

Article

Performance Evaluation of WMA Containing Re-Refined Acidic Sludge and Amorphous Poly Alpha Olefin (APAO)

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Abstract: The use of waste materials has been increasingly conceived as a sustainable alternative to conventional materials in the road construction industry, as concerns have arisen from the uncontrolled exploitation of natural resources in recent years. Re-refined acidic sludge (RAS) obtained from a waste material—acidic sludge—is an alternative source for bitumen. This study's primary purpose is to evaluate the resistance of warm mix asphalt (WMA) mixtures containing RAS and a polymeric additive against moisture damage and rutting. The modified bitumen studied in this research is a mixture of virgin bitumen 60/70, RAS (10, 20, and 30%), and amorphous poly alpha olefin (APAO) polymer. To this end, Marshall test, moisture susceptibility tests (i.e., tensile strength ratio (TSR), residual Marshall, and Texas boiling water), resilient modulus, and rutting assessment tests (i.e., dynamic creep, Marshall quotient, and Kim) were carried out. The results showed superior values for modified mixtures compared to the control mix considering the Marshall test. Moreover, the probability of a reduction in mixes' moisture damage was proved by moisture sensitivity tests. The results showed that modified mixtures could improve asphalt mixtures' permanent deformation resistance and its resilience modulus. Asphalt mixtures containing 20% RAS (substitute for bitumen) showed a better performance in all the experiments among the samples tested.

Keywords: warm mix asphalt (WMA); re-refined acidic sludge (RAS); amorphous poly alpha olefin (APAO); moisture susceptibility; permanent deformation



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1. Introduction

For the past several years, the asphalt industry's technological improvements have been increasingly explored in an attempt to overcome the increasing concerns about global warming and rising energy consumption. Utilizing warm mix asphalt (WMA) and recycled materials in the road construction industry are two measures aiming at achieving this objective [1]. Limited funding for the road infrastructure construction and maintenance has urged engineers and researchers to evaluate the mechanical properties of pavements using recycled materials [2].

WMA is a sustainable technology in the pavement industry that allows the mixing and compaction temperatures to be reduced by 10–38 °C compared to the conventional hot mix asphalt (HMA). In this research, the WMA is manufactured by adding a non-foaming organic WMA additive, Sasobit. The WMA brings about substantial energy savings, resulting in diminished asphalt mix production expenses and noxious gas emissions [3,4]. Employing these techniques along with using recycled waste materials in pavement construction are of basic components of sustainable development principles into the road construction sector due to the high consumption of mineral materials in road projects and construction. Attributed to the above, the engineering properties of various waste materials

in the construction industry have been studied in recent years with the aim of diverting waste materials from landfills. For instance, in recent years, the road construction industry has become a severe environmental threat owing to its heavy metal toxicity, abundance, and persistence in the environment. [5].

An old and dangerous oil refining method incorporates sulfuric acid to extract oil from the residual sludge [6]. Acidic sludge, with an unpleasant odor and devastating effects on the environment, is one of the by-products of the oil refining process, and 15 barrels of acidic sludge is produced per 100 barrels of oil [7,8]. Various commercial products are currently manufactured from acidic sludge such as surfactants, sulfuric acid, light liquid hydrocarbons, ammonium sulfate, activated carbon, furnace and boiler fuels, bitumen, and isogamous thermal insulators [9]. Due to the similarity of acidic sludge properties to the conventional bitumen (color, viscosity, and nonpolar asphaltenes), researchers have made efforts to refine, neutralize, and modify this hazardous material, some of which have been successful [9–11].

Landfill and stabilization of acidic sludge involve the discharge and seepage of heavy metals through the soil and into the surrounding environment; thus, it is a long-term source of soil and water contamination. Diminishing the quantity of acidic sludge produced in oil refineries is an effective strategy that can be adopted for proper waste management [12]. The modified acidic sludge product, which is called re-refined acidic sludge (RAS) in this study, can be employed as a potential substitute for a portion of bitumen used in asphalt mix production. The lower production cost of RAS (compared to virgin bitumen) and the use of recycled materials from environmentally hazardous materials are two decisive factors for using this product as an alternative to bitumen in the road construction industry.

In 2017, a study evaluated the use of acidic sludge powder as an additive in filler and bitumen in terms of strength of asphalt mixtures. Results showed that employing this material had a positive effect on the functional properties of HMA [13]. Another study evaluated the fatigue performance of HMA modified using acidic sludge. The four-point bending test results showed that acidic sludge's application had no adverse effect on the fatigue performance of asphalt mixtures [14].

Moreover, the effect of acidic sludge powder (amorphous carbon powder) on the moisture sensitivity of asphalt mixtures modified with ground tire rubber was studied in 2019. The results suggest that the use of these two recyclable materials not only reduces environmental damages but improves the resistance of the tire-modified asphalt mixture to moisture damages [15]. In a recent study, the effect of acidic sludge as a filler on stone mastic asphalt (SMA) mixtures was evaluated. The outputs of this research revealed that incorporating 50% of acidic sludge powder in filler improved the mechanical performance of SMA mixtures [16].

The need to modify the bitumen used in the pavement industry to improve its service life, considering climate conditions, is undeniable. Responding to a need to improve the plastic deformation resistance of the mixture containing RAS and its workability, amorphous poly alpha olefin (APAO) polymer, as a bitumen modifying additive, can increase the resistance of asphalt mixtures to plastic deformation and fatigue. Previous researches indicated that bitumen modified with this polymer has lower shear strength, and mixing temperature and an optimum workability at lower temperatures, as well as a good anti-aging property [17–20].

Toward elucidating previous research, this article evaluates RAS as an alternative to bitumen in WMA and examines the effect of this product on the mechanical properties and moisture sensitivity of asphalt mixtures containing APAO polymer. The investigation of APAO polymer and RAS and their functional comparison in asphalt mixtures was another objective of this study. Conventional bitumen tests, mechanical and durability tests of asphalt mixtures containing RAS and APAO were employed for this research.

2. Materials and Methods

2.1. Experimental Plan

Conventional bitumen tests, including penetration, softening point, and ductility tests, were performed on RAS-modified bitumen. Various mixture tests, including Marshall, moisture sensitivity (indirect tensile strength, retained Marshall stability, and Texas boiling test), resilient modulus, and permanent deformation tests (dynamic creep test and deformation strength rest), were carried out on more than 105 asphalt mix samples to investigate the mechanical and durability properties of asphalt mixtures containing RAS-modified bitumen. Figure 1 shows a scheme of the experiments performed in this study.

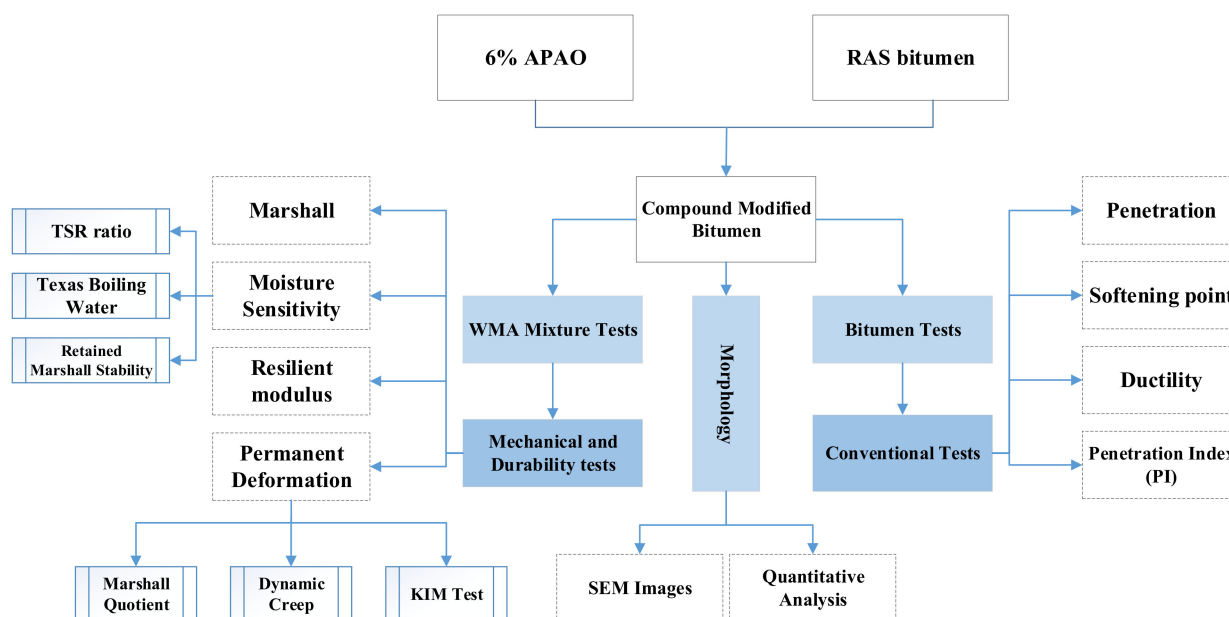


Figure 1. Experimental plan scheme.

2.2. Materials

2.2.1. Virgin Bitumen

A 60/70 penetration grade bitumen, as the virgin bitumen, was used in this study because of its highly favorable adaptation to a mild climate. It was manufactured by Pasargad Oil Company (Iran), and its basic properties are listed in Table 1.

Table 1. Properties of virgin bitumen.

Property	Value	Test Method	Unit
Specific gravity (25 °C)	1.03	ASTM D70	kg/m ³
Penetration grade (25 °C)	65	ASTM D5	0.1 mm
Softening point	49	ASTM D36	°C
Ductility (25 °C)	<100	ASTM D113	cm
Flash point	286	ASTM D92	°C

2.2.2. Re-Refined Acidic Sludge (RAS)

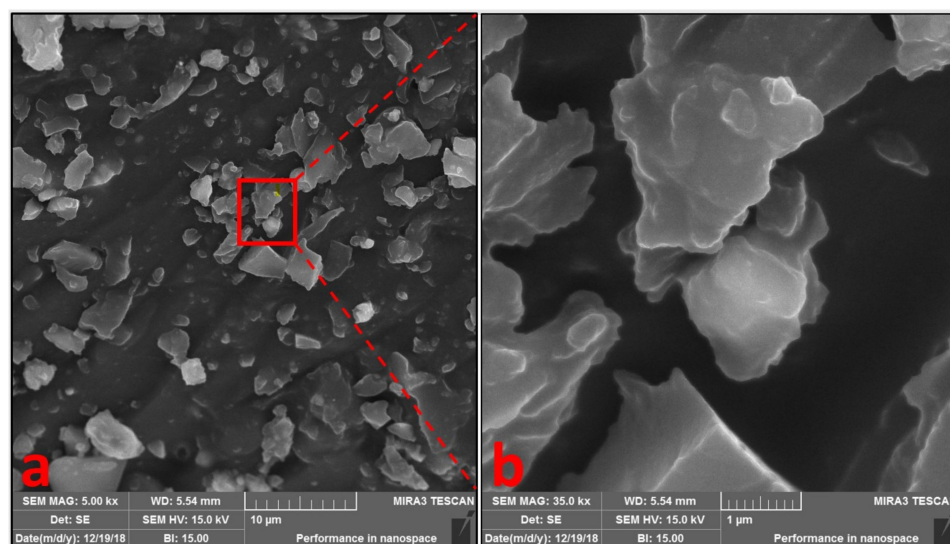
The RAS additive was supplied by the Alborz Green Company and has been patented in Iran. The RAS holds a pH close to 7, which is significantly larger than that of the original product (acidic sludge with a pH of close to 1). In Table 2, there are some information related to the RAS material properties, and in Figure 2, the appearance of the RAS is given at 80 °C.

Table 2. Properties of the re-refined acidic sludge (RAS).

Property	Value	Test Method	Unit
Color	Black	-	-
Odor	No scent	-	-
pH	<7	-	-
Physical shape	Semisolid (at 25 °C)		
Penetration grade (25 °C)	28	ASTM D5	0.1 mm
Softening point	78	ASTM D36	°C
Ductility (25 °C)	40	ASTM D113	cm
Melting point	109	ASTM D92	°C

**Figure 2.** The physical appearance of the RAS.

To investigate further, SEM images were used for analyzing the morphology of the RAS surface, and EDX (Energy Dispersive X-ray) spectroscopy was also carried out to access the sample chemical information and its spectroscopy. As observed in the microscopic photographs (1–10 μm) shown in Figure 3, the RAS surface is quite bumpy and forms a heterogeneous structure. Given the details about the surface of the material, it might be inferred that the bumpy surface of RAS signifies an improved level of moisture susceptibility brought about by hydrophobicity [21]. It is worth noticing that, due to the probable errors usually occurs in EDX technique, more techniques can be employed to have a precise experimental validation.

**Figure 3.** SEM images of RAS at (a) 10 μm and (b) 1 μm .

As shown in Figure 4, the highest percentages of the given result, carbon, calcium, sulfur, and a metal oxide, are the major constituents of this material in the presence of 70% carbon in RAS.

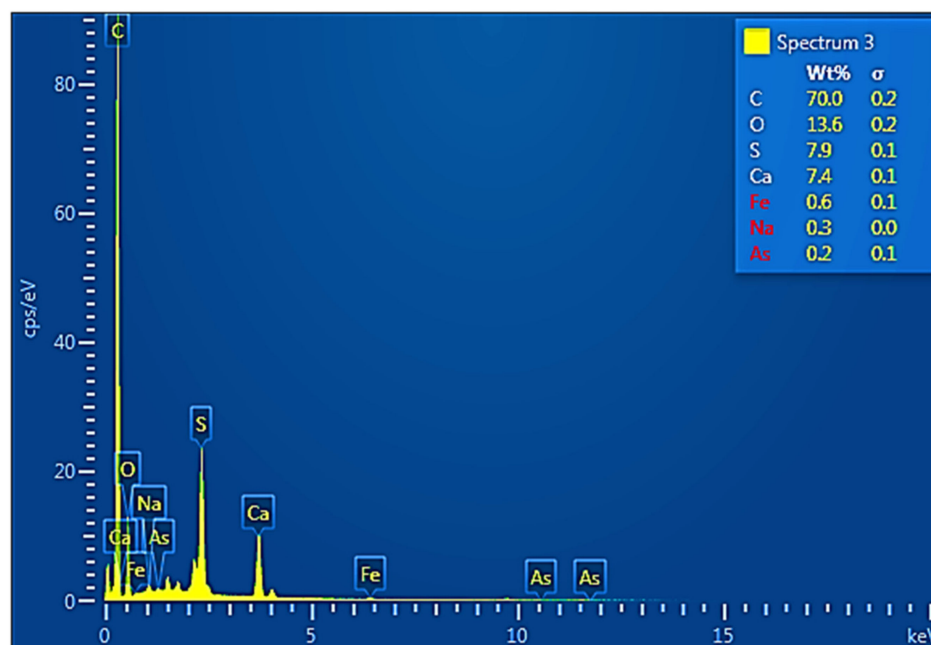


Figure 4. The spectrum (EDX) of the RAS.

Another noteworthy result of this spectrum is the presence of 13.6 wt % of oxygen (O), indicating various compounds (such as SiO_2 , Fe_2O_3 , CaO , and SO_3) that oxygen can be derived from. The effect of the calcium element at 6.9 wt %, under the composition of wollastonite (CaSiO_3), could increase the surface free energy of the material [22]. Based on previous research, increasing the surface free energy of material could increase resistance to moisture sensitivity [23]. However, it cannot be referred to just one or two elements for determining the impact of surface free energy on the moisture susceptibility of a material.

Inductively coupled plasma (ICP) spectroscopy is an atomic emission spectroscopy technique using an electric current to create a hot plasma environment through electromagnetic induction [24,25]. Comparatively, this method is more sensitive than others (in terms of EDX), and has a better detection threshold, and is better replicable thanks to its constant temperature. There are two groups of results yielded by the experiment presented in Table 3.

According to Table 3, there are low Pb levels of the hazardous metals cadmium, arsenic, and mercury. Heavy metal elements, such as iron, magnesium, nickel, zinc, copper, aluminum, manganese, and chromium, are compounds of this material. Interestingly, there is a high level of sulfur and calcium in this product, which exist as oxide compounds (CaO and SO_3).

Table 3. Elements and oxides of RAS.

Elements				Oxides			
Elements	Quantity (ppm)	Elements	Quantity (ppm)	Oxides	Quantity (ppm)	Oxides	Quantity (ppm)
Al	220	As	<0.1	MgO	530	Na ₂ O	515
Ba	15	Bi	<0.01	MnO	32	CaO	28,260
Ca	20,200	Cd	<0.01	Mo	105	P ₂ O ₅	1810
Mg	320	Co	<0.01	SrO	154	Li	2
K	88	Cr	33	V ₂ O ₅	<0.1	ZnO	1370
Li	1	Cu	88	Se	<0.1	Sc	<0.1
P	790	La	<0.01	Ga	<0.01	Bi	<0.01
Sb	<0.1	Mo	70	La	<0.01	Ag	<0.01
Se	<0.1	Fe	1160	Cr ₂ O ₃	48	NiO	19
S	19,100	Mn	25	CuO	110	Al ₂ O ₃	416
Sr	130	Ni	15	Fe ₂ O ₃	1658	K ₂ O	88
Sn	<0.1	Pb	30	PbO	35	Ba	17
Sc	<0.1	V	<0.1	SO ₃	47700	TiO ₂	30
Na	515	Zn	1100	SnO ₂	<0.1	Sb ₂ O ₅	<0.1
Ti	18	Ag	<0.01	Cd	<0.01	As ₂ O ₃	<0.1
Hg	<0.01	Ga	<0.01	CoO	<0.01	Hg	<0.01
LOI: 90.90%							

2.2.3. Aggregates

Aggregates as the main constituent of asphalt mixtures have an essential effect on its performance, durability, and stability. The aggregates, including filler used in this study, had a calcareous origin (i.e., limestone). The gradation curve of the mixture is shown in Figure 5, which was selected based on Iran Highway Asphalt Paving Code (No. 234) [26].

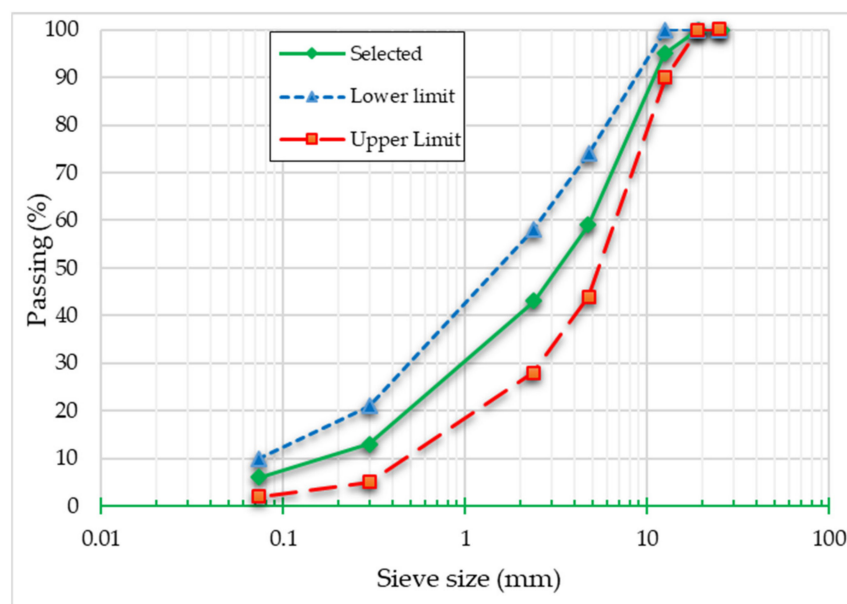


Figure 5. Gradation curve of aggregates.

The chemical properties of aggregates, their gradation, and physical properties significantly impact asphalt mixes' performance. In this study, X-ray fluorescence (XRF) was used to determine the aggregates' chemical properties. The physical and chemical properties of the aggregates used in this study are shown in Tables 4 and 5.

Table 4. Physical properties of aggregates.

Description	Test Result	Test Method	Unit
Los Angeles Abrasion value	22.3	ASTM C131	%
Particle shape flakiness index	16	BS 812	-
Fractured particles in coarse aggregates	93	ASTM D5821	%
Max. water absorption of coarse aggregates	2.2	AASHTO T85	%
Max. water absorption of fine aggregates	2.4	AASHTO T84	%

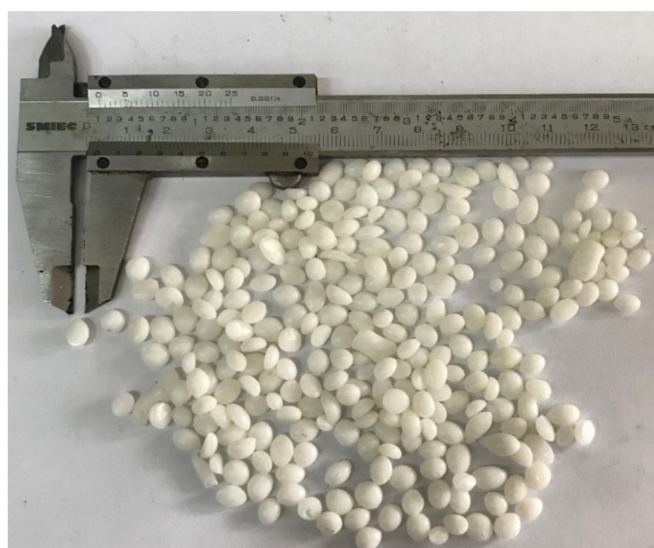
Table 5. The chemical composition of coarse aggregates (XRF).

Component	CaO	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	MgO	SO ₃	K ₂ O	L.O.I
Value %	49.05	10.19	0.714	1.203	0.769	0.098	0.397	37.47

2.2.4. Amorphous Poly Alpha Olefin (APAO)

APAO polymer is a non-crystalline and saturated plastic material, and, as a polymeric bitumen modifier can be easily mixed with bitumen at 145 °C. APAO polymer was also used to improve the asphalt mix's properties (containing RAS).

The polymer provided for this study was Evonik Vestoplast® EP-901 with granulated particles. Previous researches evaluated APAO polymer-modified bitumen at 2–8%, 6% of which are in good adaptability with bitumen; thus, 6% of APAO polymer was used in this research work [20,27]. Figure 6 shows this polymer's appearance, and Table 6 presents its technical properties.

**Figure 6.** Amorphous poly alpha olefin (APAO) polymer.**Table 6.** Technical properties of APAO polymer.

Property	Result
Solubility (in bitumen)	15–20%
Stirrer	Ordinary stirrer (800 RPM)
Stability	Stable, without double bonding
Thermal Stability (High temperature)	Very good—No loss below 60 °C
Thermal Stability (Low temperature)	Up to 15 °C
Anti-aging properties	Proper durability against ultraviolet and heat
Softening point [°C](DIN EN 1427)	117 ± 4
Penetration grade[mm/10](DIN EN 1426)	8 ± 2
Viscosity [mPa·s@190 °C] (DIN 53 019)	1000

2.3. Sample Preparation

In this study, in order to produce WMA samples, Sasobit was added to virgin bitumen as an organic additive. 1.5% of Sasobit (by weight of bitumen) was blended through the dry method according to previous studies [28–30]. For mixing bitumen and APAO polymer, a shear mixer at 800 rpm for 5 min was used in the laboratory. Moreover, the amount of APAO used in this study was 6% of bitumen containing RAS.

The asphalt mixtures were produced using the standard Marshall mix design procedure, according to ASTM D1559. The optimum binder content (OBC) was selected based on Marshall stability and flow, density, and void analysis. Table 7 shows the designator and the OBC of each mixture that had light changes in OBC (less than 0.2%); therefore, the OBC of 5.1% was selected for all mixtures.

Table 7. Mixture design properties.

Mix Designator	RAS (%)	APAO Content (%)	OBC (%)
W0	0	0	5.2
WA	0	6	5.2
WA10	10	6	5.2
WA20	20	6	5.1
WA30	30	6	5.05

It should be noted that using 40% and higher RAS contents in the mixtures did not allow an ease mixing with the aggregates. Moreover, a sample containing RAS in the WMA (without polymer) was manufactured, but its coating was not good so that it was not taken into consideration in the testing plan.

2.4. Tests

2.4.1. Conventional Bitumen Tests

The penetration grade (25 °C), softening point test, and ductility of bitumen at 25 °C were conducted according to ASTM D5, ASTM D36, and ASTM D113 standards, respectively. PI index, which was calculated by the obtained results of penetration grade and the test results of softening point tests [31], was employed because of possible changes in temperature susceptibility of the bitumen due to polymer modification and its effects on the penetration grade and softening point changes [32–34].

2.4.2. Marshall Test

Marshall test was performed using the Marshall mix design method according to ASTM D1559 standard. After mixing, both sides of each sample were compacted by 75 blows dealt with a Marshall hammer to simulate heavy traffic.

2.4.3. Moisture Sensitivity

Asphalt mixtures show a problematic issue in the presence of water. In this research, three different experiments were conducted to evaluate asphalt mixtures' moisture sensitivity.

Texas Boiling Water Test

Texas boiling water test, which is performed according to ASTM D3625 standard, is a simple, inexpensive method to evaluate the effect of water on bitumen-aggregate's adhesion. The purpose of this test is not an accurate analysis of mixture stripping but rather a quick and qualitative observation of mixture resistance against stripping. The image processing technique (MATLAB software) was employed to quantify the Texas boiling test results and precisely analyze the specimens before and after the test.

Tensile Strength Ratio (TSR)

This test, conducted in accordance with AASHTO T283, is used by roughly 81% of asphalt institutes in Canada and the United States, and is the most prevalent method

used for assessing asphalt mix moisture sensitivity [35]. Specimens (100 mm diameter and 63.5 mm height) were divided into two groups, unconditioned and freeze-thaw conditioned, and prepared with a target air void of 6.5–7.5%. The air void was obtained by adjusting the number of hammer blows in the AASHTO T245 standard method or changing the number of compression rotations in the AASHTO T312 standard method.

The specimens were vacuumed (70–80% of saturation) and were stored at $-18\text{ }^{\circ}\text{C}$ for 16 h. Then the specimens were taken out and immediately placed in a $60\text{ }^{\circ}\text{C}$ water bath for 24 h. Subsequently, after placing the specimens in the water bath at $25\text{ }^{\circ}\text{C}$ for 2 h, the samples' strength undergoing freeze-thaw cycle and without freeze-thaw cycle were tested at the loading rate of 50 mm/min. The indirect tensile strength of each sample was evaluated using Equation (1).

$$\text{ITS} = \frac{2000}{\pi t D} P, \quad (1)$$

where ITS is the indirect tensile strength (kPa), P is the maximum load (N), t is the sample thickness (mm), and D is the sample diameter (mm). The tensile strength ratio (TSR) (Equation (2)) was applied to examine moisture susceptibility of asphalt mixtures with a TSR threshold of 0.8.

$$\text{TSR} = \frac{\text{ITS}_{\text{Cond}}}{\text{ITS}_{\text{Uncond}}} \times 100, \quad (2)$$

ITS_{Cond} and $\text{ITS}_{\text{Uncond}}$ are average tensile strength of samples which undergo freeze-thaw cycle and those without a freeze-thaw cycle, respectively.

Retained Marshall Stability (RMS) Ratio

Retained Marshall stability test is a conventional test for assessing asphalt mix moisture sensitivity, which is a ratio of Marshall stabilities for conditioned specimens (kept in a $60\text{ }^{\circ}\text{C}$ water bath for 24 h) to unconditioned specimens (immersed in water at $60\text{ }^{\circ}\text{C}$ for 30 min) [36]. RMS value is calculated according to Equation (3), using the average stability of each group.

$$\text{RMS} = \frac{\text{MS}_{\text{con}}}{\text{MS}_{\text{uncon}}} \times 100, \quad (3)$$

where MS_{con} , MS_{uncon} , and RMS are Marshall stability for conditioned specimens (kN), for unconditioned specimens (kN), and retained Marshall stability (RMS), respectively.

2.4.4. Permanent Deformation

Dynamic Creep Test

Permanent deformation is known as rutting which is a significant failure in asphalt pavements. Among all methods developed for evaluating permanent deformation of asphalt mixtures, the dynamic creep test has become a familiar and essential experiment for assessing asphalt mixtures' resistance against rutting, which is performed according to BS EN12697-25A standard. The test provides an exceptionally accurate analysis of asphalt mixtures' rutting performance.

Samples were examined under 200 kPa of stress with a frequency of 1.0 Hz at $50\text{ }^{\circ}\text{C}$. Additionally, the samples had undergone a pre-loading process—which took 10 min and involved a static pressure of 10 kPa—before applying the main load. This experiment's results are presented as cumulative permanent strain curves against loading cycles.

Kim Test

Deformation strength test, a static experiment, is one of the simplest tests conducted to assess the rutting resistance of asphalt mixtures. This test aims to analyze the potential of permanent deformation of asphalt mixtures [37–39]. The loading frame of the KIM test and the performance of loading are shown in Figures 7 and 8 respectively.



Figure 7. The loading frame of the KIM test.



Figure 8. Loading performance of the Kim test; (a) before and (b) after loading.

This test comprised submerging the specimens in a 60 °C water bath for 30 min, drying the specimens' surface, placing the samples in a loading frame with a 40 mm diameter head and a curvature radius of 10 mm, and loading the samples with a rate of 50.8 mm/min. The value of deformation strength was subsequently calculated according to Equation (4). Samples with higher deformation strength levels demonstrated better resistance to rutting and permanent deformation.

$$S_D = \frac{0.32 P}{\left[10 + \sqrt{20y - y^2}\right]^2}, \quad (4)$$

where S_D is deformation strength (MPa), P is the maximum load (N), and y is the vertical deformation (mm).

According to research previously conducted by Fakhri et al. in 2017, the rutting resistance determined by the static test (Kim test) was correlated perfectly ($R^2 = 0.93$) with the parameters obtained from the wheel track test [28]. Figure 9 shows the Pearson

correlation calculated in their study. Kim's test results could provide a proper estimation of asphalt mixtures' rutting damage potential.

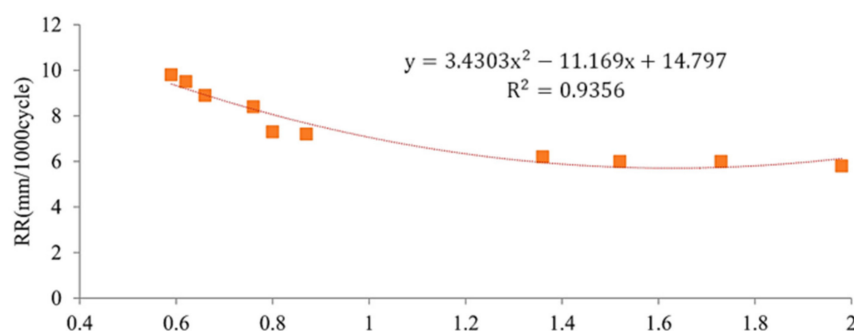


Figure 9. Kim test and wheel track correlation [28].

Marshall Quotient

Marshall Quotient (MQ) is the Marshall stability ratio to the Marshall flow of asphalt mixtures. The higher the MQ value of a mix, the higher the stiffness and resistance to deformation and, subsequently, higher resistance to rutting [40–42].

2.4.5. Resilient Modulus (M_R)

The resilient modulus (M_R) is a critical parameter in flexible pavement design and performed according to ASTM D4123 standard using UTM 14 in this research. The resilient modulus test is usually conducted at two temperatures, 25 and 40 °C. At 25 °C, the resilient modulus could be related to the mixture's resistance to fatigue [43,44]. After preloading the samples, the average M_R was measured in 5 loading cycles. Afterward, samples were rotated 90 degrees, and their resilient modulus was re-measured. The required values of this experiment are summarized in Table 8.

Table 8. Resilient modulus test parameters.

Parameter	Value
Loading pattern	Semi-sinusoidal
Loading duration (s)	0.15
Rest duration (s)	0.9
Number of preloading cycles	50
Applied load (N)	450
Poisson's coefficient	0.35
Test temperature (°C)	25

The resilient modulus is calculated from Equation (5).

$$M_R = 1000 \times P \times (\vartheta + 0.27) \times \frac{H}{\Delta H}, \quad (5)$$

where M_R is resilient modulus (MPa), P is the applied load (N), ΔH is the average reversible deformation per cycle, ϑ is Poisson's coefficient, and H is the sample height [45].

3. Results and Discussion

3.1. Conventional Bitumen Tests

Increasing the percentage of bitumen in modified asphalt mixes reduces the degree of penetration, which means that RAS will increase the bitumen's hardness (viscosity). According to previous studies, the APAO polymer also reduces the penetration degree [18].

As shown in Figure 10, reduction in penetration grade means reducing the flow and increasing the viscosity and consistency at medium temperatures. Adding only 10% RAS

can turn a 60/70 bitumen into a 40/50 bitumen. The lower penetration of bitumen improves its elastic behavior, increasing the asphalt resistance to permanent deformation and rutting at temperature of 25 °C.

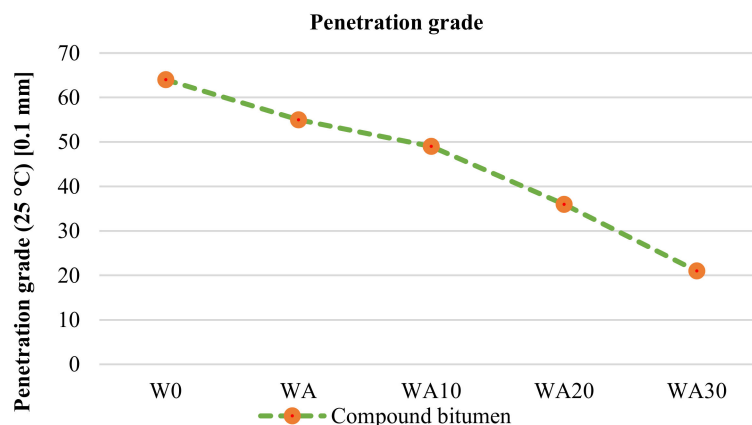


Figure 10. Penetration grade of bitumen.

The changes in the softening point of neat bitumen with increasing percentages of RAS, according to Figure 11, indicated a decrease in the thermal sensitivity of the modified bitumen. Increasing the softening point means that the performance of the bitumen viscosity is delayed by increasing temperature.

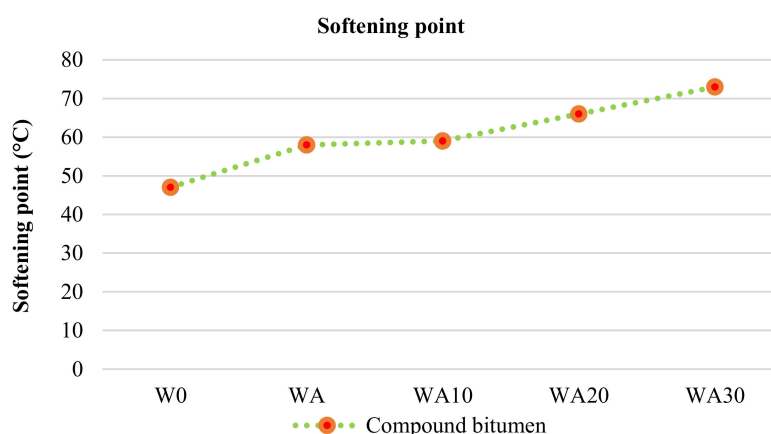


Figure 11. The softening point of bitumen.

PI index is obtained from the results of penetration tests at 25 °C and the softening point temperature of bitumens, and it is also assumed that the penetration grade of bitumens at their softening point temperature is equal to 800 dmm. The smaller the number obtained, the higher the thermal sensitivity of the desired bitumen is, which is different for each bitumen. The PI index is derived from Equation (6).

$$PI = [1952 - 500 \log (\text{Pen}25) - 20 \text{ SP}] / [50 \log (\text{Pen}25) - \text{SP} - 120], \quad (6)$$

Pen25: Penetration grade at 25 °C. SP: Softening point.

The PI values for bitumen used in pavement construction are usually in the range of −2 to +2, which increases with decreasing the degree of penetration of this number [34], and the higher the PI value, the lower the thermal sensitivity of the bitumen. Figure 12 shows the changes in PI versus the percentage of additive. As can be seen, modified bitumens have lower thermal sensitivity than pure bitumens. Due to the increase in PI index after adding bituminous acid sludge with polymer, it can be concluded that RAS can improve the thermal sensitivity of neat bitumen 60/70.

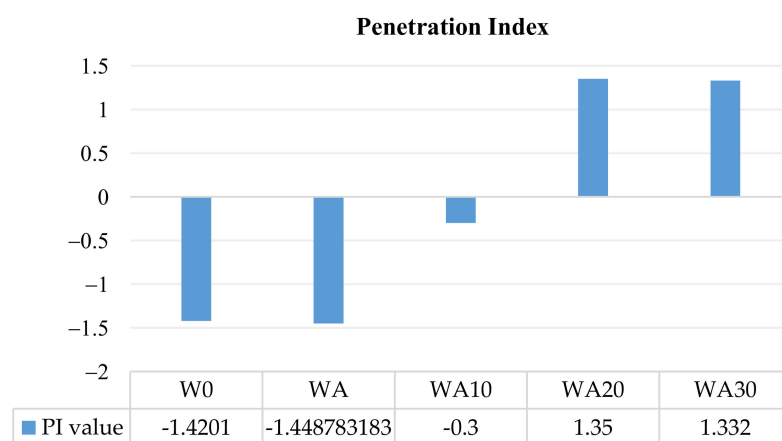


Figure 12. PI values of bitumen types.

Ductility is the length of a specimen just before it is torn in centimeters. The results given in Figure 13 showed that modified bitumen has lower ductility, which indicates the lower potential of this compound for resistance to cracking in cold regions.

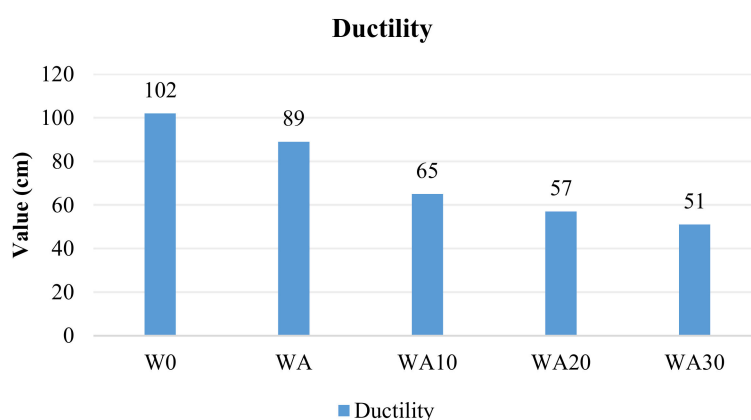


Figure 13. Ductility test results.

3.2. Marshall Test

Marshall test results are given in Table 9, which shows the samples containing RAS and polymer have higher stability. According to these results, all samples meet the minimum requirement of 800 kg (80 kN) of Marshall stability based on the Iran Highway Asphalt Paving Code (No. 234) [26]. According to the obtained results, the increase in RAS amount causes an increase in the resistance of the Marshall stability, and the highest amount is related to samples containing 20% of RAS. Additionally, the application of APAO significantly enhanced high-temperature performance.

Table 9. Marshall stability and flow results.

Sample	Marshall Stability (kN)	Flow (mm)
W0	9.624	3.2
WA	11.079	3.0
WA10	13.376	2.9
WA20	15.562	2.6
WA30	14.509	2.7

3.3. Moisture Susceptibility Test

3.3.1. TSR Ratio

The ITS test was performed based on the AASHTO T283 standard, and the results of this experiment were used to evaluate the mixture resistance to moisture. For each of the mixes, six samples were prepared, and three of them were conditioned. As observed in Figure 14, mixture resistance in modified samples is higher than the control sample, both in conditioned and unconditioned samples.

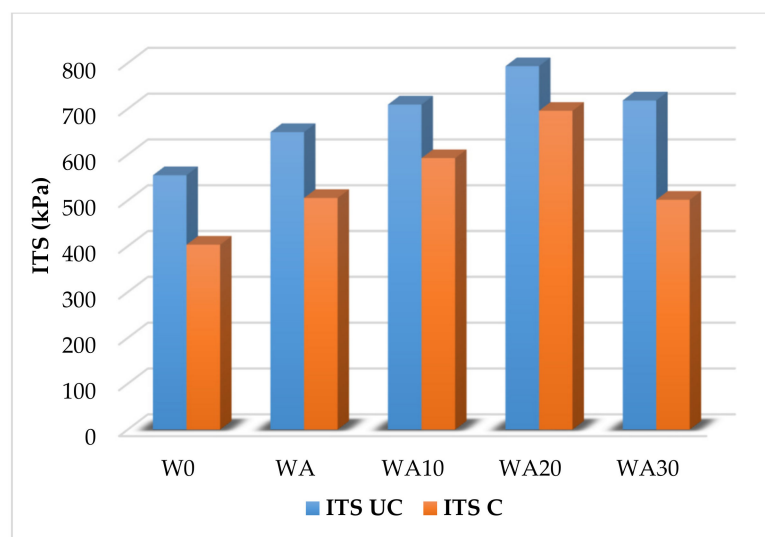


Figure 14. Indirect tensile strength (ITS) test results of conditioned (C) and unconditioned (UC) specimens.

The TSR ratio is defined as an index to evaluate the performance of asphalt mixtures against moisture damage. The minimum amount of TSR for the satisfactory durability of asphalt mixtures is 80% based on Iran Highway Asphalt Paving Code (No. 234) [26]. TSR values given in Figure 15 show that the sample containing 20% of RAS and polymer has the highest TSR.



Figure 15. Tensile strength ratio (TSR) of asphalt mixtures.

3.3.2. Texas Boiling Water Test

To have a precise analysis of this test, colored photos were rendered in black and white with a pixel range of 0–225, using MATLAB software. Figure 16 shows colored, black-white, and binary images related to one of the boiling water test specimens in which black and white areas refer to the coated and uncoated parts of the aggregates with bitumen, respectively.

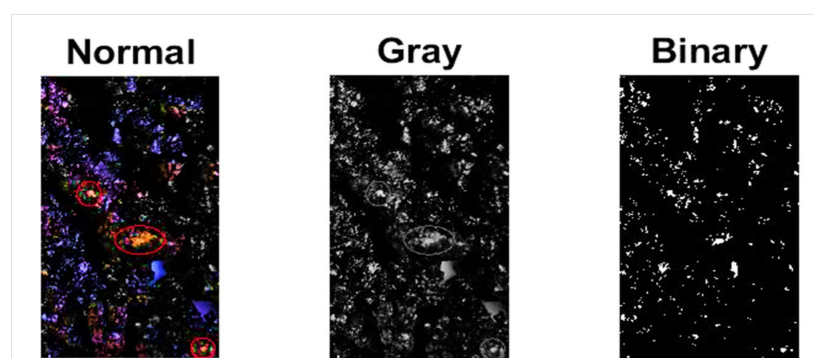


Figure 16. Colored, black-white, and binary images processed in MATLAB.

Table 10 shows the numerical analysis results of samples with different percentages of RAS and polymer. After boiling in water due to the hydrophobicity properties of RAS, it leads to a significant decrease in the white pixels, which shows the better coverage of bitumen coating.

Table 10. Image processing results in boiling water experiment.

Sample	Aggregate Coating (%)
W0	72.3
WA	75.36
WA10	77.5
WA20	86.1
WA30	79.8

The results show that 20% RAS had the best results of covering bitumen on the aggregates, and the control sample as well as 30% RAS, did not satisfy the criteria of 85% of the bitumen coverage around aggregates.

3.3.3. Retained Marshall Stability (RMS) Ratio

As stated before, one of the evaluation approaches of moisture sensitivity of asphalt mixtures is the RMS test. The test values of conditioned (24 h immersed) and unconditioned (30 min immersed) samples, along with RMS values, are reported in Table 11. The recommended value of RMS is 80% [46]. The mixtures containing RAS and polymer had better results than those of the control sample. Among them, 20% of the RAS sample had a better performance in this test, which is adapted to other moisture susceptibility tests done in this study.

Table 11. Retained Marshall stability (RMS) test results.

Sample	Unconditioned Sample Test Value (kN)	Conditioned Sample Test Value (kN)	RMS (%)
W0	962.40	810.53	84.19
WA	1129.68	963.34	85.27
WA10	1337.76	1167.23	87.25
WA20	1556.26	1368.92	87.96
WA30	1450.92	1211.62	83.5

As mentioned before, AASHTO T283, Texas boiling, and the RMS tests were employed to evaluate the asphalt mixture's performance against moisture sensitivity. The noticeable point in all three experiments was the positive effect of adding RAS mixtures, in which 20% of the RAS was the optimum percentage to achieve the best performance against water sensitivity among the specimens.

3.4. Permanent Deformation

3.4.1. Kim Test

The results of the Kim deformation resistance test are shown in Table 12. Based on the results, the mixture's deformation resistance decreased after adding 10% of RAS, but with the addition of RAS by 20%, led to a decrease in resistance. The mixture's strength with acid sludge bitumen increased by 30% compared to the control sample, but less than the strength of the mixture with 20% of acid sludge bitumen, which shows that using 20% RAS could be considered as the optimal percentage. Adding 30% of RAS to the mixture increases the mixture's hardness and reduces the workability. Therefore, the mixture's strength has decreased due to a lack of proper mixing.

Table 12. Values of force, displacement, and resistance in Kim test.

Sample	Stability (kN)	Y (mm)	S _D (MPa)
W0	4634	2.72	5.22
WA	4289	2.58	4.92
WA10	3955	3.62	4.04
WA20	5442	2.77	6.09
WA30	5054	3.14	5.41

3.4.2. Dynamic Creep Test

In this study, the dynamic creep test has been employed according to the specifications stated previously to evaluate the rutting susceptibility of the mixtures. As shown in Figure 17, except for the control sample, none of the samples entered the third stage of the curve and did not experience failure. The dynamic creep test was continued up to 10,000 cycles, and the cumulative strain of each sample was recorded. The results show that increasing the percentage of RAS up to 20% reduces the cumulative strain, and then, by increasing the percentage of acid sludge by 30%, the cumulative strain increases.

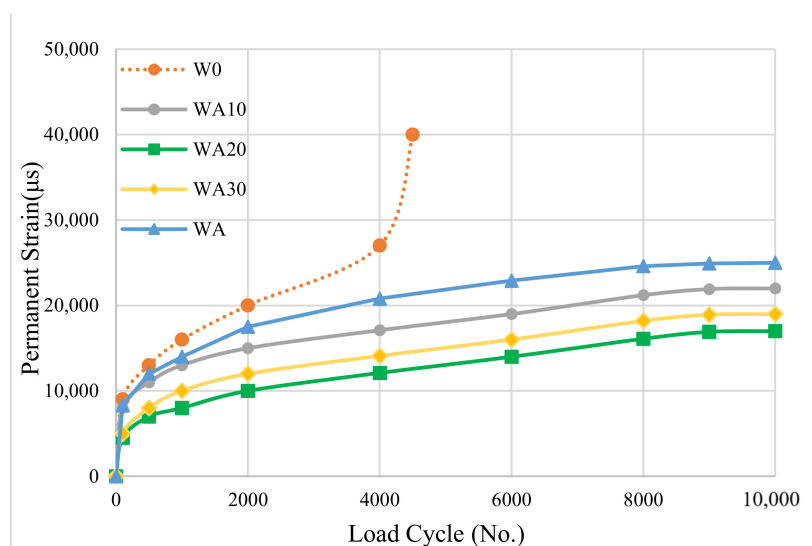


Figure 17. Permanent strain vs. load cycle.

It can be concluded that adding RAS to the asphalt mixture up to 20% could improve the resistance of the mixture to permanent deformation due to increasing the stiffness of the sample mixtures, and adding 30% of this additive due to high stiffness caused problems in workability and compression process. The lower compaction of the asphalt mixture makes the mix less resistant to deformation, so adding 30% RAS cannot reduce permanent deformation resistance. Therefore, the use of 20% of RAS can be considered as the optimal

percentage to achieve the best rutting resistance, which is entirely consistent with the result obtained from the Kim test.

3.4.3. Marshall Quotient

According to Marshall stability and flow tests results, MQ values are calculated and presented in Figure 18. The value of MQ of the modified mixtures is higher than that of the control mix. The MQ is a measure of the material's resistance to shear stresses, permanent deformation, and rutting [47].

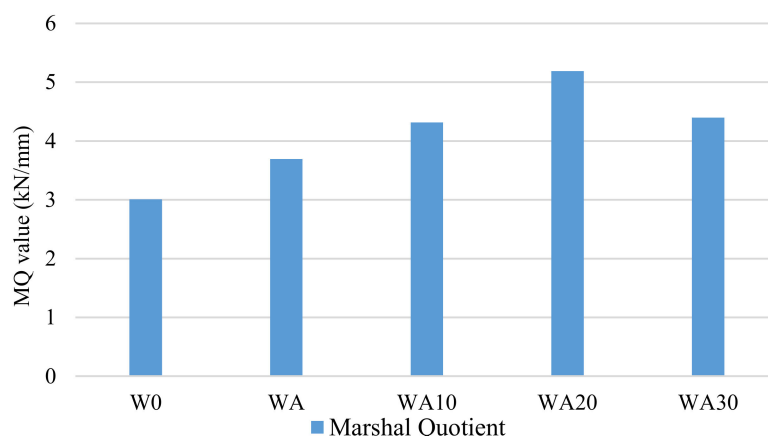


Figure 18. Marshall quotient.

3.5. Resilient Modulus (M_R)

The resilient modulus test was conducted at 25 °C. As shown in Figure 19, increasing the percentage of RAS and in turn increases the resilient modulus of the samples compared to the control sample. In other words, the addition of polymer and RAS increases the stiffness and results in higher resistance of the mixture to permanent deformation. However, the excessive increase in the modulus of resistance reduces the pavement's flexibility and makes it brittle.

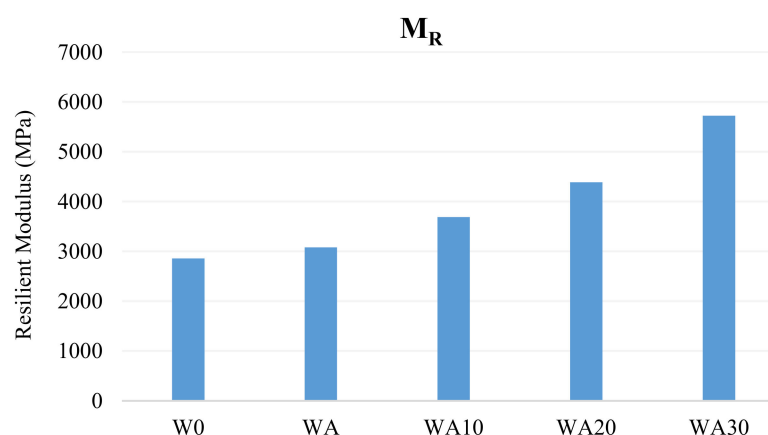


Figure 19. Results of resilient modulus (M_R) test of asphalt mixtures.

4. Conclusions

This preliminary study conducted a multiple laboratory performance evaluation of RAS modified binder (i.e., conventional bitumen tests), as well as the moisture susceptibility and permanent deformation properties of WMA mixtures containing APAO and RAS were analyzed. The results of the experiments performed in this research are as follows.

1. The addition of RAS and APAO to bitumen reduced the degree of penetration, and the lowest penetration value is related to bitumen contained 30% of RAS and polymer.

Moreover, samples containing RAS additive had a higher softening point than the control sample, with the highest amount at 30% of RAS, indicating a more rigid bitumen that results in this compound's lower potential for resistance to cracking in cold regions. Based on PI calculation, increasing the percentage of RAS has a more favorable effect on bitumen's thermal sensitivity.

2. RAS and polymer modified bitumen have increased the asphalt mixture's Marshall stability compared to the control sample, with the highest increase (about 50%) in stability is related to the mixture containing 20% RAS and APAO.
3. The addition of RAS and APAO to the asphalt mixture has increased the resistance of asphalt mixture against moisture damage, supported by results from the conducted tests in which 20% RAS had the superior durability. Morphology and chemical analysis also show more details about the RAS. It could increase the moisture resistance in asphalt mixtures but for proving that, it needs more investigations.
4. Samples with RAS have a higher M_R due to their higher stiffness than the control sample, which indicates that the modified asphalt mixture had appropriate resistance to permanent deformation, which can be a suitable mixture for paving with less thickness.
5. The tests related to permanent deformation showed that the addition of 20% RAS and APAO had an excellent effect on the asphalt mixture resistance against permanent deformation. Moreover, the Kim test results were compatible with the dynamic creep test as an important test for determining the rutting potential of asphalt mixtures.
6. Based on the laboratory test results, the APAO in combination with RAS modified asphalt mixture had better permanent deformation resistance at high temperatures and moisture stability than that of the control mixture. However, this is a laboratory-based study, and the environmental effects of these additives should be carefully considered in the future.

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