Journal of University of Babylon for Engineering Sciences, Vol. (26), No. (6): 2018.

Production of Hard Grade Bitumen for Using in High Modulus Asphalt Concrete

Basim H. Al-Humeidawi

Roads and Transport Department, College of Engineering, University of Al-Qadisiyah, Al-Diwaniyah province, Iraq

Basim.alhumeidawi@qu.edu.iq

Mutaz Kadhim Medhlom

Civil Engineering Department, College of Engineering, University of Al-Mustansiriya/Baghdad, Iraq

mkm_mutaz@yahoo.com

Kassim kadhim Hameed

Chemical Engineering Department College of Engineering, University of Al-Qadissiyah

Kassim.alshemary@qu.edu.iq

Huda A. Kadhim

Highway and Transport Department, College of Engineering, Mustansiriyah University, Baghdad, Iraq

hu.eng89@gmail.com

Abstract

The conventional Hot Mix Asphalt (HMA) may suffer from several distress such as fatigue cracks and rutting. These distresses increase with severe climate conditions and reputation of traffic load. The High Modulus Asphalt Concrete (HMAC) designed according to the French method (EME) can be considered as one of the important solutions for these distresses. The production of HMAC requires hard grade bitumen. The current research involved a novel way to produce hard grade bitumen (asphalt binder) to be consonant with the requirements of hard grade bitumen used for a HMAC. The experimental work involved mixing polymer and cross-linking agent with conventional bitumen to get the new bitumen. Since the most concern with hard grade bitumen and HMAC is the fatigue cracks, Crumb Rubber (CR) was added to the obtained bitumen to improve the fatigue performance of the pavement. Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electronic Microscope (SEM) tests were carried out on the conventional and hard grade bitumen. The optimal value of additives was selected to meet the requirement of hard grade bitumen was 4% Novolac from weight of bitumen and 10 % of Hexamine from weight of Novolac (i.e. 0.4% of weight of bitumen), while the selected CR ratio to improve flexibility was 0.5% from weight of bitumen. FTIR and SEM test results showed that a mechanical interaction was occurring between the bitumen and the additives leads to greatly improve the mechanical properties of resulting bitumen. The obtained bitumen is satisfied to required standards and can be used for HMAC according to French method.

Key words: Hard Grade Bitumen, High Modulus Asphalt Concrete (HMAC), Polymer, Novolac, Hexamine, Crumb Rubber, Enrobés À Module Élevé! (EME).

1. Introduction

The flexible pavement is one of worldwide used pavement type. Its structure usually composes of asphalt concrete layer(s), granular base and subbase courses resting on a well-prepared subgrade. Asphalt concrete and bituminous base have the major effect on structural capacity of the pavement since they carry the most portion of the applied load (traffic and environmental loads). Although the state of granular bases and subgrade courses may have a significant impact on permanent deformation (rutting) and other majors distresses in flexible pavement, many studies demonstrated that the design of asphalt mixture and material adequacy (quality and gradation of aggregate, and binder properties) have a significant effect on these distresses. [1]

As the traffic intensity and axle load increase rapidly during the last decades, the highway agencies around the world facing big challenges to provide heavy duty asphalt cement to carry that growing demand without sever deformation. One of the new techniques has been proposed in France is producing high modulus hot mix asphalt can sustain these demands. Enrobés à module élevé (EME) has been introduced during the last three decades in France as new mechanism for designing a HMAC mainly used in base and binder courses of bituminous pavements. This technology was transferred recently to several countries such as UK, Australia, and some of European Union countries [2][3]. The EME can be considered a performance based method taken into the account the moisture susceptibility and fatigue performance of the pavement. It also puts high restriction on permanent deformation of flexible pavement (Rutting).

EME method producer requires hard grade bitumen; this type of bitumen either obtained as refinery produced or may be achieved using some additives. Many additives have been used to modify the original characteristics of bitumen. The most commons types were polymers and several studies have been carried out in this aspect [4]. Each type of polymer has special properties and gives specific effect on bitumen performance.

The using of High Modulus Asphalt Binder (HMAB) significantly improves the mechanical characteristics of Hot Mix Asphalt (HMA). These properties include stiffness, elastic recovery and resistance to cracking. These enhancements consequently cause reduction in pavement thickness by about 20% [5]. However, the use of this type of bitumen at a temperature lower than -10 ^{0}C should be with caution due to the cracking probability [5][6]. Some additives may be used with HMAC to enhance cracking resistance especially in very cold regions [7][8]. These additives can improve the cracking resistance and keeping a high stiffness modulus for asphalt mixture. Several studies have been carried out to produce Polymer Modified Bitumen (PMA) to be used in manufacturing of high modulus asphalt concrete. One of these was the using of Fiber/polymeric compound asphalt to produce high modulus asphalt concrete [9]. The outcome HMA showed a significant resistance to permanent deformation during hot climate in summer time in addition to overall improvement of mechanical properties of HMA.

Another study was conducted to obtain high modulus asphalt concrete using HMAB [10]. During that study HMAB was tested by using scanning electron microscope to evaluate the characteristics of this type of binder. The results of the study showed that, the permanent deformation (rutting depth) and deflection of layers made with mixtures contain HMAB is much smaller than that produced with conventional or SBS modified bitumen.

The main objective of this research is to produce a heavy grade asphalt binder used for production of HMAC. The research plan involved using newly economic sustainable materials to produce this HMAB (hard grade bitumen). These material were phenol formaldehyde solid resin (Novolac) and cross-linking agent Hexamine (Hexamethylene tetramine) to meet the requirement of EME class 2. To complete this task several percentage and deferent ways of mixing were adopted to select the optimum percentage of Novolac and Hexamine. The results of previous study of author showed that using of Novolac and Hexamine can increase the viscosity and cohesion of bitumen and consequently improve the properties of obtained asphalt mixture [11].

Since the obtained asphalt binder has low penetration grade, a Crumb Rubber (CR) was added to obtained binder to maintain the flexibility properties of a new binder [7][12][13].

As it's well know that the HMAB include three types: hard grade asphalt binder (refinery produced), asphalt modified with harder addition (natural asphaltite) and polyolefin asphalt modification [10][14]. The obtained binder can be considered as hard grade bitumen meets the requirement of binder used in EME design method for high modulus asphalt concrete produced in similar way of obtaining polyolefin asphalt modified [5].

2.1.Methodology of the research

2.2. Materials

2.1.1. Conventional asphalt binder

In this research, conventional bitumen binder with a penetration grade of 40-50 was brought from al Nasiriya refinery, south of Iraq. The bitumen was tested according to the specification requirement of EME2. The properties of conventional binder (penetration, softening point, ductility, viscosity and thin film oven tests) were tested through some of conventional tests described in international standards mentioned correspondingly. Table 1, illustrates the tested properties of conventional bitumen. All tests were conducted according to BS EN (British Standards - European Standards) Standards according to requirements of hard grade bitumen and EME2 method except the ductility of bitumen was tested according to ASTM since it is not specified as a requirement for hard grade bitumen and EME2 method, and due to unavailability of test equipment of force ductility according to BS EN 13589.

Table 1. The properties of conventional binder

No.	Test Type	Results	Requirement (according to Iraqi specification)	Reference standard
1	Penetration (0.1 mm)	43	40-50	EN 1426 [15]
2	Softening point (0C)	51		EN 1427 [16]
3	Flash point	290	≥ 232	EN 2592 [17]
4	Ductility (cm)	121	>100	ASTM D 113-99 [18]
After Rolling Thin Film Oven Test				
5	%Retained penetration	58	≥ 55	EN 12607–1+ EN 1426
6	Softening point (ºC)	54		EN 12607–1+ EN 1427
7	%Mass loss 163 °C, 50gm, 5 hr	0.36	≤ 0.5	EN 12607–1 [19]

2.1.2. Modifier

The Novolac polymer (Phenol formaldehyde solid resin) and cross-linking agent Hexamethylene tetramine (H.M.T.A) (See Figure 1, for Novolac and Figure 2 for Hexamine) were used in this research as a new technique to produce hard paving grade asphalt binder. The properties and composition of Novolac have been illustrated in Table 2. Hexamine is a hardener added to cross-linking the Novolac. The hard grade bitumen binder is usually used to produce a high modulus asphalt concrete according to French method (EME). Since, the most concern of hard paving grade asphalt is the fatigue cracks, a low percentages of Crumb Rubber (CR) were tried to enhance the fatigue resistance of asphalt mixture. Particles of crumb rubber (CR) (see figure 3.) used in this research were reclaimed from old scrap tyres. They contain rubber hydrocarbons such as natural rubber, butyl rubber, and styrene–butadiene rubber (NR, BR, and SBR). it also provides a chance to recycling this waste material and reduce the environmental pollution during disposal them [4].

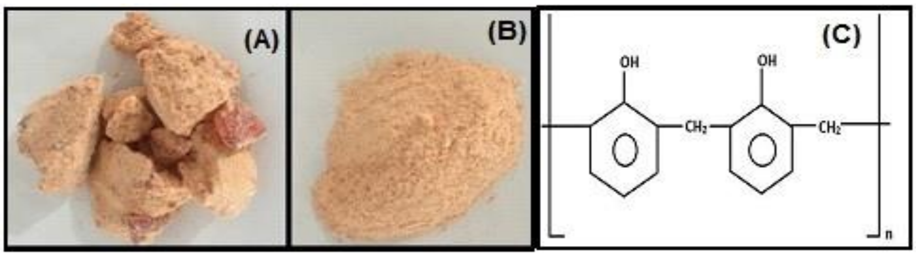


Figure 1.Novolac (Phenol formaldehyde solid resin) :(A) Novolac particles, (B) Grinded Novolac, (C) Structure of Novolac [20].

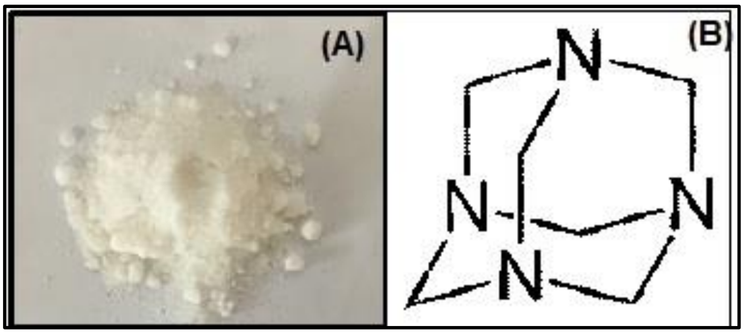


Figure 2. Hexamine (H.M.T A) :(A) Hexamine powder, (C) Structure of Hexamine [20].

Figure 3. Crumb Rubber (CR)

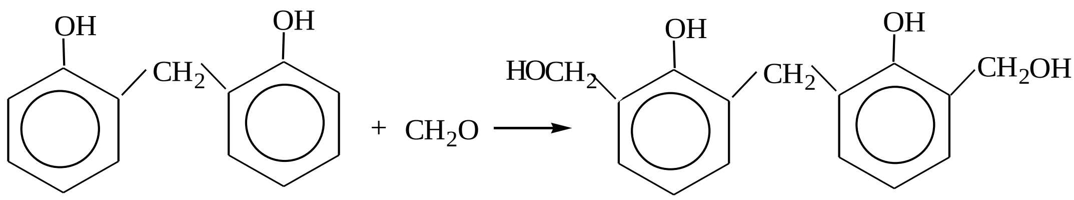
Table 2. Characteristics and composition of Novlac resin [20].

Properties	Value
Phenol-Formaldehyde (wt%)	100
Density (gm/cm^3)	1.25
Melting point ºC	95
Thermal conductivity (W/m º C)	0.2
Viscosity (poise)	13-18
Properties medium	Under acidic conditions (HCL)

The addition of Hexamine to Novolac produces cured Novolac (Novolac resol) which has more reactivity to interact with polar components in asphalt and consequently increase the physical interaction between asphalt molecular. The following equation shows a simple explanation to that interaction [21].

$$(CH2)6\ N4 + 6H2O \longrightarrow 4NH3 + 6CH2O$$

(H.M.T.A) + (moisture and heat) (Ammonia) + (formaldehyde)



(Novolac) (Formaldehyde) (Cured Novolac)

2.3. Description of production process

To describe the mixing operation of conventional asphalt with additives to obtain hard grade bitumen, the following steps were adopted in sequence:

1. Novolac was grind by using the electric mill to be used as dry powder additive with conventional bitumen binder.
2. The bitumen binder (40-50) grade was heated to a temperature of 150 ºC in an oven, then the appropriate weight was added to pre-heated automatic shear mixer. Novolac was added gradually to bitumen binder to avoid agglomeration of polymer and mixed automatically at temperature of160 ºC with cross-linking agent Hexamine (H.M.T.A). The duration of mixing was 1 hour to achieve good blending and dispersion of the polymer into the bitumen binder. The Novolac was added as a percentage from asphalt binder while the Hexamine was added as percentage from Novolac to act as a hardener cross-linking for the Novolac. Different percentages of Novolac (1%,2%,3%,4%,5%) and Hexamine (5%,10%,15%) were used. Conventional tests of bitumen binder (penetration, softening point, ductility, flash point) have been conducted for each percent of (Novolac and H.M.T.A) to find the optimal ratio of additives to obtain hard grade bitumen. The produced bitumen met the requirement of hard grade bitumen used in EME2 method, see Table 3, to produce a HMAC.
3. The CR was added to the produced hard grade bitumen (with optimal ratios of Novolac and H.M.T.A) and mixed by shear mixer. Three ratios (0.5%, 0.75%, 1%) for CR from bitumen weight were tried.
4. The adopted ratio of CR was based on keeping approximately same penetration value and improving ductility value by using the minimum amount of CR.

Table 3. Specifications for hard paving grade bitumen – Properties applying to all hard paving grade bitumen [22]

Properties		Test methods	Units	Classes		
				2	3	4
Penetration at 25 ºC		EN 1426	0,1 mm	15-25	10-20	5-15
Softening point		EN 1427	ºC	55-71	58-78	60-76
Resistance to hardening	Change of mass	EN 12607-1	%	≤ 0,5		
	Retained penetration		%	≥ 55		
	Increase in softening point		ºC	≤ 8		
Flash point		EN ISO 2592	º C	≥ 235		
Solubility		EN 12592	%mass	≥ 99,0		

2.4. Experimental tests

2.3.1. Preliminary test for the additives

Infrared spectrometry (IR) is a practical method adopted the infrared to analysis the samples by identify the infrared absorption of samples and consequently provide chemical information about the molecular structures (molecular bonds) of chemical compound. Spectral data is gathered in wide range wave lengths [23]. Fourier transform infrared spectroscopy (FTIR) considered as a specific type of infrared spectrometer device was used for testing the main two additives used in current research as shown in Figure 4. The functional groups of Novolac were identified using Fourier transform infrared spectrometer device as shown in Figure 5. The test was conducted in the Department of Chemical Engineering, University of Al-Qadisiyah.

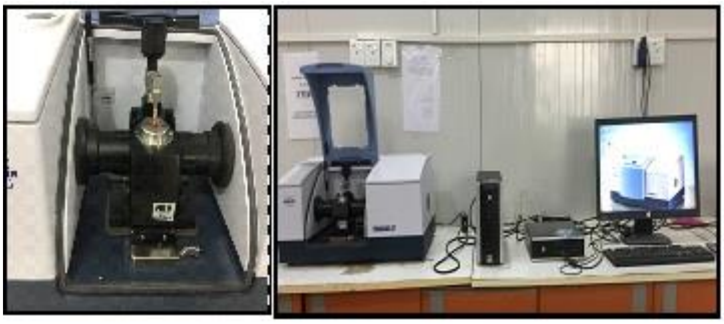


Figure 4. FTIR testing equipment

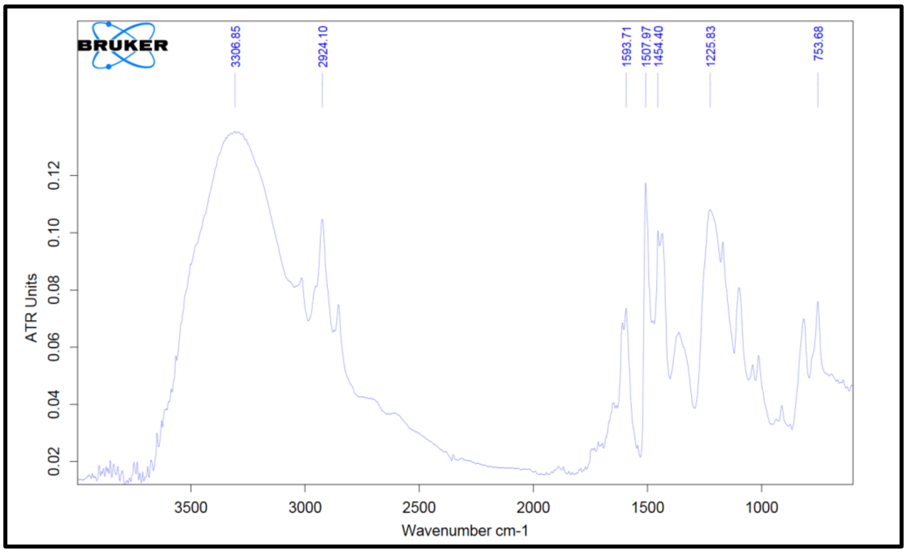


Figure 5. FTIR test results of Novolac

The spectral data of the sample showed the following elements were included with Novolac: Phenols (O-H bonds), Aldehyde (C-H bonds), Alcohols (C-O bonds), Aromatic (C=C bonds) as shown in Table 4. This justification based on wave number of elements appear in the test results comparing with correlations chart used to interpret FTIR results.[24].

Table 4. Summary of the FTIR result of Novolac

Types of vibration	Frequency (cm^{-1})	Elements from test	Their frequency
phenols (O-H)	3200-3400	Phenols (O-H bonds)	3306.85
Aldehyde (C-H)	2800-2900	Aldehyde (C-H bonds)	2924.1
Alcohols (C-O)	1000-1300	Alcohols (C-O bonds)	1225.83
Aromatic (C=C)	1475-1600	Aromatic (C=O bonds)	1507.97

FTIR test was also carried out for Hexamine, and the spectral data are illustrated in Figure 6. The results showed that the following elements were included with Hexamine: absorption band of CH2 at (1465 cm^{-1}), (C-H) aliphatic at (3000cm^{-1}) (N-H) in the Amine group at (3500 cm^{-1}), and (C-N) at (1350Cm^{-1}). The interpretation also based on the previous mentioned chart used to interpret FTIR result [24].

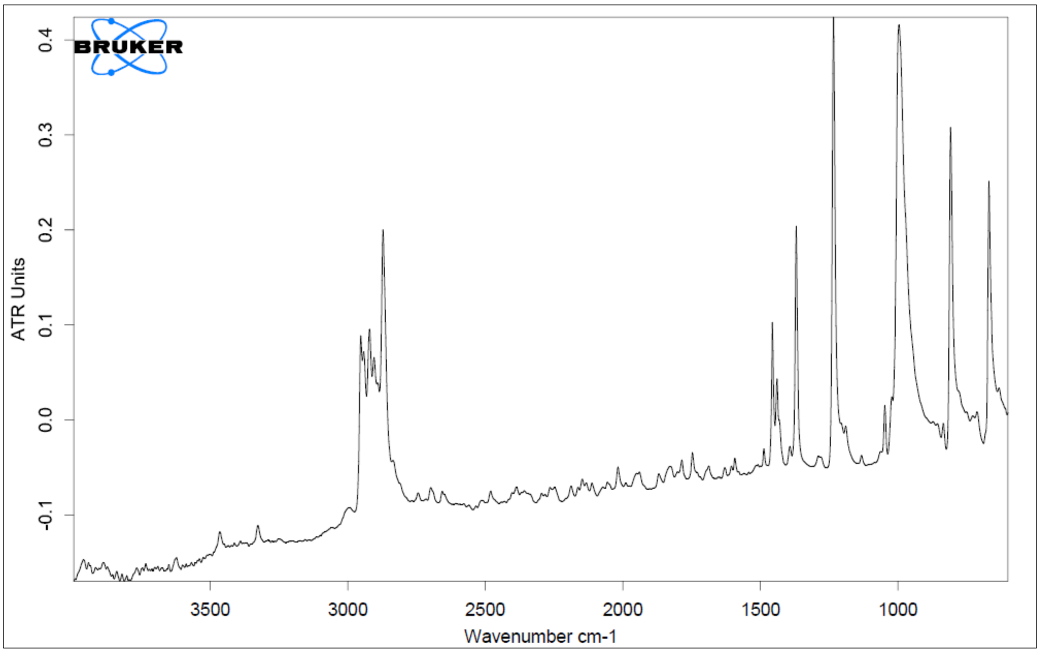


Figure 6. FTIR test results of Hexamine

2.3.2. Laboratory tests of bitumen binder

The standard tests of bitumen were made for both conventional and produced hard grade bitumen. These tests include penetration, softening point, ductility, flash point, solubility, rotational viscosity and rolling thin film oven (RTFO). The tests were also repeated for hard grade bitumen after adding the CR.

2.3.3. FTIR and SEM tests for obtaining bitumen

In order to understand how the obtained binder can blend or react with another component of asphalt mixture, FTIR (mentioned previously in section 2.3.1) and SEM (Scanning Electronic Microscope) tests were carried out on the obtained binder. SEM /EDS (Energy disperse x- ray spectroscopy) analysis is one of three modes used in SEM test. It was used in this research to obtain compositional information of a material by applying beams of electrons on it to get information about their composition (element concentrations and compositions) [25]. Small and thin samples of (pure and hard grade bitumen binder) at solid state were tested by SEM equipment. This test have been conducted in a Central service laboratory, College of Education for Pure Sciences, University of Baghdad. Although these tests (FTIR and SEM) are not standard tests of bitumen, but they were conducted to give an indication about the type of interaction between the bitumen and additives and to show the homogeneity of obtained bitumen. [10][23][26].

3- Results and discussion

3.1. Laboratory tests results of hard grade bitumen

The selection of the optimal value of additives was based on the physical properties of asphalt required to produce hard grade bitumen. Table 5 shows the results of the whole laboratory tests for different percentage of the main additives. The following Figures (7, 8, and 9) show the penetration, softening point and ductility tests results with the minimum and maximum limits as recommended by the specifications of hard paving grade bitumen. Although the ductility of hard grade bitumen was not specified as a requirement, there are some studies showed that the minimum acceptable value for ductility at 25 ^{0}C for hard grade bitumen is 10 cm [6]. In addition, it may give some indication about the flexibility and cohesion properties of bitumen. Even the obtained hard grade binder within the requirements of binder used for EME, the forthcoming tests for HMAC (rutting, fatigue, and moisture sensitivity) should confirm that. These tests are the part of the future work of authors.

Table 5. Selection of optimum ratio of additives

Test	Natural asphalt (40-50) grade	% of Novolac from weight of asphalt binder														
		1%			2%			3%			4%			5%		
		% of Hexamine (H.T.M.A) from weight of asphalt Novolac														
		5%	10%	15%	5%	10%	15%	5%	10%	15%	5%	10%	15%	5%	10%	15%
Penetration (1/10mm)	43	43	30	28	39	27	22	29	20	14	23	17	12	13	11	8
Softening point (°C)	51	50	51	53	51	52	54	53	55	55	56	61	62	61	65	67
Ductility (cm)	121	113	105	78	91	85	59	65	44	37	38	18	11	17	8	6
Flash point (°C)	290	> 300														
		After Thin Film Oven Test														
% Retained penetration, of original	58	71	65	67	69	59	57	77	60	58	82	63	61	82	64	61
Increase in softening point (°C)	3	3	3	2	4	4	2	3	2	2	1	2	1	3	1	3
Ductility of Residue (cm)	72	73	57	71	61	45	36	46	34	26	27	20	15	23	18	14
%Mass loss 163 °C, 50gm, 5 hr	0.36	0.11	0.08	0.10	0.14	0.12	0.09	0.11	0.10	0.12	0.12	0.09	0.04	0.06	0.09	0.11

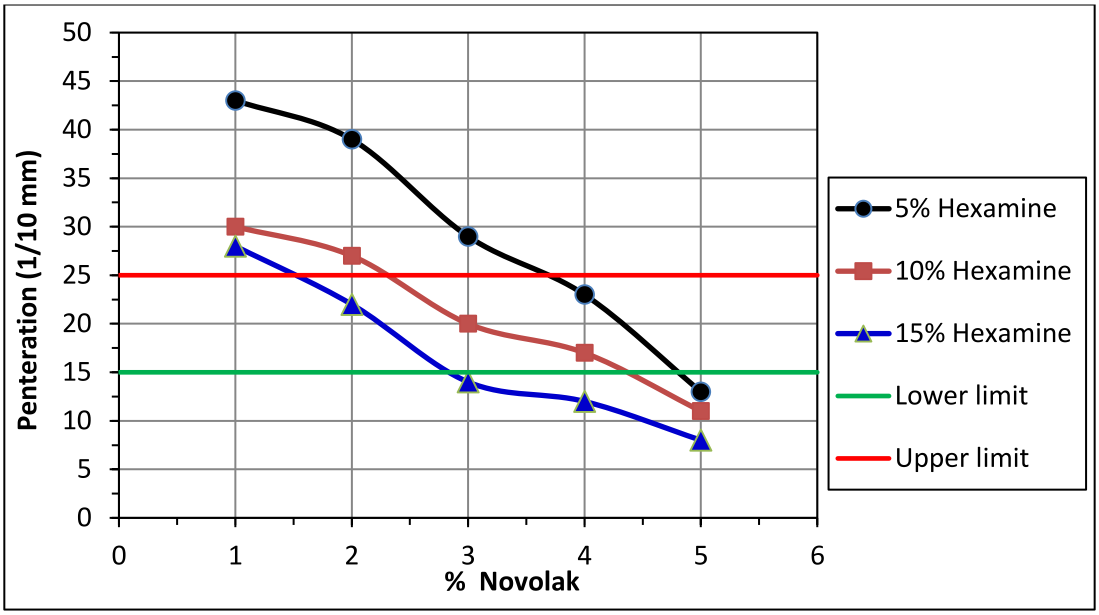


Figure 7. Penetration of bitumen has different ratios of Novolac and Hexamine [22].

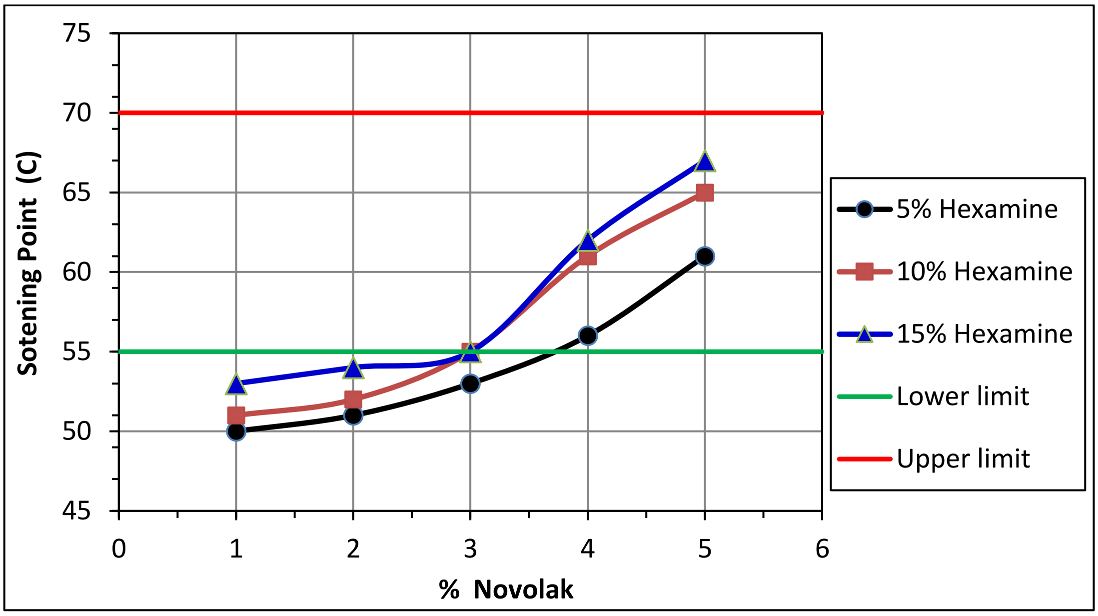


Figure 8. Softening point of bitumen has different ratios of Novolac and Hexamine [22].

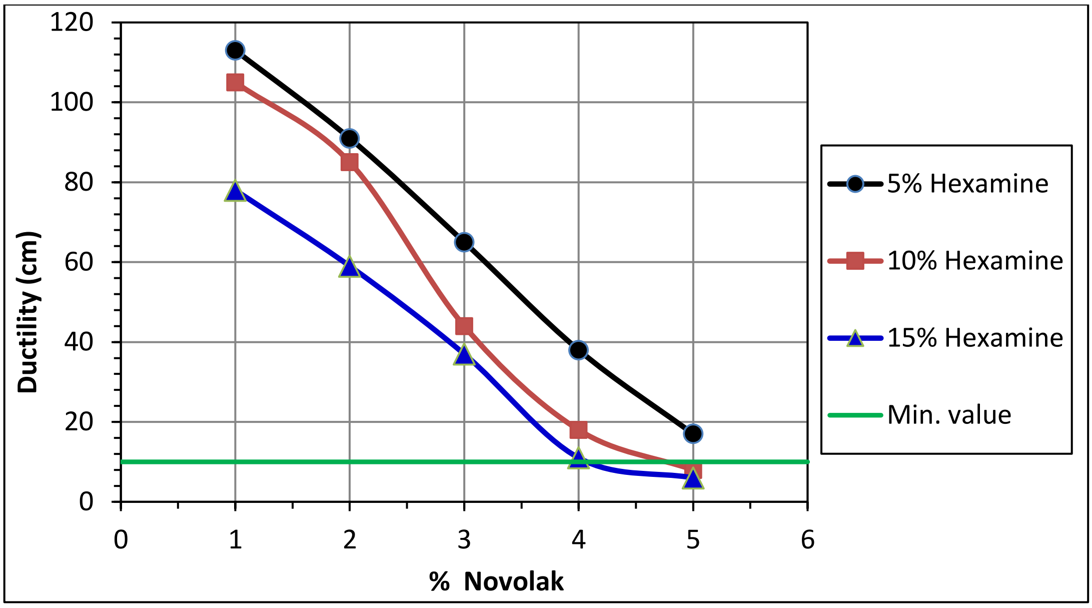


Figure 9. Ductility of bitumen has different ratios of Novolac and Hexamine [6].

The optimal value of additives was selected to meet the requirement of hard grade bitumen used in an EME2 method to produce a HMAC (see Table 5) was 4% Novolac of weight of bitumen and 10 % of Hexamine from weight of Novolac (i.e. 0.4% of weight of bitumen). Table 6 also shows all tests results for selected ratios of additives. The dynamic (rotational) viscosity was adopted to measure the viscosity of bitumen because of using the capillary tube procedure may cause the tube to be clogged due to using CR in the next step [27].

Table 6. Specifications for hard paving grade bitumen [22] and the obtained physical properties of bitumen (without CR)

Property	Test method	Test result	Requirement (according to EME2)
Penetration,100gm,25 °C,5sec (1/10 mm)	EN 1426 [15]	17	(15-25)
Softening point (ºC)	EN 1427 [16]	61	(55-71)
Flash point, (ºC)	EN 2592 [17]	> 300	≥ 235
Solubility	EN 12592 [28]	99	≥ 99
Dynamic (Rotational) viscosity at 135 ºC (Pa sec.)	EN 13302 [29]	0.85	0.6
Kinematic viscosity at 135 ºC (mm^2/sec.)	EN 12595 [30]	817	≥ 600
Ductility,25 ºC, 5 cm/min, (cm)	ASTM D 113-99 [18]	18	
After Thin film oven test BS EN 12607-1 (163°C, 50gm, 5 hr.)			
Softening point (ºC)	EN 12607–1+ EN 1427	63	≥ orig. min. + 2 °C
Retained penetration of original (%)	EN 12607–1+ EN 1426	63	≥ 55
Increase in softening point(°C)	EN 12607–1+ EN 1427	2	≤8
Mass loss (%)	EN 12607–1 [19]	0.09	≤ 0.5

CR was added to optimal ratio of Novolac and Hexamine to improve the fatigue performance of asphalt mixture as reported in literature. The adopted ratio of CR was 0.5 % from bitumen weight as shown in Table 7. The selection was based on keeping approximately same penetration value and improving ductility value by using the minimum amount of CR. The details of test results are shown in Table 8.

Table 7. Selection of CR ratio to be added to hard grade bitumen

Property	0.5 % CR	0.75 % CR	1 % CR
Penetration,100gm,25°C,5sec (1/10 mm)	18	20	24
Softening point (ºC)	61	61	62
Ductility,25 ºC, 5 cm/min, (cm)	24	31	36

Table 8. Specifications for hard paving grade bitumen [22] and the obtained physical properties of bitumen after addition of CR

Property	Test method	Test result	Requirement
Penetration,100gm,25°C,5sec (1/10 mm)	EN 1426	18	(15-25)
Softening point (ºC)	EN 1427	61	(55-71)
Flash point, (ºC)	EN 2592	> 300	≥ 235
Solubility	EN 12592	98.6	≥ 97 [31]
Dynamic (Rotational) viscosity at 135 ºC (Pa sec.)	EN 13302	0.87	0.6
Kinematic viscosity at 135 ºC (mm^2/sec.)	EN 12595	844	≥ 600
Ductility,25 ºC, 5 cm/min, (cm)	ASTM D 113-99	24	
After Thin film oven test BS EN 12607-1 (163°C, 50gm, 5 hr.)			
Softening point (ºC)	EN 12607–1+ EN 1427	65	≥ orig. min. + 2 °C
Retained penetration of original (%)	EN 12607–1+ EN 1426	63	≥ 55
Increase in softening point(°C)	EN 12607–1+ EN 1427	4	≤8
Mass loss (%)	EN 12607–1	0.10	≤ 0.5

3.2. FTIR test result

FTIR test has been conducted on two sample of asphalt (pure and hard grade asphalt) as shown in Figure 10. The results of the test revealed there are no new function groups (appear as peaks) in FTIR results for hard grade asphalt compared with pure asphalt. This can be indicated there are no new chemical components and consequently, there is no major chemical reaction occurred due to production of hard grade bitumen (asphalt) by adding the mentioned polymer and other additives. However, what was happening can be explained by physical blending or interaction of polymer with asphalt in presence of heat and mechanical mixing. During the blending process, Hexamine cured the Novolac resin to make cross-linking polymer. This polymer tends to surround the bitumen molecules to protect them from the additional aging process during the pavement life. This behaviour can be confirmed by increasing the area under curve for hard grade bitumen (see Figure 10); also by the results of thin film oven test presented in Table 6,8, in which; it can been seen higher % retained penetration and less loss of weight for hard grade bitumen compared with pure asphalt. Also, the addition significantly increases the viscosity of bitumen which in turn, increases the cohesion between bitumen molecular and the adhesion between bitumen and aggregate in asphalt mixture. This action significantly improves the mechanical properties of bitumen and leads to obtain hard grade bitumen used to produce high modulus asphalt concrete.

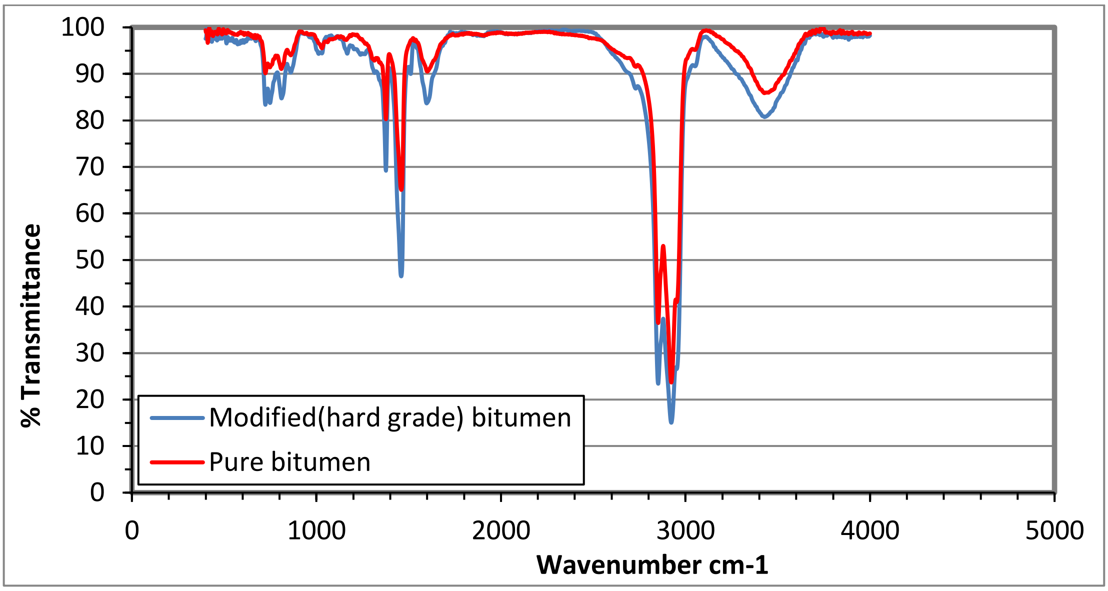


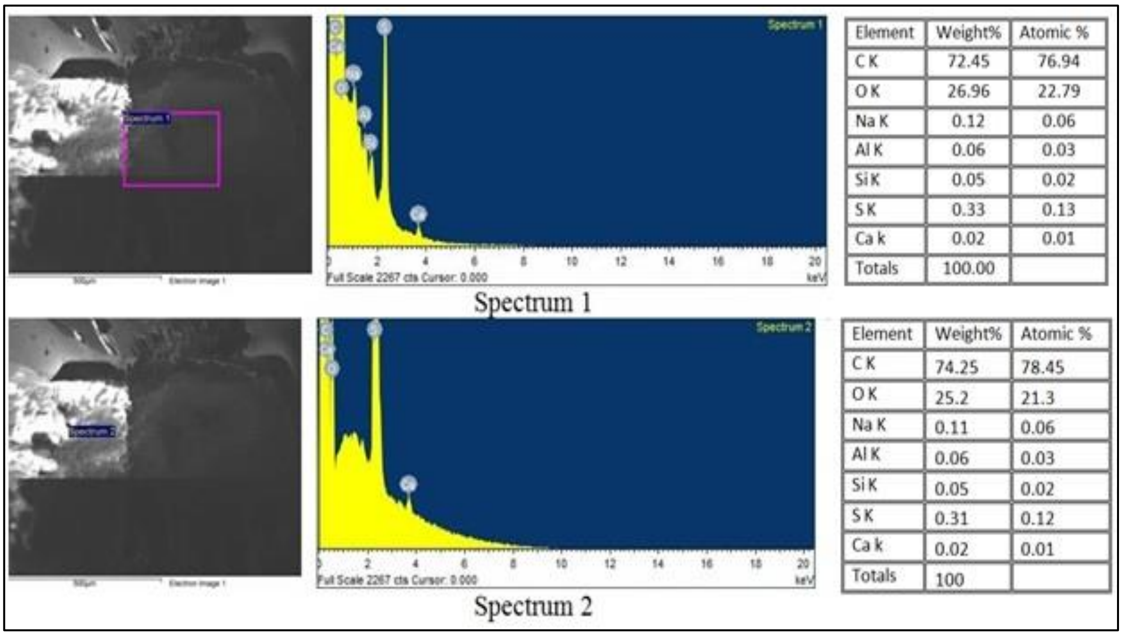
Figure 10. FTIR test results for pure and hard grade bitumen

3.3. SEM/EDS test result

The test was carried out on unmodified and modified (hard grade) bitumen. Two spectrums were tested for each sample of unmodified and modified bitumen. The outputs of the test were presented as element analysis shows the percent of elements in each spectrum. Minor differences can be seen between the percentages of elements in the two spectrums for each sample due to the different places of machine focusing during the test.

Figure 11 and Figure 12 show the tests results for the pure and hard grade bitumen respectively. These figures show no significant changes in weight of elements between pure and hard grade bitumen which confirmed there is not a major chemical reaction. However, there are some changes in weight of both carbon (C) and oxygen (O) elements, because the additives contained both of these elements and they may combine to the original components of bitumen. In addition, the mixing process may bring some amount of oxygen to the bitumen. Some elements (such as manganese element (Mg) and chloride element (Cl)) also appeared in hard grade bitumen due to the addition of crumb rubber. Although the asphalt is mixture of hydrocarbons, the hydrogen does not appear in the results of element analysis due to its low atomic number [25][32].

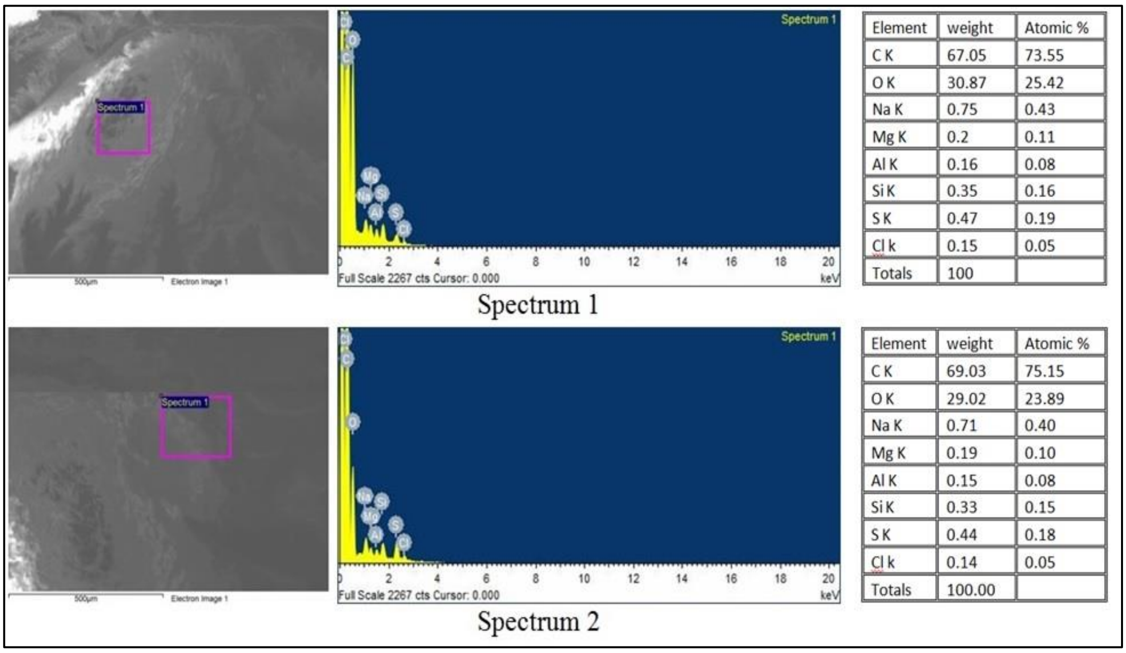
The picture of hard grade bitumen obtained from SEM /EDS test (shown in Figure 12) reveals that the addition made the obtained bitumen more homogenous and consequently improves their cohesion and mechanical properties.



Element	Weight%	Atomic %
C K	72.45	76.94
O K	26.96	22.79
Na K	0.12	0.06
Al K	0.06	0.03
Si K	0.05	0.02
S K	0.33	0.13
Ca k	0.02	0.01
Totals	100.00	

Element	Weight%	Atomic %
C K	74.25	78.45
O K	25.2	21.3
Na K	0.11	0.06
Al K	0.06	0.03
Si K	0.05	0.02
S K	0.31	0.12
Ca k	0.02	0.01
Totals	100	

Figure 11. Elementary analysis for conventional 40-50 (unmodified) bitumen



Element	weight	Atomic %
C K	67.05	73.55
O K	30.87	25.42
Na K	0.75	0.43
Mg K	0.2	0.11
Al K	0.16	0.08
Si K	0.35	0.16
S K	0.47	0.19
Cl k	0.15	0.05
Totals	100	

Element	weight	Atomic %
C K	69.03	75.15
O K	29.02	23.89
Na K	0.71	0.40
Mg K	0.19	0.10
Al K	0.15	0.08
Si K	0.33	0.15
S K	0.44	0.18
Cl k	0.14	0.05
Totals	100.00	

Figure 12. Elementary analysis for hard grade (modified) bitumen

The SEM/EDS test results showed slightly more oxygen in obtained bitumen, which may give more concern about more aging for the obtained asphalt with long-term performance. However, previous studies showed that the rate of aging with long term performance for modified asphalt is quiet less than that of conventional asphalt, which reduce the concern of aging with long term performance [23]. This can also be confirmed by the comparing the results after RTFOT for unmodified and hard grade asphalt (modified), in which the percent of retained penetration is higher for modified asphalt than that of unmodified asphalt and the loss in heat is quite less for modified bitumen as shown in Table .6,8.

2. Summary and conclusions

The current research involved newly method for production of hard grade bitumen used in design of high modulus asphalt mixture according to EME method from conventional bitumen 40-50 grade. The process consisted of addition of polymer and cross-linking agent (Novolac and Hexamine); so, it is similar to production of polyolefin asphalt modified. Since the most concern with hard grade bitumen is the fatigue performance of mixture, CR was added to improve this behaviour.

The main outputs of the study can be listed below:

1- Several percentage of additives were tried to get the optimum ratio of addition. The optimum ratios of additives to produce hard grade bitumen were 4%, 0.4 % and 0.5% of bitumen weight for Novolac, Hexamine and CR respectively.
2- The mechanical properties of obtained bitumen were corresponding to that of hard grade bitumen binder used in design of HMAC according to EME method. This achievement is helping to use the obtained binder (hard grade) in forthcoming work to produce high modulus asphalt concrete.
3- The increasing of oxygen in obtained bitumen may increase the aging process during the early stage of pavement life. The aging of bitumen at an earlier stage is good for pavement, regarding to the performance (decreases the moisture damage, improve rutting resistance). The rate of aging decreases with long-term performance of pavement as confirmed by RTFOT (in which the percent of retained penetration is higher for modified asphalt binder than that of unmodified asphalt and the loss in heat is quite less for modified bitumen) and previous study.
4- Although, the FTIR and SEM tests are not standard tests for binder, they were used to show if there are major chemical changes were occurred during the blending process. These tests showed no major chemical changes were occurred in spite of the great improvement in mechanical properties of binder; also, they clearly showed the homogeneity of obtaining binder.

Acknowledgement

Authors would like to thank the staff and administrations of Roads and Transport Department, and Chemical Department in University of Al-Qadisiyah college of Engineering for their assistance in completing the experimental work.

References

[1] AODAH, H.H., 2013, "*Performance prediction model for bituminous mixes", Ph.D.Thesis, Department of Civil Engineering, Indian institute of technology roorkee*.

[2] Petho, L. and P. Bryant, 2015, "*High modulus asphalt (EME2) pavement design in Queensland"*. In *AAPA International Flexible Pavements Conference, 16th, 2015, Gold Coast, Queensland, Australia*.

[3] Petho, L. and E. Denneman, 2016, "*Implementing EME2, the French high modulus asphalt in Australia", 6th Eurasphalt & Eurobitume Congress*: Prague, Czech Republic.

[4] Rodrigues, C. and R. Hanumanthgari, 2015, "*Polymer modified bitumens and other modified binders", The Shell Bitumen Handbook*, ICE Publishing: Bristol, UK. p. 149-183.

[5] Geng, H., C.S. Clopotel, and H.U. Bahia, 2013, "*Effects of high modulus asphalt binders on performance of typical asphalt pavement structures".* Construction and Building Materials. **44**: p. 207-213.

[6] Espersson, M., 2014, "*Effect in the high modulus asphalt concrete with the temperature".* Construction and Building Materials. **71**: p. 638-643.

[7] Moreno-Navarro, F., *et.al.*, 2014, "*The use of additives for the improvement of the mechanical behavior of high modulus asphalt mixes".* Construction and Building Materials. **70**: p. 65-70.

[8] Moreno-Navarro, F., *et.al.*, 2016, "*High-Modulus Asphalt Mixtures Modified with Acrylic Fibers for Their Use in Pavements under Severe Climate Conditions".* Journal of Cold Regions Engineering. **30**(4): p. 04016003.

[9] Montanelli, E.F., 2013, "*Fiber/polymeric compound for high modulus polymer modified asphalt (PMA)".* Procedia-Social and Behavioral Sciences. **104**: p. 39-48.

[10] Zou, X., *et.al.*, 2015, "*Modification mechanism of high modulus asphalt binders and mixtures performance evaluation".* Construction and Building Materials. **90**: p. 53-58.

[11] Al-Humeidawi, B.H., *et.al.*, 2016,"*Evaluation of Moisture Damage and Stripping of Asphalt Concrete Prepared With New Additives of Polymer Modified Bitumen".* Journal of Babylon University/Engineering Sciences. **24**(1): p. 117-128.

[12] Moreno, F., *et.al.*, 2012,"*The effect of crumb rubber modifier on the resistance of asphalt mixes to plastic deformation".* Materials & Design. **47**: p. 274-280.

[13] Beer, C. and V.s. Davies, 2015, "*Alternatives asphalts*", in *The Shell Bitumen Handbook*. ICE Publishing: Bristol, UK. p. 643-.

[14] Ma, T., *et.al.*, 2015, "*Laboratory performance characteristics of high modulus asphalt mixture with high-content RAP".* Construction and Building Materials. **101**: p. 975-982.

[15] BS EN 1426, 2015, "*Bitumen and bituminous binders — Determination of needle penetration"*, British Standards Institution: London, United Kingdom.

[16] BS EN 1427, 2015, "*Bitumen and bituminous binders — Determination of the softening point — Ring and Ball method".* British Standards Institution: London, United Kingdom.

[17] BS EN ISO 2592, 2001,"*Methods of test for petroleum and its products — BS 2000-36*, in *Determination of flash and fire points — Cleveland open cup method" — (Identical with IP 36-2002)*. British Standards Institution London, United kingdom.

[18] American society for testing and materials, 2009, "*Standard test method for ductility of bituminous materials", Annual Book of ASTM standards,Road and paving materials;vehicle pavement system.(04):03*.

[19] BS EN 12607-1, 2014, "*Bitumen and Bituminous binders - Determination of resistance to hardening under influence of heat and air - part 1:RTFOT method"*. British Standards Institution London, United Kingdom.

[20] Ali Aziz AL- janabi, 2004, *"Thermomechanical investigation of resin modified rubber composites "*, in *M.Sc.Thesis, Depatment of Chemical Engineering, University of technology* .

[21] Harris P.W, S.N., 1994, *Resins from Alkyl phenols for the tyre industry and non-tyre rubber industry*.

[22] BS EN 13924, 2015, "*Bitumen and bituminous binders — Specification framework for special paving grade bitumen, hard paving grade bitumen"*. British Standards Institution: London, United Kingdom.

[23] Yao, H., Q. Dai, and Z. You, 2015, "*Fourier Transform Infrared Spectroscopy characterization of aging-related properties of original and nano-modified asphalt binders"*. Construction and Building Materials. **101**: p. 1078-1087.

[24] Pavia DL, L.G., Kriz GS, Vyvyan JA, 2008. "*Introduction to Spectroscopy": Gengage Learning*.

[25] Kutchko, B.G. and A.G. Kim, 2006, Fly *ash characterization by SEM-EDS.* Fuel. **85**(17): p. 2537-2544.

[26] Arabani, M. and M. Pedram, 2016, "*Laboratory investigation of rutting and fatigue in glassphalt containing waste plastic bottles".* Construction and Building Materials. **116**: p. 378-383.

[27] Asphalt Institute, 1996, *"Superpave mix design ", Superpave Series NO.2(SP-2),The Asphalt Institude,* Lexington ,Kentucky: .

[28] BS EN 12592, 2014, "*Bitumen and Bituminous binders - Determination of solubility"* British Standards Institution London, United Kingdom.

[29] BS EN 13302, 2010, "*Bitumen and bituminous binders — Determination of dynamic viscosity bituminous binder using a rotating spindle apparatus"*. British Standards Institution: London, United Kingdom.

[30] BS EN 12595, 2014, "*Bitumen and Bituminous binders - Determination of kinematic viscosity ".* British Standards Institution London, United Kingdom.

[31] Frank Beer, *et.al.*, 2015, "*Alternative asphalts"*, in *The Shell Bitumen Handbook*. ICE Publishing: Bristol, UK. p. 643-686.

[32] Stojilovic, N., 2012, "*Why Cannot We See Hydrogen in X-ray Photoelectron Spectroscopy?"* Journal of Chemical Education. **89**(10): p. 1331-1332.

انتاج اسفلت صلب لغرض الاستخدام في انتاج خرسانه اسفلتيه ذات معامل مرونه عالي

باسم حسن شناوة

إدارة الطرق والنقل، كلية الهندسة، جامعة القادسية، محافظة الديوانية، العراق

Basim.alhumeidawi@qu.edu.iq

معتز كاظم مضلوم

قسم الهندسة المدنية، كلية الهندسة، الجامعة المستنصرية، بغداد، العراق

mkm_mutaz@yahoo.com

قاسم كاظم حميد

قسم الهندسة الكيميائية كلية الهندسة، جامعة القادسية

Kassim.alshemary@qu.edu.iq

هدى علي كاظم

دائرة الطرق والمواصلات، كلية الهندسة، الجامعة المستنصرية، بغداد، العراق

hu.eng89@gmail.com

الخلاصة

قد تتعرض الخرسانة الاسفلتية الاعتيادية لعدة اضرار مثل شقوق الكلل والتخدد وتتفاقم درجة هذه الاضرار مع الظروف المناخية القاسية والحمل المروري المتكرر. يمكن اعتبار الخلطة الاسفلتية العالية المرونة المصممة وفقا للطريقة الفرنسية (EME)) أحد الحلول المهمة للحد من هذه الاضرار. ان انتاج هذا النوع من الخليط يتطلب اسفلت (كمادة رابطة) عالي الصلابة. البحث الحالي يتضمن طريقة جديدة لأنتاج الاسفلت بتدرج اختراق نوع (hard) ليلائم متطلبات الاسفلت العالي الصلابة المستخدم لأنتاج الخلطة الاسفلتية العالية المرونة المصممة وفقا للطريقة الفرنسية. العمل المختبري يتضمن خلط البوليمر ومصلبه مع الاسفلت الاعتيادي لأنتاج الاسفلت الجديد العالي الصلابة. نظرا لأن شقوق الكلل هي أكثر المخاوف المتعلقة بالأسفلت (كمادة رابطة) العالي الصلابة والخلطة الاسفلتية العالية المرونة، لذلك تمت إضافة حبيبات المطاط الى الاسفلت المنتج العالي الصلابة لتحسين أداء التبليط في مقاومة شقوق الكلل. تم اجراء اختباري مطياف الاشعة تحت الحمراء ومسح المجهر الالكتروني للأسفلت الاعتيادي وللأسفلت العالي الصلابة. كانت القيمة المثلى المختارة للمضافات لانتاج الاسفلت العالي الصلابة المستخدم في الخلطة الاسفلتية العالية المرونة هي 4% و 0.4%من وزن الاسفلت للنوفولاك والهكسامين على التوالي، بينما نسبة حبيبات المطاط لتحسين المرونة هي 0.5%. أظهرت نتائج الاختبارات المجهرية والاشعة تحت الحمراء للاسفلت المنتج حدوث تداخلا ميكانيكيا بين الاسفلت والمضافات أدى الى تحسن كبير في الخواص الميكانيكية للاسفلت المنتج. الاسفلت المنتج قد حقق متطلبات الاسفلت العالي الصلابة وبالتالي يمكن استخدامه في انتاج الخلطة الاسفلتية العالية المرونة المصممة وفق الطريقة الفرنسية.

الكلمات المفتاحية: البيتومين عالي الصلابة، الخرسانة الإسفلتية، البوليمر، مخلفات الاطارات، النوفولاك، طريقة (EME)