

## Comparative Rheological and Mechanical Characteristics of Different Warm-Mix Asphalt Additives Under Aging Conditions

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### ABSTRACT

*This study compares the rheological and mechanical characteristics of three different kinds of warm-mix asphalt additives (WMAA) namely: natural zeolite (NZ), synthetic zeolite (SZ) and manufactured zeolite (MZ). 40/50 Dora penetration grade bitumen and one dosage of each WMAA were chosen. The resultant WMA binders were subjected to penetration, softening point, ductility, elastic recovery, Furol viscosity, elastic modulus, temperature susceptibility, aging, cracking index, compatibility, extensional viscosity, and shear strength tests. Test results depict that the rheological and mechanical characteristics of NZ and MZ binders are better than SZ binder against resistance to high and low temperature effects.*

### Keywords:

*Rheological characteristics; Mechanical characteristics; Warm asphalt additives; Aging; Shear strength.*

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### 1. INTRODUCTION

Warm mix asphalt (WMA) technology was innovated in Europe to solve the environmental problems (minimize gases emissions) and to give several advantages such as: reduce plant emission and fumes, minimize consumption of energy and cost, enhance compaction and better workability, implementation in cold weather, minimized cracking due to thermal effects, minimize mixing and compaction temperatures by 20-30°C than hot-asphalt mixtures, and far hauling distance [1-7].

Warm mix asphalt additives (WMAA) kind plays significant role in manufacturing desired binder and mix characteristics. Thus, chosen of WMAA type is importance and depends on bitumen. Different kinds of WMAA are available worldwide, such as natural zeolite (NZ), synthetic zeolite (SZ), sasobit, LEA, Evothorm, Terex-foaming, Cecabase, Asphamin [8].

In one aspect, no process was explored towards using of WMAA in paving construction or rehabilitation in Iraqi projects. In another aspect, aging is preliminary factor influencing the age of an asphalt pavement. Asphalt short-term aging (STA) happen through the production and construction STA stage. Upon aging, the physicochemical characteristics of the asphalt vary, resulting harder asphalt (i.e. causes cracking failure). Any cracks noticed at the surface of the pavement may exceed the process of aging due to the increased subject to air and result in additional pavement distress, leading to premature paving failures [9].

Few studies have been searched and encouraged on using WMAA in paving application due its environmental and economic benefits. Ivan et.al [10] showed that the mix contained Cecabase exhibited significant improvement against resistance to rutting.

Nishant et.al [11] depicted that using of Evothorm II in asphalt mixtures minimized the mixing and compaction temperatures of about

30°C and enhanced the mechanical traits of mixes under aging conditions.

Arega et al. [12] investigated the effect of WMAA and aging on the rheology of four bitumen treated with high and low natural wax dosages, and five WMAA (Evotherm DAT, Evotherm 3G, Sasobit, Rediset WMX, and Cecabase RT 945). It was concluded that the effect of STA on bitumen stiffness depended on bitumen kind and additives. Besides, WMAA may decrease the viscosity of STA binders, especially those treated with higher natural wax dosage.

Xiao et al. [13] performed research to evaluate the effect of STA on the rheological traits of non-foaming WMAA binders. Four binders with four non-foaming WMAA were adopted. It was noticed that the non-foaming WMAA can decrease the bitumen viscosity and thus minimize the mixing and compaction temperatures for the bitumen mix.

However, complimentary studies are needed to give in details more information about other kinds of WMAA and its benefits in improving the rheological and mechanical characteristics of bitumen under STA effects.

## 2. OBJECTIVES OF THE RESEARCH

The preliminary objectives of this research are to: (1) Compare the rheological and mechanical characteristics of three WMAA (natural zeolite (NZ), synthetic zeolite (SZ) and manufacture zeolite (MZ)) in terms of: penetration, softening point, ductility, elastic recovery, Furol viscosity, elastic modulus, temperature susceptibility, compatibility, cracking index, durability, and shear strength under aging effects; and (2) select the best WMAA among three kinds.

NZ, SZ, and MZ WMAA were chosen for the following reasons: it's convenient for Iraqi climate; and it added to bitumen at lower dosage. For each WMAA kind, one dosage was blended into the 40/50 Dora penetration grade bitumen (D40).

## 3. TESTS PROCEDURES

### 3.1. Bitumen and WMAA

Bitumen utilized in this research was D40 and its physicochemical characteristics were tabulated in Table 1. The results indicated that D40 satisfy ASTM [14] and SCRIB [15] specifications for penetration graded bitumen.

Table 1: Properties of D40

Characteristics	Value	SCRIB [15]	ASTM [14]
Penetration (25°C, 100g, 5s, dmm)	40	40-50	40-50
Softening point, °C	50	-	50-58
Ductility ((25°C, 5 cm/min, cm)	150 <sup>+</sup>	>100	>100
Elastic recovery (%): (25°C) (15°C)	81.82 84.85	- -	- -
Sp. gr (25 °C/ 25 °C)	1.053 2	-	1.01-1.06
Flash point (COC, °C)	277	>232	>240
Loss on heat (5hrs, 163 °C, %)	0.380	-	0.2 max
P.I.	-1.668	-	-2 to +2

Three types of WMAA namely: NZ, SZ, and MZ were selected as chemical additives for D40 at recommended dosages (1.5-3% wt. of bitumen) by supplier. Physicochemical characteristics of the WMAA were tabulated in Table 2 and 3.

Table 2: Physicochemical characteristics of MZ

Characteristics	MZ
C31H64, %	60
C28H58, %	30
Maltene, %	10
Bulk density, g/cc	0.90
Solubility, %	Insoluble in water, benzene, and ether

Table 3: Physicochemical characteristics of NZ and SZ

Characteristics	NZ	SZ
Ingredients	Na <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> . 2SiO <sub>2</sub> .27H <sub>2</sub> O	Na <sub>2</sub> O.Al <sub>2</sub> O <sub>3</sub> . 2SiO <sub>2</sub>
SiO <sub>2</sub> , %	39.46	32.8
Al <sub>2</sub> O <sub>3</sub> , %	28.35	29.1
Na <sub>2</sub> O, %	13.16	16.1
CaO, %	0.26	--
MgO, %	0.26	--
Fe <sub>2</sub> O <sub>3</sub>	0.84	--
K <sub>2</sub> O	0.29	--
L.O.I., %	15.13	21.2
Surface area, m <sup>2</sup> /g	7.7	--
Bulk density, g/cc	0.730	0.568
Water absorption, %	18.5	--

### 3.2. D40/WMAA production

For the production of WMAA binders from D40, one dosage (1.5% wt of D40) for each WMAA was mixed under (135±5°C, 500 rpm, and 3±1 minutes) conditions to obtain a homogeneous binder. Three modified binders (NZ, SZ, and MZ) were thus produced from D40.

### 3.3. Testing rheological and mechanical characteristics of WMA binders

The rheological and mechanical tests conducted on WMAA binders (i.e. NZ, SZ, and MZ) before and after STA as per ASTM [14] include: Penetration (D-5); Softening point (D-36); Ductility (D-113); Elastic recovery (D-3633M); Furol viscosity (D-88); Elastic modulus; Temperature Susceptibility; Compatibility; Cracking characteristics; Durability (STA); and Shear strength (D-5).

## 4. RESULTS AND DISCUSSIONS

### 4.1. Penetration and softening point

Penetration and softening point of WMAA binders before STA were examined and the results are depicted in Figure 1 and 2. The results notify that WMAA are effective in improving D40 characteristics. Penetration at 25°C, and 46.1°C for NZ, SZ and MZ were found to be 37, 41.5, and 46.5, and 203, 209, and 229, respectively. From these results, it can be notified that NZ well done against shear resistance in moderate to high temperatures than other WMAA. As compared with D40, the percent decrease in penetration of NZ at 25°C and 46.1°C was noticed to be 7.5% and 5%, respectively.

The softening point, R&B traditional test was utilized to examine the high-temperature rutting characteristics of WMAA binders. From Figure 2, R&B before STA for NZ, SZ and MZ were noticed to be 52, 48, and 46, respectively. It is shown that NZ exhibited R&B higher than SZ and MZ. It exhibited 4% higher R&B than D40 (i.e. NZ more resistance against rutting than D40, SZ and MZ). This is because that NZ contains water (27H<sub>2</sub>O) in its chemical composition which evaporates during mixing process.

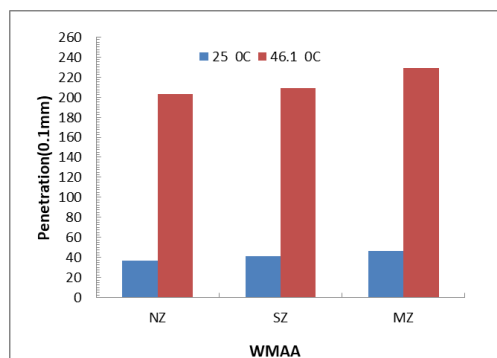


Fig. 1 Penetration of WMAA

### 4.2. Ductility and elastic recovery

The ductility (Du) and elastic recovery (Er) test was performed to examine the elasticity

characteristics of WMAA binders. Figure 3 shows Du at 25°C and 15°C for WMAA binders. Apparently, different WMAA kinds with the same dosage resulted in different Du and Er. Testing Figure 3, it can be noticed that all WMAA having Du values of 100+ at 25°C and 15°C.

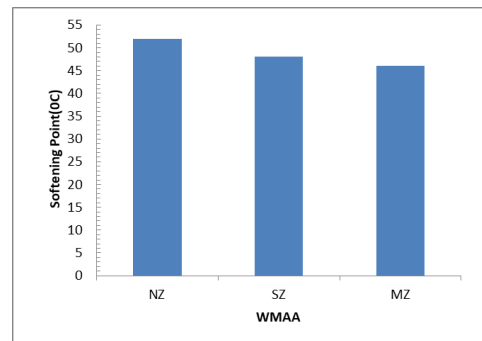


Fig. 2 Softening point of WMAA

Similarly, Er percentage at 25°C and 15°C for NZ, SZ, and MZ were found to be 77%, 79% and 79%, and 83%, 78% and 84%, respectively. Testing Figure 4, it can be notified that no significant in Er values at 25°C. In contrast, NZ and MZ give similar values at 15°C. These results notify that all WMAA have the same elasticity at medium temperatures, whereas, NZ and MZ gives higher elasticity than SZ at 15°C (i.e. NZ and MZ increase the tensile strain of asphalt-mix layer in flexible pavement).

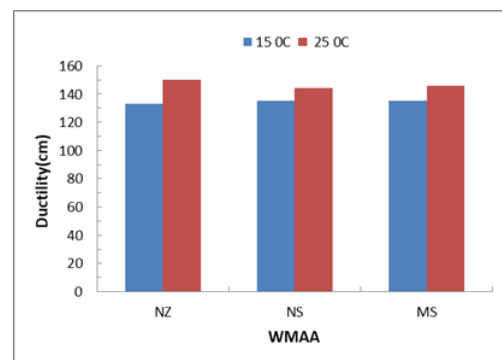


Fig. 3 Ductility of WMAA

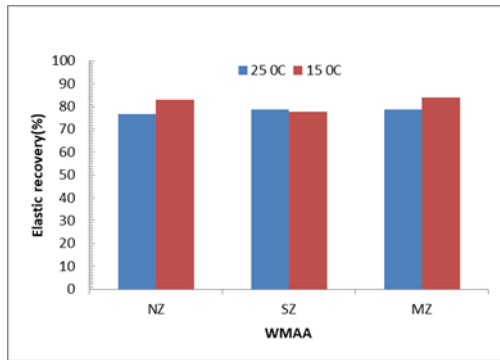


Fig. 4 Elastic recovery of WMAA

**4.3. Furol viscosity**

Furol viscosity (FV) of WMAA binders was examined using Saybolt-Furol viscometer at different temperatures to find out the mixing and compaction temperatures of WMAA mixtures. These temperatures are such that the Saybolt-Furol viscosities are 85 and 140 s, respectively based on the Asphalt institute manual. Figure 5 depicts the FV. It can be noticed that the addition of WMAA decreases the FV of D40 (i.e. decreases the mixing and compaction temperatures of hot-mix asphalt). It was notified that the addition of NZ, SZ and MZ to D40 reduced the mixing temperatures by 26, 22 and 27°C, respectively, whereas, the compaction temperatures were reduced by 22, 20 and 24°C, respectively. These findings complied with those reported by Ali Topal et.al [16], which illustrated that the mixing and compaction temperatures can be minimized between 20-30°C.

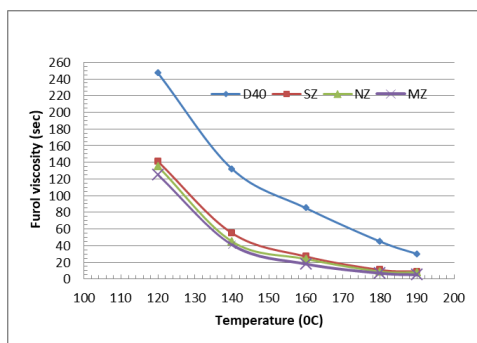


Fig. 5 Furol viscosity of WMAA

**4.4. Elastic modulus**

The elastic modulus (Em) of WMAA binders was calculated based on the binder characteristics utilizing equation 1 as derived from the Van der polnomograph and as mentioned by Al-Hadidy et.al [18].

$$E_m = 1.157 / 10^7 * \lambda^{-0.368} * 2.718^{-P.I.*(R\&B - T_{binder})^5} \quad (1)$$

Where:

$E_m$ = the binder elastic modulus (N/mm<sup>2</sup>),  
 R&B = the aged binder softening degree (°C),  
 $T_{binder}$ = the temperature of the binder layer (°C),  
 P.I. = the aged binder ‘Penetration Index’ and  
 $\lambda$ = the time of loading (sec.).

Equation 1 is only applicable when:

$$0.01\text{sec} < \lambda < 0.1\text{sec},$$

$$-1.0 < P.I. < 1.0,$$

$$20^\circ\text{C} < (R\&B - T_{asp}) < 60^\circ\text{C}.$$

Table 4 shows the  $E_m$  and WMAA at different temperatures. It was found that the  $E_m$  values at 40°C of NZ, SZ, and MZ binder are 0.710, 0.155, and 0.055 Mpa, respectively. It can be seen that NZ binder has higher  $E_m$  at 40°C than SZ and MZ binders. This indicates that NZ binder is more resistance to rutting and fatigue at high temperatures. Besides, MZ found to have higher  $E_m$  at -10°C than NZ and SZ.

Table 4: Elastic modulus of WMAA

WMAA	Em, Mpa			
	-10°C	10°C	25°C	40°C
D40	3020	390	38	0.389
NZ	2620	374	41	0.710
SZ	3870	467	38	0.155
MZ	3900	428	29	0.055

**4.5. Temperature susceptibility**

The penetration index (P.I.) and penetration ratio (P.R.) equations (2 and 3) [17] were adopted to examine the effect of WMAA addition on temperature susceptibility of D40.

$$P.I. = [(20 - 500A) / (1 + 50A)] \quad (2)$$

$$A = [(\log \text{pen.}@T - \log 800) / (T - R\&B)]$$

$$P.R. = (P_2) / (P_1) \quad (3)$$

Where:

T = Testing temperature

R&B= Softening degree.

$P_1$  and  $P_2$  = Penetration at 25°C and 46.1°C, respectively.

Figure 6 and 7 shows between the P.I. /P.R. and WMAA. The P.I. values of NZ, SZ, and MZ binder are -1.36, -2.09, and -2.27, respectively. It can be noticed that NZ binder in the normal range of P.I. (-2.0 to +2.0). This depicts that NZ binder is less susceptible to temperature varies than SZ and MZ. As well as, the P.R. values of NZ, SZ, and MZ binder are 5.486, 5.036 and 4.925, respectively insure P.I. findings.

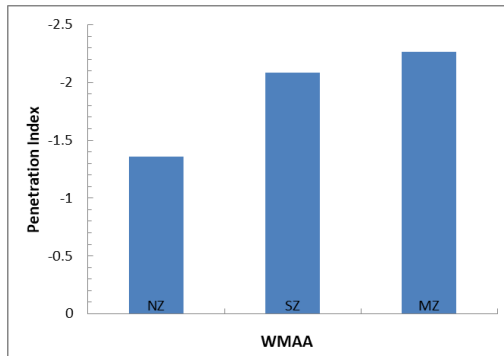


Fig. 6 Penetration index of WMAA

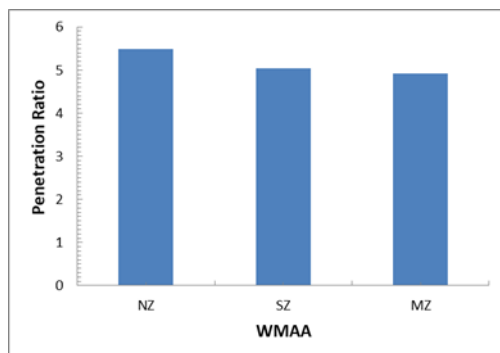


Fig. 7 Penetration ratio of WMAA

#### 4.6. Compatibility tests

The compatibility between WMAA and D40 was examined by passing the binder at the mixing temperature of each binder through a 0.075mm US sieve. It was noticed that the WMAA binders thus prepared can be stored for future use.

#### 4.7. Cracking characteristics at low temperatures

Cracking in flexible pavements caused by thermal influences is effort and non-economic pavements distress in many situations of absolutely cool regions. The reason is primary due to reduction in temperatures, which puts paving materials under tensile stresses (i.e. fracture failure) [17].

Cracking index (CI) described in equation 4 and as reported by Al-Hadidy et.al [17] can be predicted thermal cracking.

$$CI = 10.033 * \text{sq. root}(AI) + 0.334 \log(P) +$$

$$3.3148(SP) - 166.204 \quad (4)$$

Where

AI = aging index,

P = penetration (25 °C),

SP = aged binder softening point (°C).

Figure 8 shows the CI for WMAA binders. From this Figure, it was noticed that the

CI values of NZ, SZ, and MZ binder are 32.83, 26.05, and 30.04, respectively. This notifies that the NZ and MZ perform better towards thermal cracking effects than SZ due to their higher temperature resistance.

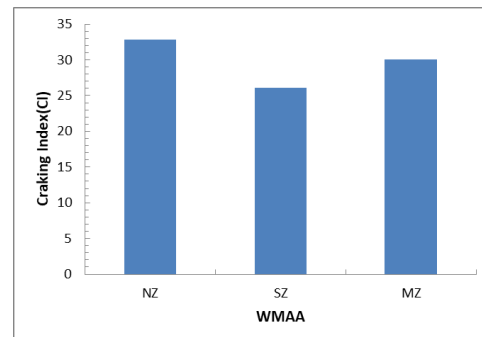


Fig. 8 Cracking index of WMAA

#### 4.8. Extensional viscosity

The extensional viscosity ( $\lambda_b$ ) of WMAA binders in Mpa.s was calculated from equation 5 reported by Al-Hadidy et.al [17, 18].

$$\lambda_b = 3 * 10^{-6} \{ 1.3 * 10^{[3 + (R \& B^{-T_{\text{binder}}})/10]} \} (5)$$

Where:

R&B and  $T_{\text{binder}}$  (as defined above).

Table 5 shows  $\lambda_b$  of WMAA binders. It can be noticed that the  $\lambda_b$  of NZ, SZ and MZ at 25°C 6.935, 7.781 and 6.18 Mpa.s, respectively. Similarly, these values at 60°C were depicted to be  $2.19 \times 10^{-3}$ ,  $2.46 \times 10^{-3}$  and  $1.95 \times 10^{-3}$ . This depicts that increasing in temperature leads to decrease in binder  $\lambda_b$ .

Table 5: Extensional viscosity of WMAA

WMAA	$\lambda_b$ , Mpa.S	
	25°C	60°C
D40	9.796	$3.09 \times 10^{-3}$
NZ	6.935	$2.19 \times 10^{-3}$
SZ	7.781	$2.46 \times 10^{-3}$
MZ	6.18	$1.95 \times 10^{-3}$

#### 4.9. Aging (durability) characteristics

Penetration, Du, SP and Er tests were performed to examine the STA characteristics of the WMAA binders. STA usually occurs in mixing plant and during construction process. A.I was adopted to examine the change in consistency (hardening) of WMAA. A.I was determined from (aged penetration at 25°C / virgin penetration at 25°C). Table 6 depicts the test results, including penetration, SP, Du, Er, A.I. and P.I. values measured on WMAA samples after STA. It can be noticed that SZ binder depicted higher A.I than NZ and MZ, due to higher bonds between SZ



additive and D40, resulting in prevention of the brittleness of the resultant binders (improved STA traits). The Du and Er of aged WMAA binders notifies that SZ and MZ binders depicted higher plasticity than NZ at 15°C (i.e., more resistance to deformation). The Du and Er at 25°C and 15°C varied with the type of WMAA. The STA WMAA samples exhibited slight decrease in Er at both tested temperatures. It was found that that all WMAA binders having aged Du values greater than 100cm for both tested temperatures, except for NZ which has 85cm Du at 15°C.

The aged P.I. value of SZ was noticed to be -0.642 and it's at preferable range (-1.0 < P.I. < -0.5) as reported in KSLA nomograph [19].

Table 6 shows that the percentage loss in heat and air of NZ, SZ and MZ are 0.010, 0.499 and 0.239%, respectively. It is clear that NZ and MZ perform well than SZ against the air and heat effects (i.e. durability of NZ and MZ is better than SZ).

Table 6: STA characteristics of WMAA

WMAA	Pen. dmm	Du, cm		Er, %		SP °C	P.I.	A.I.	Loss of heat and air, %
		25°C	15°C	25°C	15°C				
NZ	23	121	85	88	87	57.5	-1.101	0.622	0.0103
SZ	28	127	118	84	88	58	-0.642	0.674	0.499
MZ	21	130	120	85	88	57	-1.355	0.452	0.239

**4.10. Shear strength**

The cone penetration assay (CPA) was introduced to assess the WMA shear strength and as shown in Figure 9. 1:1 wt. ratio of WMAA binder and CaCO3 as a filler (100 passing 0.075mm) were: (1) blended at the WMAA mixing temperature; (2) WMAA: filler was placed into tin vessel and the later was left in laboratory for 40±5 min and then it was cured in water at 30°C for 60 min; (3) the CPT was applied on the samples with cone weight (w) of 200±5 g until the CPA dial reading (h in dmm) became stable; and (4) the shear stress τ (kPa) of WMAA: filler at the inclined cone surface with angle (α/2 = 15°) was calculated using equation 6:

$$\tau = [981 * w * \cos^2(\alpha/2)] / [3.14 * h^2 * \tan(\alpha/2)] \quad (6)$$

Triplicate samples were tested for each WMAA kind. Figure 10 depicted that the shear stress of WMAA are of NZ, SZ and MZ are 9.86, 10.88 and 8.15 kPa, respectively. It can be noticed

that the shear stress of SZ is better than NZ and MZ.

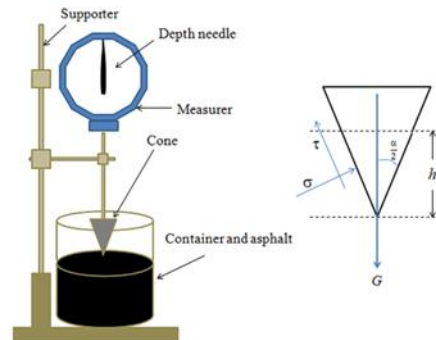
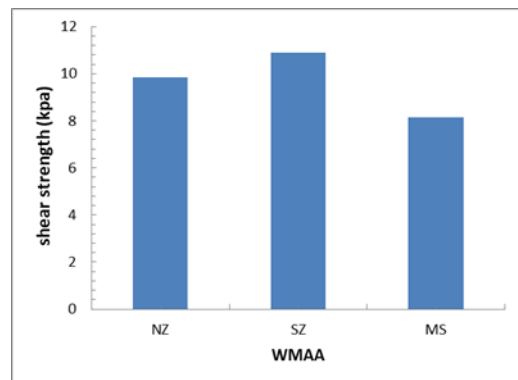


Fig. 9 Shear strength test

Fig. 10 Shear strength of WMAA



**5. CONCLUSION**

Based on the limited tests performed on WMAA, the following conclusions were documented:

1. NZ perform well against shear and rutting resistance, as well as, it is less susceptible to temperature change than SZ and MZ. NZ exhibited 7.5% and 5% lower penetration at 25°C and 46.1°C, respectively, and 4% higher softening points than D40. Besides, NZ is less susceptible to temperature changes than SZ and MZ.
2. The Du and Er results notify that all WMAA have the same elasticity at medium temperatures, whereas, NZ and MZ gives higher elasticity than SZ at 15°C (i.e. NZ and MZ gives more tensile strain for flexible pavement layers).

3. Addition of NZ, SZ and MZ to D40 reduced the mixing temperatures by 26, 22 and 27°C, respectively, whereas, the compaction temperatures were minimized to 22, 20 and 24°C, respectively.
4. NZ binder has higher Er at 40°C than SZ and MZ binders. This indicates that NZ binder is more resistance to rutting and fatigue at high temperatures. Besides, MZ noticed to have higher Er at -10°C than NZ and SZ.
5. Compatibility test insures that the prepared WMA binders can be stored for future use.
6. The  $\lambda_b$ , A.I, and shear stress of SZ at 25°C and 60°C was found to be higher than those for NZ and MZ.
7. The Du and Er of aged WMAA binders indicates that SZ and MZ binders noticed higher plasticity than NZ at 15°C (i.e., more resistance to deformation). The aged WMAA samples exhibited slight decrease in Er at 25°C and 15°C with aged Du values greater than 100cm; and
8. Durability and cracking resistance of NZ and MZ were noticed to be better than SZ (i.e. more resistance to STA effects).

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## مقارنة الخصائص الريولوجية والميكانيكية لأضافة الزيولوت على الأسفلت الدافئ تحت ظروف التعتيق

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جامعة الموصل - كلية الهندسة - قسم الهندسة المدنية

### المخلص

تقارن الدراسة الحالية الخصائص الريولوجية والميكانيكية لثلاث أنواع من الزيوليت كمادة مضافة على الاسفلت الدافئ (الزيولوت الطبيعية، الزيولوت الصناعي، والزيولوت المنتج محليا). استخدم اسفلت الدورة نو النفاذية 40-50 مع نسبة واحدة من المضافات الثلاث. خضعت المواد المنتجة (الاسفلت والمواد المضافة) الى اختبارات النفاذية، نقطة الليونة، الاستطالة، الاستطالة المسترجعة، لزوجة فورل، معامل المرونة، تأثير الحرارة، التعتيق، دليل التشققات، التجانس، اللزوجة، و مقاومة القص. أوضحت النتائج بأن الخصائص الريولوجية والميكانيكية للزيولوت الطبيعي والمنتج محليا أفضل من تلك للزيولوت الصناعي تجاه مقاومة تأثير درجات الحرارة العالية والواطئة.

### الكلمات الداله :

الخصائص الريولوجية، الخصائص الميكانيكية، مضافات لأسفلت الدافئ، التعتيق، مقاومة القص.