphalt binder at temperatures (85 - 115°C) to reduce the mixing and compaction temperatures by 30 to 50°C. It has high viscosity at lower temperatures and low viscosity at high temperatures. Sasobit forms a crystalline network structure in the asphalt binder [12], Fig.1(a) shows Sasobit particles. Sasobit contains the organic additives are mixed with asphalt binder, which melts at about 100°C and these chemically change the temperature viscosity behavior of the asphalt binder, so the mix remains workable at a low temperature [13]. Several previous studies evaluated the use of Sasobit in producing warm asphalt mixes, and concluded that the engineering properties of asphalt mixes have been enhanced [14-16]. Fig.1 (b) shows Aspha-min shape, it is a fine powder, which creates foaming to asphalt binder; the lubricating action keeps the mix workable at a temperature range between (120-135)°C . When it is added at the same time as the binder, crystallized water is released, this creates a foaming effect that leads to a slight increase in binder volume and reduces the binder's viscosity. Aspha-min is a synthetic sodium aluminum silicate, also referred to as zeolite [17].

Asphalt mixtures are normally divided into four categories, HMA, cold mix asphalt (CMA),WMA and half warm mix asphalt (HWMA) as shown in Fig.2.In general, HMA properties are much better than cold mix asphalt, so HMA is being used for the higher volume traffic. The cold mix asphalt needs a curing time to open up to traffic. Therefore, the cold mix asphalt is not used for higher traffic volume

roads. To overcome some of these problems, an asphalt mix was suggested at lower temperature than conventional (HMA) mixtures that was called half-warm asphalt. The production temperature of HWMA is in the range of 66- 100°C, while for (HMA) it ranges between 138 - 160 °C as shown in Fig. 2 [18]. The main advantages of producing HWMA are reducing temperatures of production and placement, producing fewer fumes, less emissions at the plant, less energy consumption and less aging of the binder [18]. HWMA especially containing recycled materials is considered a sustainable technique of producing asphalt mixes at low temperature. A mix of aggregate and bitumen is heated at a range of temperature between 70°C to 100°C , while WMA is produced and mixed at temperatures between 120 and 140°C [19].



(a) : Sasobit (b) : Aspha-min

Fig. 1. (a): Sasobit, (b): Aspha-min [17]

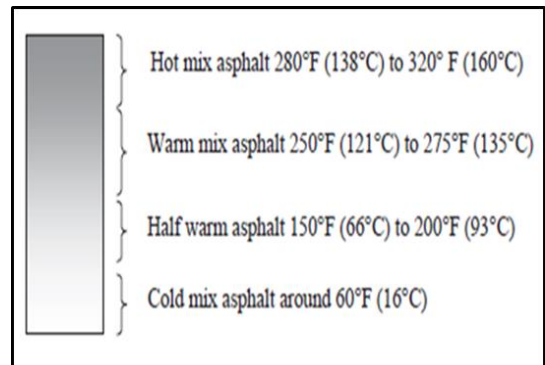


Fig. 2. Classification by Temperature of Asphalt Mixtures [18]

2. Research Objectives

The objectives of this study are summarized as follows:-

- Investigating the engineering properties of HWMA developed using Ethoxylated Nonyl Phenol (NP9).
- Produce environmental friendly asphalt mixes by reducing gases emissions.
- Make a cost analysis of producing HWMA using NP9.

3. Materials

Pavement materials consist of asphalt binder, additives and aggregates.

3.1 Asphalt Binder

Asphalt cement used in this study was supplied by El-Nasr Company –Suez City. It has a penetration grade of 60/70. Table 1 lists the physical characteristics of asphalt binder.

3.2 Aggregate

The coarse aggregates used are crushed dolomite stones. The aggregates gradation is presented in Table 2, while Table 3 represents the aggregates physical properties.

Table 1. Physical Characteristics of Asphalt Binder

Property	AASHTO Designation No.	Result	Specification Limits
Softening Point, °C	T 53	48	45-55
Penetration at 25°C.	T 49	66	60-70
Flash Point, °C	T 48	270	≥ 250
Kinematic Viscosity at 135°C , Centistoke	T 201	426	≥ 320

Table 2. Aggregate Gradation

Sieve Size (Inch)	% Passing (Mix)	Specification Limits (4C -Mix) [20]
1.50	100	100
1.00	100	100
3/4	97	80-100
1/2	86	-----
3/8	74	60-80
No.4	48	48-65
No.8	40	35-50
No.16	27	-----
No.30	20	19-30
No.50	15	13-23
No.100	11	7-15
No.200	7.93	3-8

Table 3. Aggregate Physical Properties

Property	AASHTO O Spec. No.	Agg. (1)	Agg. (2)	Fine Agg. (Sand)	Filler	Spec. Limits (4C-Mix)
Los Angeles abrasion %	AASHTO 96-(2006)	26.0	26.0	----	----	40 Max.
Bulk (S.G)	AASHTO (85-77)	2.55	2.57	2.490	2.75	----
Saturated and dry surface (S.G)	ASHTO (85-77)	2.63	2.62	2.532	----	----
Apparent (S.G)	AASHTO (85-77)	2.72	2.70	2.574	----	----
% Water absorption	AASHTO (85-77)	2.40	1.80	1.30	----	5 Max.

3.3 Ethoxylated Nonyl Phenol (NP9)

Nonylphenol (NP) and nonylphenol ethylates (NPEs) are produced in large volumes, with uses that lead to widespread release to the aquatic environment. NP is a clear to pale yellow viscous liquid at room temperature with moderate water solubility and moderate vapour pressure [21-24]. NP has moderate volatility. Researchers have shown that, in some locations there may be water-to-air volatilization that results in significant atmospheric concentrations of NP substances [25]. In the atmosphere NP is degraded rapidly by hydroxyl radicals and is not expected to be persistent in air [25]. Fig.3 shows the component of NP9, while its properties are presented in Table 4.

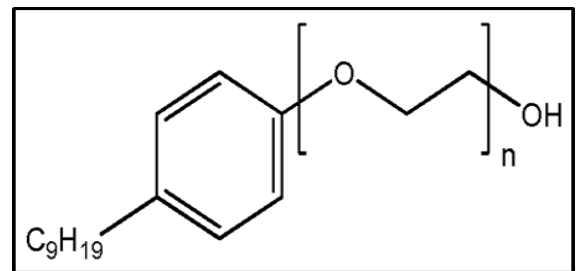


Fig. 3. Chemical Component of NP9 [25]

Table 4. Chemical and Physical Properties of NP9

Chemical Formula	C15H24O
Molar Mass	220.35 g/mol
Appearance	Light yellow viscous liquid with phenolic smell
Density	0.953 g/cm ³
Melting Point	-8 to 2°C
Boiling Point	293 to 297°C
Solubility in Water	6 mg/L (pH 7)

4. Experimental Procedure and Testing

4.1 Marshall Test

Marshall test was carried out in accordance with ASTM D6927–15 [26]. The test aims to determine Marshall parameters as stability, flow, air voids (AV %), voids in mineral aggregate (VMA %), mixture unit weight and Marshall Quotient (MQ). MQ is calculated using Eq. (1).

$$\text{Marshall Quotient (kg/mm)} = \frac{\text{Marshall Stability (kg)}}{\text{Marshall Flow (mm)}} \quad (1)$$

Marshall test parameters were evaluated for both control and modified mixtures. At least three specimens were prepared for each bitumen percentage and compacted by 75 blows (heavy traffic) on each face of the specimen. The specimens then were left for 24 hours and they were submerged in a water bath of 60°C for about 30-45 minutes. The final stage was testing the specimens in Marshall testing machine to determine the stability and flow.

4.2 Rolling Thin Film Oven (RTFO) Test

Rolling Thin Film Oven (RTFO) measures the effect of heat and air on a moving film simulating short-term aging of asphalt binders that occurs during production and paving operations. The test was performed in accordance with AASHTO T 240 [27]. In this test, the mass change of the sample as a percentage of the original mass and retained penetration percentage was determined. Mass change % is calculated using Eq. (2).

$$\text{Mass Change \%} = \frac{(A-B)}{A} * 100 \quad (2)$$

Where: $A = W_{bi} - W_o$ & $B = W_{ba} - W_o$

- W_o = Weight of empty bottle.

- W_{bi} = Weight of binder before aging + Weight of empty bottle.

- W_{ba} = Weight of binder after aging + Weight of empty bottle.

The retained penetration is calculated using Eq. (3).

$$\text{Retained Penetration (\%)} = \frac{\text{Average Penetration after RTFO}}{\text{Average Penetration before RTFO}} * 100 \quad (3)$$

4.3 Moisture Susceptibility Test

Asphalt mixture could be sensitive to moisture. If the stability values after submerging in water changes significantly compared to the original values of dry specimens. This variation in stability is expressed as loss of stability. It can be determined in accordance with ASTM D1559-89 [28]. Marshall specimens were prepared using the same procedures as in Marshall test and the specimens were submerged in a water bath for 24 hours at 60°C, the samples were then tested using the same procedures of Marshall stability test. Loss of Stability (LOS) is calculated by Eq. (4).

$$\text{LOS \%} = \frac{(1 - (\text{Stability of Wet Specimens}) / (\text{Stability of Dry Specimens})) * 100}{1} \quad (4)$$

4.4 Indirect Tensile Strength (ITS) Test

This test was carried out for both control and modified specimens in accordance with ASTM D6931 [29]. ITS test is related to the pavement cracking. ITS test involves loading a cylindrical specimen with compressive loads, which act parallel to and along the vertical diametrical plane. ITS was performed using Marshall Apparatus. The failure load for each specimen was recorded and the ITS value is calculated using Eq. (5).

$$\text{ITS (KPa)} = \frac{2F}{\pi HD} \quad (5)$$

Where F is the failure Load (KN), H is specimen height (m) and D is specimen diameter (m). In addition, tensile strength ratio (TSR) was investigated in accordance with ASTM D4867 [30]. The purpose of conducting this test is to determine the effect of saturation and accelerated water conditioning on the indirect tensile strength specimens. The specimens

were prepared into two categories conditioned and dry. All specimens were tested at a constant temperature of 25°C and the ITS values were measured for dry and wet specimens. TSR is calculated using Eq. (6).

$$TSR\%=(S_{tm}/S_{td}) * 100 \tag{6}$$

Where TSR is tensile strength ratio (%), S_{tm} is wet strength or average tensile strength of the moisture conditioned specimens, (kPa) and S_{td} is dry strength or average tensile strength of the dry specimens, (kPa).

4.5 Wheel Tracking Test

This test provides information about the rate of permanent deformation from a moving concentrated load in accordance with AASHTO T324 [31]. A laboratory compactor designed to prepare slab specimens with dimensions 30.50*30.50*5.0 cm. In this test, the rut depth and number of passes or time to the final rut depth are recorded.

4.6 Emissions Measurement Test

The purpose of this test is to measure the emissions during the mixing and compaction process. The emissions were divided into two parts, mainly inorganic gases and organic solvents. In this study, the tests were conducted in the national center for safety studies, occupational health, and environmental insurance work, according to the ministerial decree no. 211 for the year 2005 [32]. Both inorganic gases and organic solvent were measured.

4.7 Scanning Electron Microscope (SEM) Test

SEM uses a focused beam of high-energy electrons to generate different signals at the surface of solid specimens. The signals reveal the information on texture, chemical composition, crystalline structure and orientation of materials making up the sample. The areas ranging from 1 cm to 5 microns in width can be scanned using conventional SEM techniques with magnification ranging from 20X to approximately 30,000X and spatial resolution of 50 to 100 nm. The electrons interact with atoms in the sample, producing various signals that contain

information about the surface topography and composition of the sample. The electron beam scanned in a raster scan pattern and the position of the beam combined with the detected signal to produce an image.

5. Results and Discussions

5.1 Effect of Adding NP9 on the Binder Properties

The properties of asphalt binder modified by NP9 are presented in Table 5. It was found that penetration increased with the increase in NP9 percentages. Also, the kinematic viscosity at 90°C and 135°C decreased with the increase in NP9 percentages. Viscosity value at 10% NP9 decreased to 536 centistoke which almost near the value of virgin bitumen at 135°C (see Table 1) and increasing NP9 percentage logically will enhanced the workability. Therefore, all percentages of NP9 will tried at mixing temperature of 90°C and evaluate the asphalt mixtures using performance tests.

Table 5. Properties of Binder Modified by NP9

NP9 %	Property Penetration at 25°C, (0.1mm)	Kinematic Viscosity, Centistoke	
		135°C	90°C
0.0%	66	426	--
0.5 %	62	317	1479
0.75 %	70	313	1472
1.0 %	79	309	1463
1.5 %	95	302	1455
1.75 %	103	296	1447
2.0 %	110	288	1442
3.0 %	137	285	1428
5.0 %	196	270	1341
10.0 %	222	105	536

5.2 Marshall Test Results

The gradation used in this research was dense graded for surface asphalt course (DG-4C). The results of

Marshall test were determined at five selected percentage of asphalt cement (AC), 4.5, 5.0, 5.5, 6.0 and 6.5% by weight of aggregates. At each percent, three Marshall specimens were prepared and tested. The optimum asphalt content (OAC) as an average value of asphalt content corresponding to maximum stability, maximum unit weight and 4% air voids were determined. The results of Marshall test for control mix at 155°C as mixing temperature (HMA) at the OAC are presented in Table 6.

Table 6. Marshall Test Results for Control Mix at the OAC , (155° C ,mixing temperature)

Marshall Properties	Value	Spec. Limit (Heavy Traffic)
OAC, %	5.90	4-7.50
Stability, Kg.	1215	Min. 900
Unit Weight, gm/cm ³	2.35	---
Flow, mm	3.20	2-4
AV, %	3.60	3-5
VMA, %	16.30	Min. 15
MQ, kg/mm	380	200-500 [33]

Referring to the results presented in Table 6, it was investigated that, all Marshall parameters meet the specification limits for mix type DG-4C. Table 7 shows the effect of adding NP9 on the Marshall parameters.

The results of Marshall characteristics were determined at five selected percentage of asphalt cement (4.5, 5.0, 5.5, 6.0, and 6.5%). The OAC was calculated following same procedure as the control mixes. Also, it should be mentioned that; Marshall tests were carried out at 90 °C (mixing temperature) as NP9 was used to enhance the workability of HWMA at lower temperatures. The results indicated that, the maximum stability was recorded at 25% NP9. The density increased with an increase in NP9 percentages up to 20.0% and then decreased. The maximum density was determined at 15% NP9. That may be due to increase the NP9 percentage increase the flow of the binder, which increase the coating of aggregate and lead to increase the mixture unit weight by reducing the internal voids. Marshall flow increased with an increase in NP9 percentages up to 20.0%, and then decreased. In addition, the maximum flow was determined at 15%. The AV% and VMA%

were decreased by an increase in NP9 percentages of HWMA. The maximum AV% was recorded at 25% of NP9. In addition, it was investigated that, the asphalt cement content has a signification effect on changing AV% values at various percentages of NP9. While, the maximum VMA% was achieved at 25% of NP9. The Marshall quotient (MQ) decreased with an increase in NP9 percentages up to 20.0%, and then increased, due to a decrease in Marshall stability and flow. The maximum value of MQ was determined at 25% of NP9.

OAC was calculated at each NP9 percentage. It was found that, the OAC in HWMA mixes were changed as presented in Table 7. Also, it was found that, the OAC values increased with an increase in NP9 percentages up to 20.0%, and then decreased. From the results presented, it can be concluded that adding NP9 to asphalt mixes enhanced the engineering properties. Thus, in this research 10%NP9 was selected to perform the performance tests to minimize the cost of asphalt mixes.

Table 7. Marshall Test Results for Control (HMA) and Mixes Modified by NP9 at OAC, (HWMA)

Marshall Parameter	HMA Results	%NP9 , (HWMA)			
		10	15	20	25
OAC, %	5.90	5.75	5.80	6.0	5.60
Stability, Kg.	1215	1400	1250	1212	1450
Unit Weight, gm/cm ³	2.35	2.38	2.39	2.40	2.38
Flow, mm	3.20	3.30	3.50	3.70	3.30
AV, %	3.60	3.20	3.00	3.00	3.00
VMA, %	16.30	15.7	15.4	15.0	15.2
MQ, kg/mm	380	424	357	327	439

5.3 Rolling Thin Film Oven Test Results

Fig. 4 shows the results of mass change percentages for control and modified specimens (10% NP9) of the used binder at relative temperature. The results

indicated that, the mass change for modified bitumen using 10% NP9 at 90°C and 163°C was lower than the conventional specimen at 163°C by 39.94 and 19.82% respectively. In contrast, the retained penetration of modified bitumen using 10% NP9 was higher than unmodified bitumen by 40.26 % as shown in Fig.5. Finally, the modified bitumen using 10% NP9 at HWMA reduced initial aging compared to the conventional bitumen at HMA.

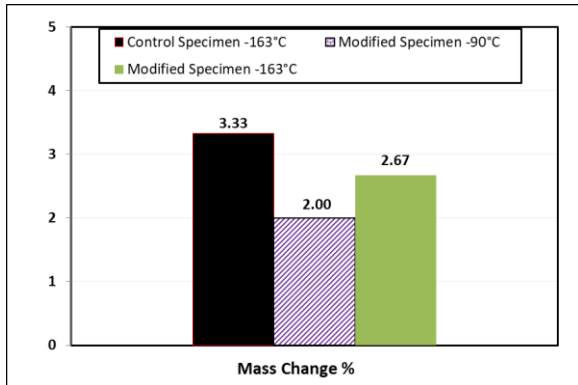


Fig. 4. Mass Change for Control and Modified Specimens by 10%NP9

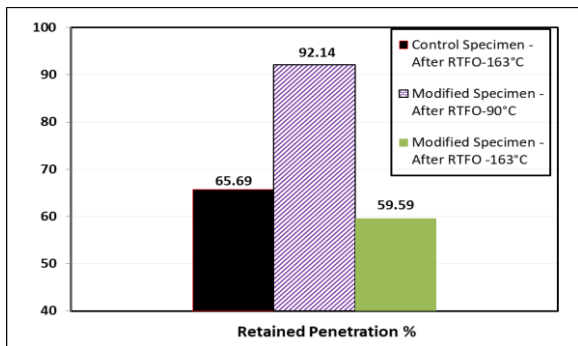


Fig. 5. Retained Penetration % for Control and Modified Specimens by 10%NP9

5.4 Moisture Susceptibility Test Results

Fig. 6 shows the results of loss of stability for control and modified mixes. The test was conducted and the results were recorded for three days. It was found that the resistance to moisture enhanced by producing HWMA compared to conventional mix (HMA). This may be due to the improvement of mixture stiffness, which leads to an increase in the resistance against cracks and failure. All results met the specification limit (20% as a maximum).

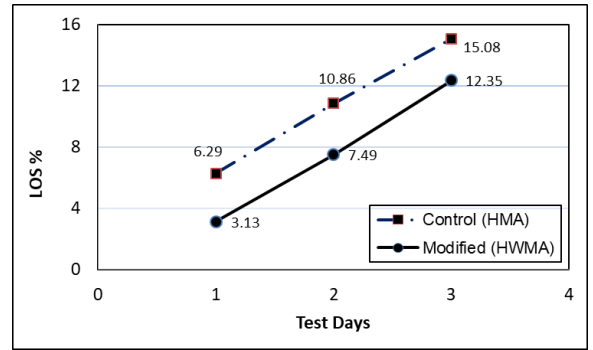


Fig. 6. LOS % for Control and Modified Mixes by 10%NP9

5.5 ITS Test Results

The ITS test was conducted for conventional (HMA) and modified specimens (HWMA), and the results are shown in Fig. 7. Producing HWMA at 90°C using 10% NP9 enhanced ITS. The enhancement of ITS value was observed at 10% NP9 at dry conditions by 5.34%. Also, in submerged conditions, specimens with 10%NP9 (HWMA) show better results than conventional mix (HMA). Fig. 8 shows Tensile Strength Ratio (TSR) for control and modified specimens. It was observed that, the TSR enhanced from 91% to 96 % by producing HWMA prepared using 10% NP9 at 90°C. All results met the specification limit for TSR which is 80% as a minimum.

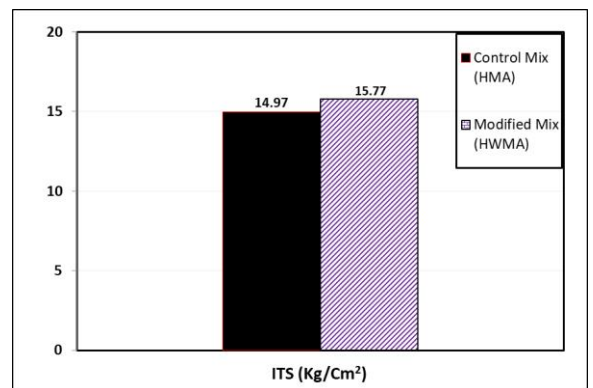


Fig. 7. ITS for Control and Modified Mixes

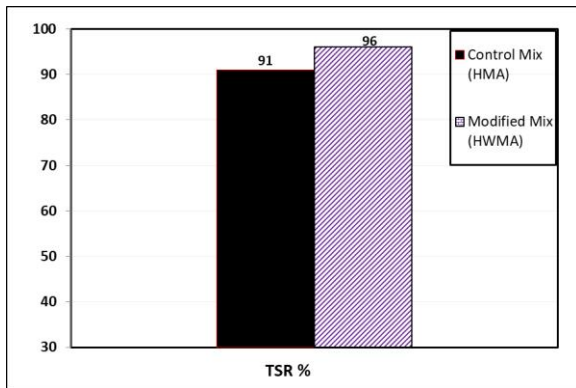


Fig. 8. TSR% for Control and Modified Mixes

5.6 Wheel Tracking Test (WTT) Results

Fig. 9 shows the relationship between rut depth in mm and loading time in minutes for control and modified mixes. The results of WTT showed that the rut depth of HWMA was lesser than the values of control mix (HMA) during the test time.

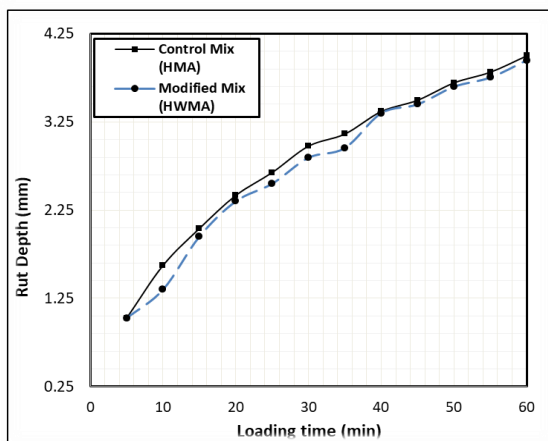


Fig. 9. Rut Depth vs. Loading Time

5.7 Emissions Measurement Test Results

Fig. 10 shows the effect of using the additives on conservation of the environment. In addition, measuring carbon dioxide (CO₂), nitrogen oxides (NO_x) and volatile organic compounds (VOCs) for the tested mixes at 155 and 90°C was conducted to analyze the side effects of using these additives in producing HWMA. It was observed that, gases emissions were decreased by manufacturing HWMA compared to HMA. Producing HWMA reduced gases emissions for CO₂, NO_x and VOCs by 28.66, 62.65 and 21.33% respectively.

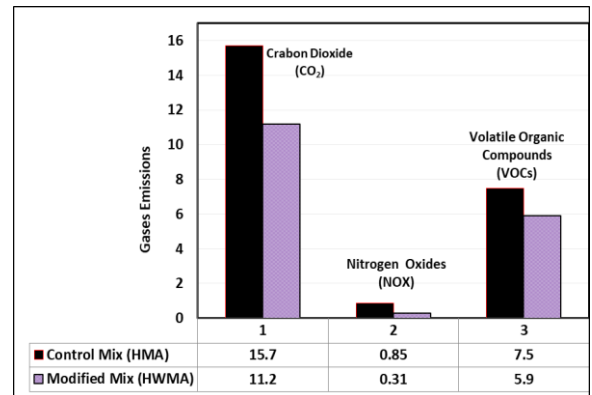


Fig. 10. Gases Emissions for Control and Modified Mixes

5.8 Scanning Electron Microscope (SEM) Test Results

Analytical tests were performed and conducted on specific selected specimens. These specimens were control specimen and specimen modified by 10% NP9. Figures 11 and 12 show SEM images for control and modified specimens. The images showed significant changes by adding NP9.

From SEM test results, a significant change was observed for all tested specimens. The results indicated that, control specimen seems smooth, featureless and shapeless and no microstructure is visible as shown in Fig.11. It can be concluded that the procedure disrupted the original structure. It was observed that, the microstructure of the treated specimen differs than control specimen; it may be due to the change in the structure of asphaltenes presented in bitumen structure. Several previous studies were conducted on bitumen specimens using SEM techniques and the same results have been recorded [34-36].

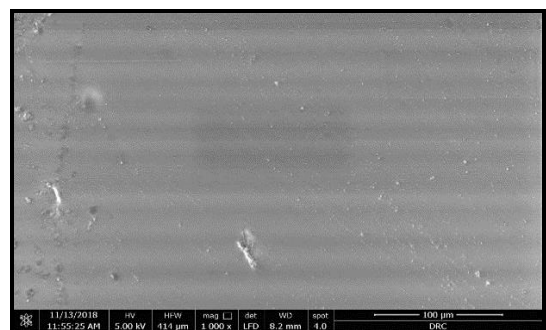


Fig. 11. SEM Image for Control Specimen

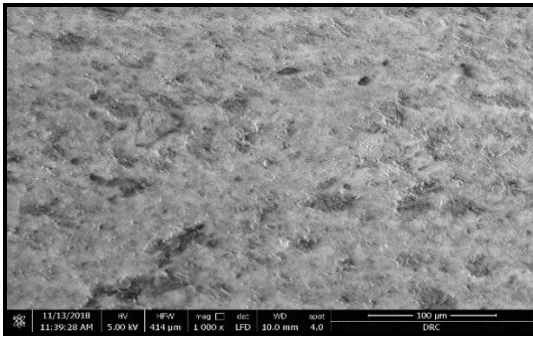


Fig. 12. SEM Image for Specimen Modified by 10%NP9

6. Cost Evaluation

The cost to prepare 1 m³ of HMA and HWMA as a function of the cost of each type of mixture and number of liters of solar are shown in Fig. 13. Generally, it was found that, the cost increased with an increase in the number of liters of solar, which is considered the result of asphalt mixes temperatures. The results showed that, the total cost of preparing 1 m³ of control mixture at 155°C was lesser than those of using NP9. The increase in cost when preparing the mixture using NP9 was small comparing to the reduction in solar consumption which estimated by 47% compared to HMA. This reduction leads to minimize the negative impact of producing HMA.

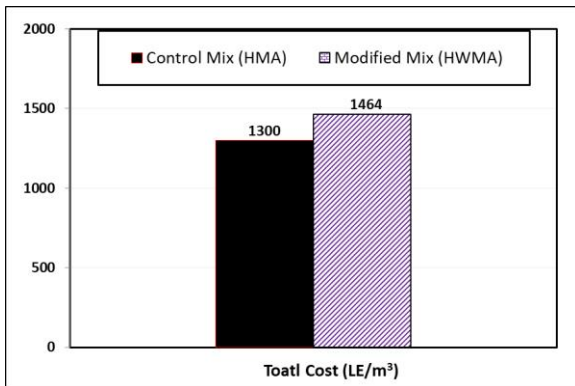


Fig. 13. Total Cost (LE / m³) of Asphalt Mixtures

7. Conclusions

The conclusions of this study summarized in the following points:

- The engineering properties of Marshall test enhanced using NP9 to produce HWMA at 90°C.

- According to performed tests results and cost analysis, 10% NP9 can be selected as the optimum additive content.
- Adding NP9 to bitumen changed its physical properties.
- ITS and TSR enhanced by producing HWMA at 90°C using NP9.
- Producing HWMA using NP9 resists rutting compared to HMA.
- Gases emissions reduced by manufacturing HWMA using NP9.
- Adding NP9 to asphalt binder changed the microstructure of the modified binder as observed in SEM analysis.
- Manufacturing HWMA increased in cost by 12.62% compared to HMA but reduce the negative effect of gases emissions.

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