NTU Journal of Pure Sciences
Double Blind Peer Review Journal
ISSN: 2789-1089 EISSN: 2789-1097
Available Online: https://journals.ntu.edu.iq/index.php/NTU-JPS
Volume 5, No. 1, 2026

Original Article

The effect of adding petroleum resins C9 and C5 on the properties of Qayyarah asphalt

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Received: 12 April 2025
Accepted: 30 May 2025
Published: 12 April 2026
DOI: https://doi.org/10.56286/h64dhy69
Article ID: 1380

Abstract

The effect of petroleum resins on Al-Qayyarah asphalt was studied to modify its chemical, physical, and engineering properties. Due to climate change, rising temperatures, and increased loads, traditional asphalt has become unsuitable for paving main roads, leading to the search for alternatives or modifications to the properties of conventional asphalt. Therefore, petroleum resins of both aliphatic and aromatic types were used due to their rapid mixing compatibility and being solid materials. In addition, bonding materials like sulfur help with the interlocking and stability of asphalt mixing over time bonding petroleum resins with asphalt compounds through free radical reactions. Petroleum resins were used at a rate of up to 5% of the weight of the asphalt. The effect of both types was significant on the properties of the asphalt, as the aromatic resins had a considerable impact achieving a Marshall stability of up to 22KN and indirect tensile strength of 1.985KPa to determine its resistance to heat and moisture. Therefore, the addition of petroleum resins to Al-Qayyarah asphalt significantly improves the performance of the asphalt, provides excellent storage stability with the asphalt, and increases its load-bearing capacity.

Keyword: Asphalt, Rheological properties, Petroleum Resin, Load, Modify Asphalt

Introduction

Hot mix asphalt is considered one of the most widely used paving materials globally. Asphalt pavements are designed to withstand various types of loads, and modern highway transportation faces many challenges, such as high traffic density, heavy loads, and high speeds [1]. Asphalt is used as a binder in the mix, comprising about 5% of the total asphalt mixture, while the remaining 95% consists of aggregates and other additives used to enhance the asphalt mixtures [2]. The performance of asphalt pavement is influenced by several factors, such as the properties of its components (binder, aggregate, and additives) and the proportions of these components in the mix. The performance of asphalt mixtures can be improved using various types of additives, including polymers, latex, fibers, and many chemical additives [3]. In recent years, petroleum resins have been used as additives in various fields. They are highly compatible with asphalt, dissolving easily and quickly with it[4]. Petroleum resins can be distinguished based on the nature of their chemical components, such as aliphatic and aromatic types. They are used for specific purposes, such as a treatment material in the asphalt paving industry. Petroleum resins are literally used in thousands of applications, especially in low-cost adhesives that melt by heating [5]. Polymeric additives alter the physical and mechanical properties of asphalt structure. Among these are low molecular weight petroleum resins, which range between 500-2000 g/mol, enhancing compatibility with asphalt [6]. Petroleum resins have been used with styrene-butadiene-styrene (SBS) polymers in asphalt mixtures, improving compatibility between rubber and asphalt and enhancing physical and rheological properties [7]. They are considered a good, successful, and cost-effective alternative. They also represent a sustainable technology for greening asphalt, characterized by important properties such as resistance to rutting, cracking, and overall failure [8]. Petroleum resins improve compatibility with rubber polymers used in asphalt modification and increase T mixing and compaction temperatures[9]. Additionally, they extend the lifespan of asphalt and reduce early aging[10]. One of the most important applications of petroleum resins is in the production of colored asphalt, as they are transparent and nearly colorless [11]. Nie and others were able to prepare a blend of petroleum resins (SBS/C9 (SPR) as modifiers for high viscosity asphalt; this study gave a good rate for preparing high viscosity asphalt with high performance and low cost [12]. Guo, Lei and others were able to improve the rheological properties of asphalt using RPP and SBS. Dynamic shear rheometer (DSR), bending beam rheometer (BBR) and other instruments were used to evaluate the rheological properties of the modified asphalt composite. In addition, Fourier transform infrared spectroscopy (FTIR) and fluorescence microscopy (FM) were used to conduct microscopic analysis of the modified asphalt, and the layer analysis method was adopted to determine the optimum RPP content. The test results show that the rheological properties of asphalt significantly improved. Moreover, the cross-linking between polymer and asphalt was enhanced by the combined addition of RPP and SBS[13]. Zhang and Tan studied the rheological properties of asphalt when adding sulfur and (styrene-butadiene-styrene) (SBS), The results showed an increase in the compatibility between the additives and asphalt, besides an improvement in the performance of asphalt after adding sulfur to the asphalt modified with SBS[14] with theuse four percentages of sulfur (0%, 15%, 30%, 45%) of the weight of the asphalt binder. It was noted through the results that adding sulfur at a low percentage such as 15% leads to softening of the original asphalt binder. When the sulfur percentage is greater than 15%, the additional sulfur acts as a filler to strengthen the asphalt binder. The addition of rubber waste led to a significant increase in the performance at high temperatures with sulfurized asphalt [15].

Kumar and others also evaluated the stability of asphalt binders using waste ethylene and propylene rubber and waste thermoplastic oils (PPO). The effect of adding sulfur was also studied. It was noted through the results that these additives led to improving the rheological properties, including the degree of softness and stability of the modified asphalt [16]. Ahmed and others were able to modify Iraqi asphalt using sulfur waste (foam) resulting from sulfur refining from Mishraq field. The study started with a comprehensive analysis of the foam composition and used different diagnostic techniques. The rheological properties of the original and modified asphalt were determined by measuring properties such as ductility, softening point, penetration, penetration index, Marshall stability, chemical immersion, and aging. The modified asphalt showed rheological properties that qualified it for use in paving operations, especially in terms of acid rain resistance and stability [17]. In this study, we will conduct a comparative study on the effect of adding C9 and C5 petroleum resin to the asphalt of Al-Qayyarah.

Materials & Method

Materials Used

Qayyarah Asphalt with the specifications shown in Table (1):

Table 1. Rheological properties of Qayyarah asphalt.

Property	Result	ASTM Limits
Penetration (25°C, 100g, 5sec,mm)	40	40-50
Softening point, °C	55	50-58
Ductility (25°C, cm)	150+	100 min
Specific gravity	1.049	1.01-1.06
Flash point, °C	265	240 min

The petroleum resins used are:

C5 Aliphatic Hydrocarbon Resin) HSU(JIANGSU) CHEMICAL TECHNOLOGY CO., LTD. China)
C9 Aromatic Hydrocarbon Resin (HSU(JIANGSU) CHEMICAL TECHNOLOGY CO., LTD. China)

Sulfur element (Al-Mishraq Sulfur Company, Mosul, Iraq)

Aggregate crushed gravel and sand were used as aggregates and the gradation chosen in the study was in the middle of the ASTM D3515 limits.

calcium carbonate as filer.

Results & Discussion

Physical and Chemical Tests

The petroleum resins were added to the asphalt for each type at five different percentages, specifically 1%, 2%, 3%, 4%, 5%with the presence of 1% sulfur as a constant percentage across all experiments for both types. The following results were obtained:

Reaction of Asphalt with C9 and Sulfur:

The following results were obtained as in table (2):

Table 2. Rheological properties of adding C9 to Qayyarah asphalt.

%C9	*%S*	*Softening point ^0C [22]*	*Penetration: 100gm/5sc/25^{0}C [23]*	*Ductility Cm/25^{0}C [24]*
0	0	55	42	+140
1	1	61	37	+140
2	1	62	36	+140
3	1	64	35	+140
4	1	66	32	+140
5	1	67	30	+140

The mixing process between asphalt and C9 was accompanied by the appearance of bubbles and gases, which continued for about half an hour during mixing at 180°C,

and then disappeared, indicating a chemical reaction between the two. In addition to a physical change characterized by hardening and increased stiffness of the samples, the percentage of C9 increased. The figure shows the relationship between the change in permeability and the degree of ductility of Qayyarah asphalt treated with C9.

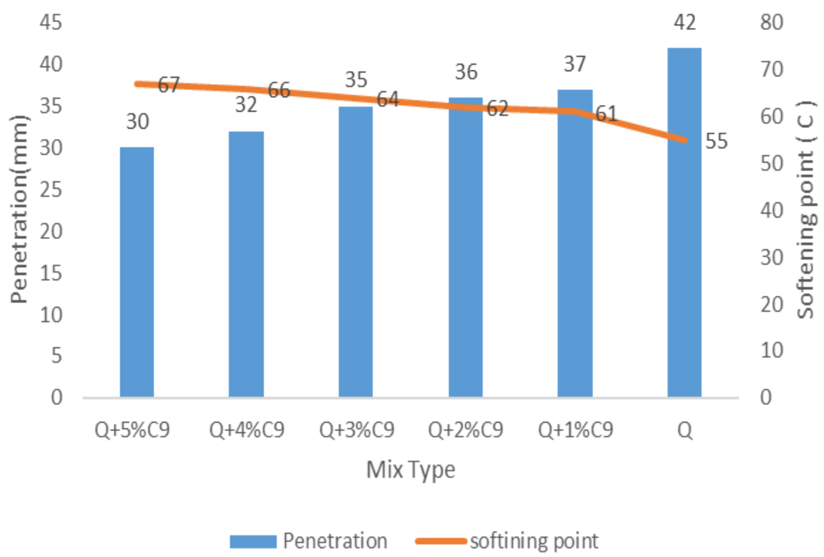

Figure 1. Illustrates the relationship between the penetration and softening point of Al-Qayyarah asphalt for mixtures C9.

C9 significantly affects the properties of the asphalt of Al-Qayyarah, where it raised the softening point to excellent degrees at (67 ºC) while maintaining good penetration and not collapsing at (30*0.1mm). The addition of sulfur applied in two primary ways. First, it facilitates oxidation reactions through dehydrogenation, leading to the formation of olefinic systems, particularly from the aliphatic fats in the asphalt, which are preferred over aromatic compounds. These olefinic systems enhance the interaction and cross-linking either among themselves or with the added resins. Second, sulfur induces vulcanization, binding asphalt and resin molecules together by the formation of sulfide compounds. Both mechanisms result in the formation of larger molecules, which in turn increases the hardness of the asphalt samples, thereby reducing the impact of oils within the asphalt and enhancing its stability [18][19][20][21].

Reaction of Asphalt with C5 and Sulfur:

The following results were obtained as in the table (3).

Table 3. Rheological properties of adding C5 to Qayyarah asphalt.

%C5	*%S*	*Softening point ⁰C [22]*	*Penetration 100gm/5sc/25⁰C [23]*	*Ductility Cm/25⁰C [24]*
0	0	55	42	+140
1	1	62	36	125
2	1	65	33	120
3	1	66	32	115
4	1	67	30	110
5	1	69	28	105

The effect of C5 is more severe on the asphalt of Al-Qayyarah and increases its hardness more than C9. This behavior is illustrated in Figure 2 below.

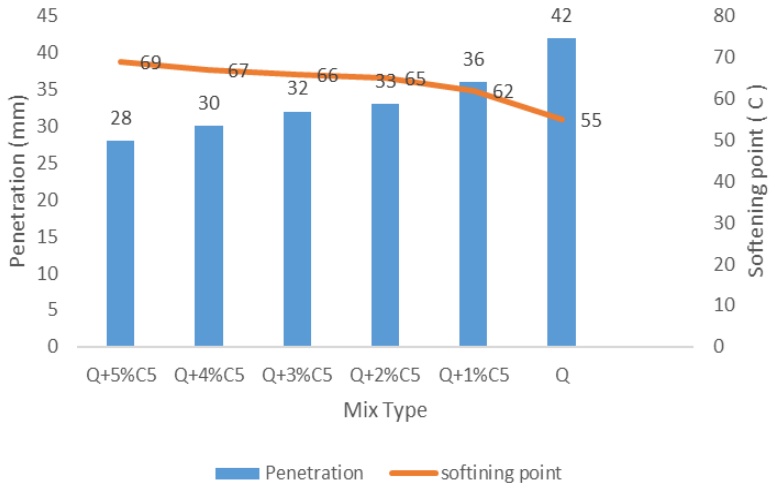

Figure 2. Illustrates the relationship between the penetration and softening point of Al-Qayyarah asphalt for mixtures C5.

figure (3) shows the relationship between the ductility of the base asphalt of grade C9 blends with the ductility of C5 mixtures, and C5 affects the ductility of the base asphalt while fully maintaining the ductility of the base asphalt with C9.

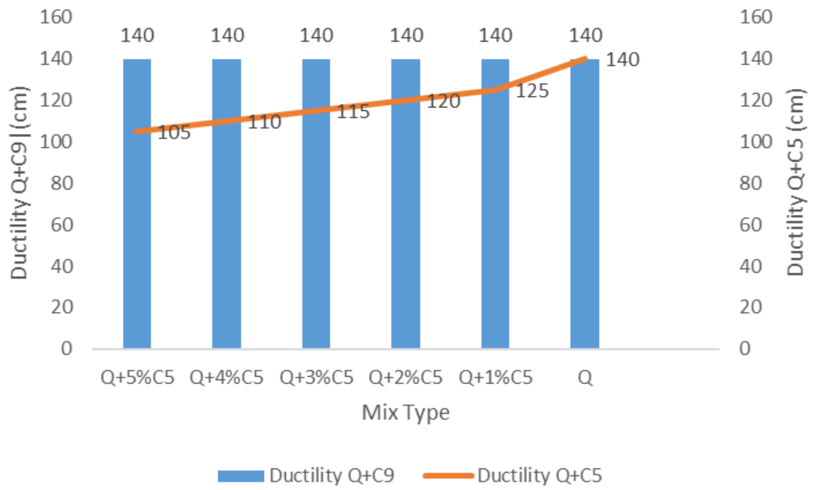

Figure 3. Effect of the Addition of C9 and C5 on the ductility of the Qayyarah Asphalt.

We note from the figures and tables above that the softening point values when adding C5 (69 °C) began to increase more than C9 (67 °C). This increase in softening point values can be attributed to the aliphatic nature of C5, as sulfur prefers it over C9, which is aromatic. Therefore, the hardness of C5 compounds with asphalt was higher, and these resins, due to their hardness, act as supporting materials for asphalt, increasing the degree of softening. The rate of change in penetration values was greater with C5, reaching (28*0.1mm) while C9 reached 300*0.1mm, which is a preferred number over the previous one. Thus, C9 aromatic resin has an advantage over C5 aliphatic resin in terms of penetration and applicability in real-world scenarios.

It appears that the rate of change in ductility values has started to decrease, reaching 105 cm with the aliphatic resin while maintaining high elongation with the aromatic resin C9. This indicates that the bonding rate between the aliphatic resin and the asphalt was higher, leading to the production of more interconnected systems and increasing the hardness of asphalt samples with C5. The reason for this is due to the aliphatic nature of C5, which oxidizes more than C9 with its aromatic nature because of the stability of the aromatic ring towards oxidation processes. Therefore, the bonding of C5 is certainly greater than the bonding of C9 with asphalt compounds. Sulfur does not react with the benzene ring except in the presence of catalysts such as aluminum chloride or in the presence of heat as a catalyst at 280c. Yetwe have not reached this temperature, so sulfur prefers aliphatic carbon over aromatic carbon [25].

Asphalt Mix Design:

The mix was designed using Marshall equipment with 75 blows per side according to ASTM D-6927[26] to determine stability and flow and to compare the effects of C9 and C5 with Qayyarah asphalt. The mixing was done at 160°C with 5% asphalt content. Three samples of each type were tested for Marshall Stability (MS), and six samples of each type were tested for Indirect Tensile Strength (ITS). Three samples were broken during the conditioned test after being heated at 60°C for 24 hours, and three samples were tested in an unconditioned state at 25°C. All tests were conducted according to ASTM 2015 standards.

Asphalt, Aggregate, and Filler Materials:

In this study Qayyarah asphalt was used with the specifications detailed in Table (1) above. Crushed gravel and sand were selected as aggregate materials. The chosen gradation for the study was centered within the limits specified by (ASTM D3515 – 01). Additionally, calcium carbonate was used as the filler material.

Performance Tests:

According to ASTM 2015, the following performance tests were conducted on the C9 and C5 mixtures

with Qayyarah asphalt:

1. Marshall Test (ASTM D-6927) [27].
2. Indirect Tensile Strength (ITS) Test (ASTM D6931 − 17) [28].

For the Marshall Test, three samples from each group were prepared. For the Indirect

Tensile Strength (ITS) Test, six samples were prepared from each group. Three of these samples were used for the unconditioned state, which were soaked in a water bath at 25°C for two hours. The other three samples were used for the conditioned state, where they were soaked at 60°C for 24 hours and then soaked at 25°C for an additional two hours. The samples were tested at a speed of 5 cm/minute until failure to determine the maximum load the sample could withstand. The Indirect Tensile Strength (ITS) was calculated using the following equation (Equation 1):

$ITS=2P/3.14dt$ …………… (1)

ITS represents the Indirect Tensile Strength (in Newtons per square millimeter, N/mm²).

P represents the maximum load (in Newtons).

d stands the sample diameter (in millimeters, mm).

t is the sample thickness (in millimeters, mm).

The effect of moisture sensitivity on the C9 and C5 samples was evaluated using the Indirect ITStest according to ASTM D6931−17. The Tensile Strength Ratio (TSR) was determined by comparing the ITS values of the conditioned group to the unconditioned group at 25°C using the following equation2:

TSR%=ITSC/ITSU*100 ……………. (2)

ITSC: Indirect Tensile Strength at 60°C

ITSU: Indirect Tensile Strength at 25°C

A higher TSR value indicates greater resistance to the effects of moisture.

Engineering Tests:

The engineering tests were conducted on the best ratios obtained at 5%.

Marshall Test:

The Marshall test was conducted for C9 petroleum resin, and the following values were obtained as shown in Table (4).

Table 4. Marshall test values for C9 mixtures with Qayyarah asphalt(Q).

Property	*Q*	*Q+1% C9*	*Q+2% C9*	*Q+3% C9*	*Q+4% C9*	*Q+5% C9*	*SCRB*
Stability, KN	11.8	13.1	14.9	16.8	20.1	21.98	8 min.
Flow, mm	3.05	2.9	3.4	3.25	4.2	3.1	2-4
Gmb, g/cm3	2.4	2.4	2.37	2.42	2.38	2.4	3-5
Air voids, %	4.1	4.2	4.3	4.5	3.8	3.37	…….
VMA, %	14.65	14.69	14.8	14.7	14.5	14.04	14 min.
VFA,%	69	70.1	69.8	69.3	70.7	71.85	65-85
Asphalt, %	5.0	5.0	5.0	5.0	5.0	5.0	5-6

Whereas:

SCRB= Specifications Required for Binders.

Gmb= Gravity maximum bulk= specific gravity

VMA= Voids in the Mineral Aggregate.

VFA= Percent Voids Filled with Asphalt.

The table above illustrates the Marshall stability properties of asphalt and its blends

containing C9 at different ratios, comparing them to the standard asphalt where the Marshall stability parameters have significantly improved compared to the original asphalt. The following graph shows the differences in Marshall stability between the original sample and the added percentages of C9.

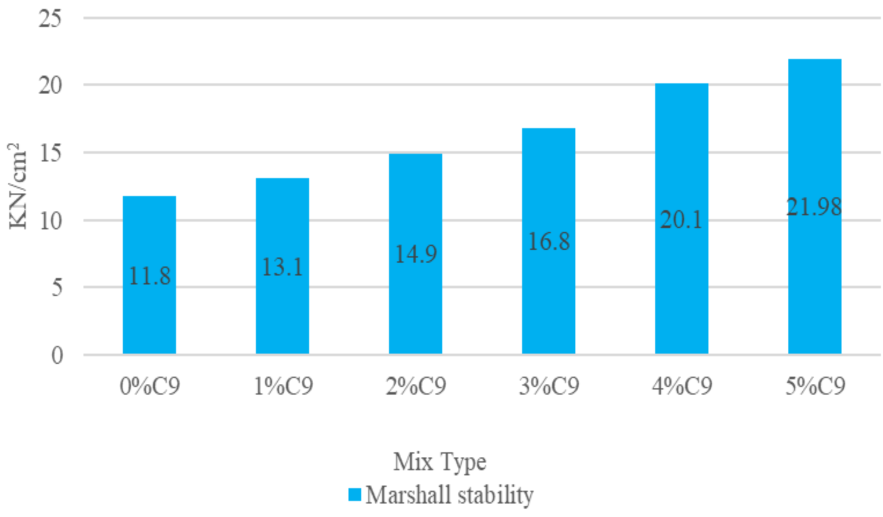

Figure 4. Illustrates the Marshall stability of C9 mixtures with the Qayyarah asphalt.

The effect of C9 is very clear on the Qayyarah asphalt, as all additives have a noticeable impact and significantly increase the stability of the Marshall models, which means a substantial increase in thermal resistance first and an increase in its ability to withstand loads second. However, the stability values of Marshall for the additions of C5 with the asphalt of Al-Qayyarah can be illustrated by the following table and figure.

Table 5. Marshall test values for C9 mixtures with Qayyarah asphalt(Q).

Property	*Q*	*Q+1% C5*	*Q+2% C5*	*Q+3% C5*	*Q+4% C5*	*Q+5% C5*	*SCRB*
Stability, KN	11.8	12.7	13.6	14.45	15.2	16.23	8 min.
Flow, mm	3.05	3.3	3.1	2.9	2.84	3.03	2-4
Gmb, g/cm3	2.4	2.5	2.46	2.5	2.43	2.37	3-5
Air voids, %	4.1	4.2	4.15	4.3	3.0	4.37	…….
VMA, %	14.65	14.9	14.78	14.98	14.79	14.94	14 min.
VFA,%	69	71	17.5	69.8	72	71.11	65-85
Asphalt, %	5.0	5.0	5.0	5.0	5.0	5.0	5-6

From the table above andthe effect of C5 petroleum resin, the Marshall stability values indicate that the effect of C9 is better. The difference in their effect on Marshall stability is attributed to the fact that the mixture containing C5 was more affected by the addition of sulfur than the mixture containing C9, because the aliphatic resin C5 is more affected than the aromatic C9, and therefore its strength weakens due to increased cross-linking and decreased hydrogen. So, the Marshall stability strength is higher as the latter is aromatic and its hydrogen content is lower than that of aliphatic C5. This results in more double bonds in C5, which is further intertwined with the presence of sulfur through vulcanization processes and the formation of interfacial sulfide compounds. On the other hand, C9 is not much affected by sulfur, but its hardness is lower than C5, as evidenced by the softening point and penetration degrees. In contrast, it maintained a ductility (140 cm) for all added ratios, which means that its elasticity is better than that of C5 mixtures. This statement is supported by the Marshall stability results, where C9 was better than C5, despite having higher softening point and lower penetration, so it is stiffer than C9. However, it breaks during the Marshall test before C9, supporting the higher tolerance and elasticity of C9 mixtures. Therefore, its addition is preferred over C5 for these reasons. Figure (6) illustrates the stability values of Marshall for C5 mixtures.

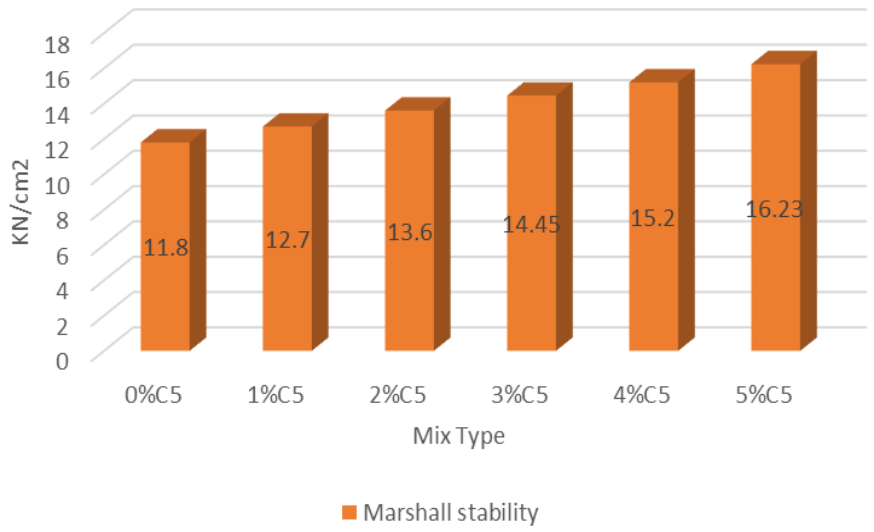

Figure 5. Illustrates the Marshall stability of C5 mixtures with the Qayyarah asphalt.

The figure above indicates that all additives meet the ASTM minimum requirements: 8 KN stability, 2-4 mm deformation, and 3-5% air voids. Therefore, all proportions are optimized for Qayyarah asphalt and can be adopted, especially if we know that petroleum resins are inexpensive compared to other additives.

Indirect Tensile Strength (ITS) Test:

The ITS test was used to evaluate the effect of humidity and temperature on C9 and C5 mixtures. Figure (7) shows the indirect tensile strength of conditioned and unconditioned asphalt samples Qayyarah, which contain different percentages of C9. This test helps assess the ability of asphalt mixtures to withstand cracking under the influence of humidity and heat, providing insight into their durability and performance in various environmental conditions.

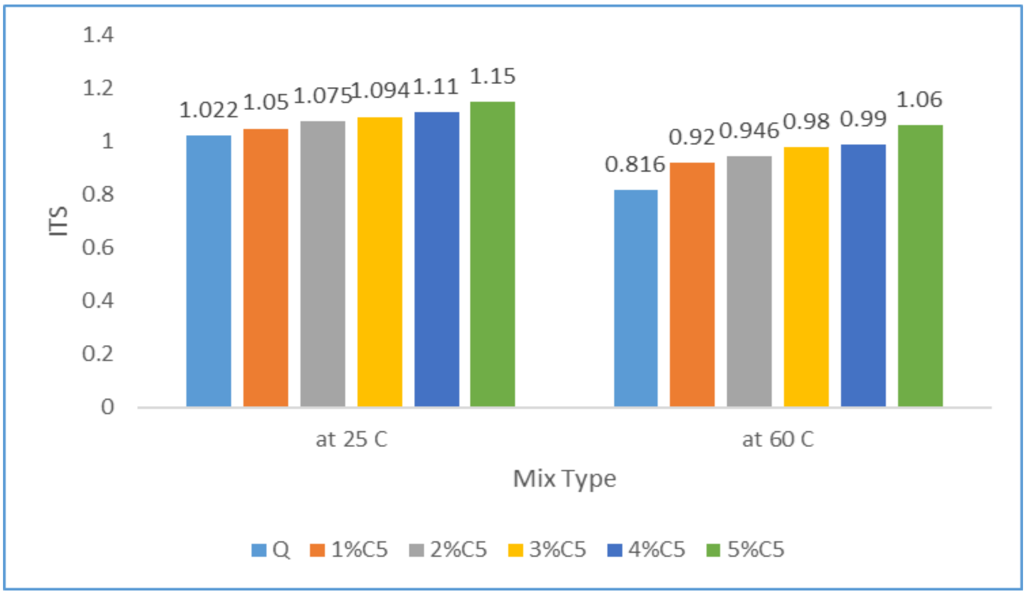

Figure 6. Illustrates the ITS values for the conditioned and unconditioned models of Qayyarah bitumen mixes and modified samples with C9.

The petroleum resin C9 has given good indirect tensile strength values, which confirms its transfer to a higher and better applicative level. What the Al-Qayyarah asphalt suffers from is its susceptibility to water over time; therefore, these figures elevate it to a higher and better level in terms of resistance to both moisture and heat. Figure (8) shows the indirect tensile strength values of C9 mixtures with Qayyarah asphalt.

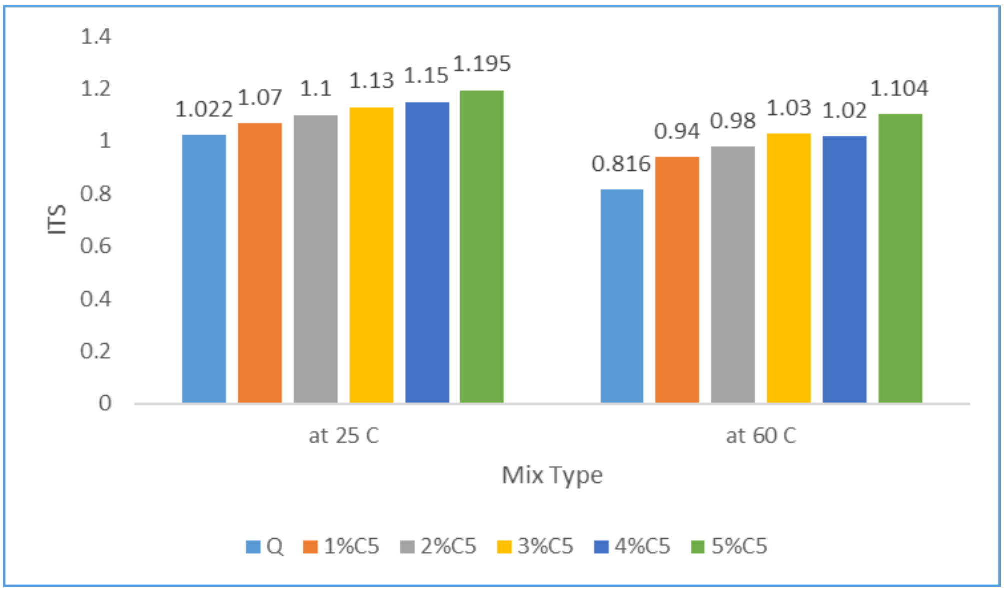

Figure 7. Illustrates the ITS values for the conditioned and unconditioned models of Qayyarah bitumen mixes and modified samples with C5.

The figures above indicate that the ITS values for the conditioned and unconditioned samples of C5 and C9 mixtures with Q asphalt are similar to each other and are significantly higher compared to the original Q asphalt. The ITS values for C5 mixtures are superior and higher than those for C9, indicating a substantial increase in resistance to damage caused by moisture, with a minimal impact from heat, water, and moisture on both C9 and C5 mixtures.

The moisture sensitivity of the C9 and C5 mixtures is assessed using the TSR (Tensile Strength Ratio) of the conditioned and unconditioned samples, as shown in figure (9).

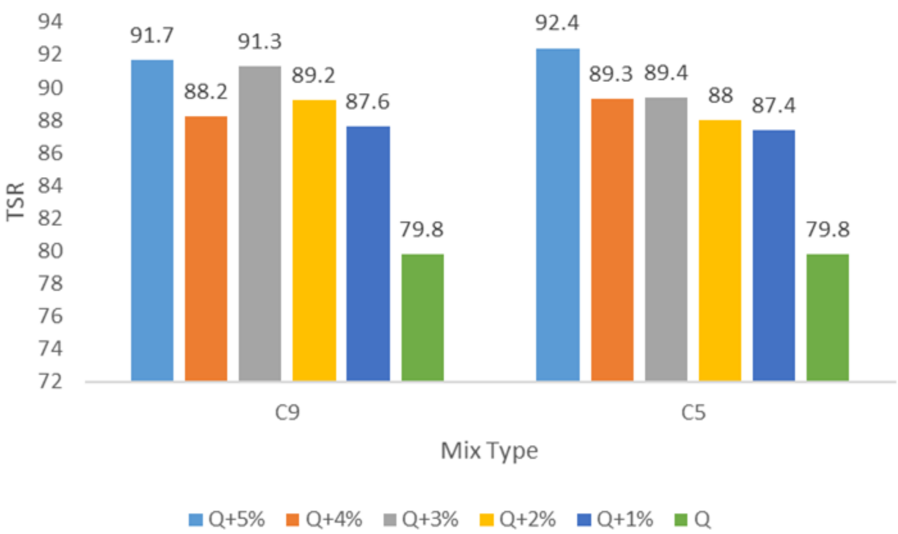

Figure 8. Tensile strength ratio of C9 and C5 mixes with Qayyarah asphalt.

C9 mixtures show a total tensile strength similar to C5, with both exhibiting comparable and doubled values compared to the original asphalt. Based on these results, C9 and C5 samples and mixtures demonstrate a high and sufficient moisture resistance, significantly improving the indirect tensile strength of both modified and unmodified Q asphalt samples, with a preference for C9, noting that Qayyarah asphalt fails in the indirect tensile test and these additives are a genuine solution to the problem of indirect tensile strength in Qayyarah asphalt.

Separation Tendency Test:

A stability test was conducted on the samples mentioned above, and all of them were successful, showing no signs of separation or change in consistency, as shown in the following table. The inspection was carried out according to the American standard (ASTM D7173-20)[29]. It is deemed that the additives have good dispersion if the difference in softening point between the lower and upper parts is less than 2.2 ᶜo. The results of storage stability can be explained in the following table:

Table 6. The change in softening point values after conducting a Separation Tendency Test.

Mix Type	*Softening point (ºC)*			
	Top (ºC)	**Bottom (ºC)**	**Difference (ºC)**	**Requirement<2.2 (ºC)**
Q+1%C9	61	61.5	0.5	pass
Q+2%C9	61.5	63	1.5	pass
Q+3%C9	64	64.5	0.5	pass
Q+4%C9	65.5	67	1.5	pass
Q+5%C9	67	67.5	0.5	pass
Q+1%C5	61.5	62	1.5	pass
Q+2%C5	65	65.5	0.5	pass
Q+3%C5	66	66.5	0.5	pass
Q+4%C5	66.5	68	1.5	pass
Q+5%C5	68	70	2	pass

The table above shows that all models successfully passed the Separation Tendency Test.

Scanning Electron Microscope Images

To illustrate the degree of homogeneity among all components, this test was conducted. The scanning microscope produces high-resolution images of the sample surface, revealing fine details ranging in size from 1 to 5 nanometers. The method of forming these images shows that scanning microscope images are three-dimensional, which helps in understanding the structure of the sample surface. As shown in the following images, they are extremely clear images, as if of a single material and not a mixture of several materials.:

Scanning Electron Microscope Images of the asphalt of Qayyarah with C9:

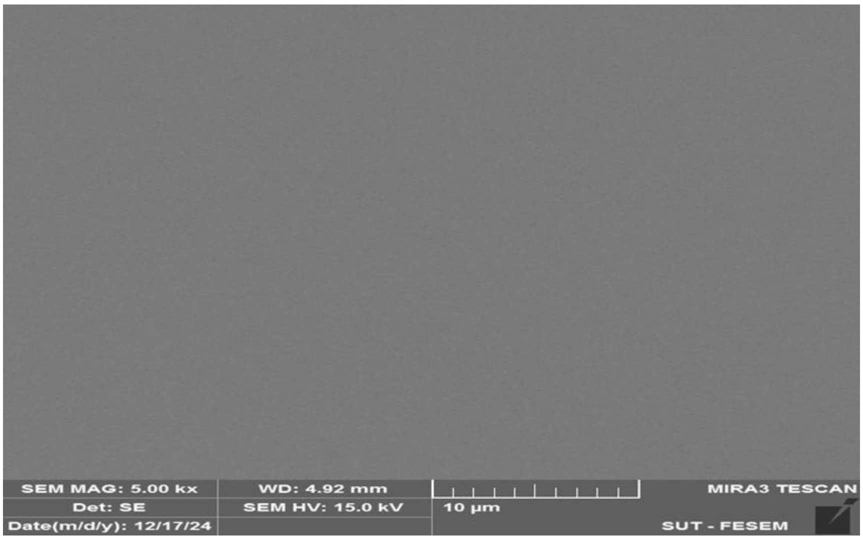

Figure 9. Scanning electron microscope images of Qayyarah asphalt modified with C9.

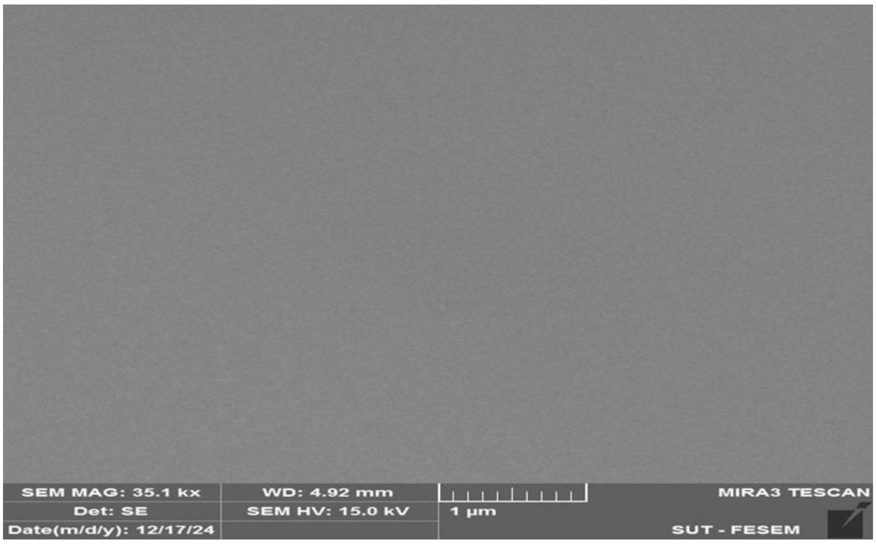

Figure 10. Scanning electron microscope images of Qayyarah asphalt modified with C9.

Scanning Electron Microscope Images of the asphalt of Qayyarah with C5:

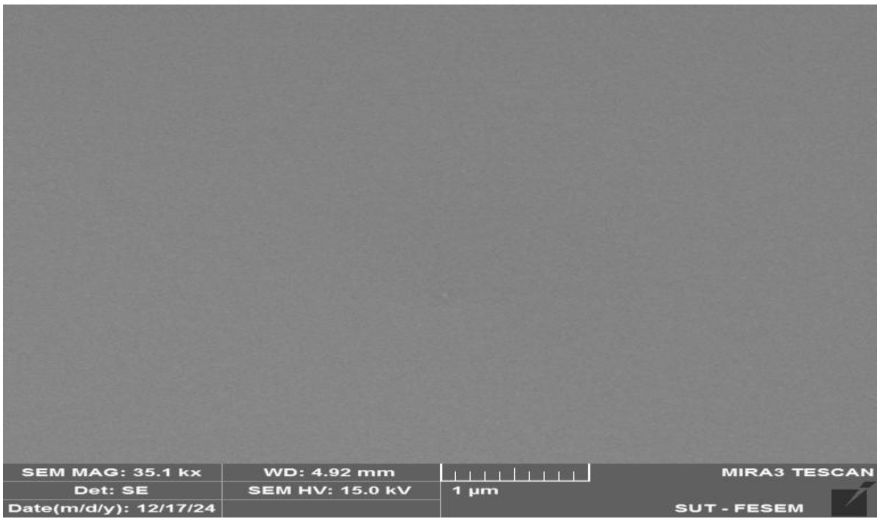

Figure 11. Scanning electron microscope images of Qayyarah asphalt modified with C5.

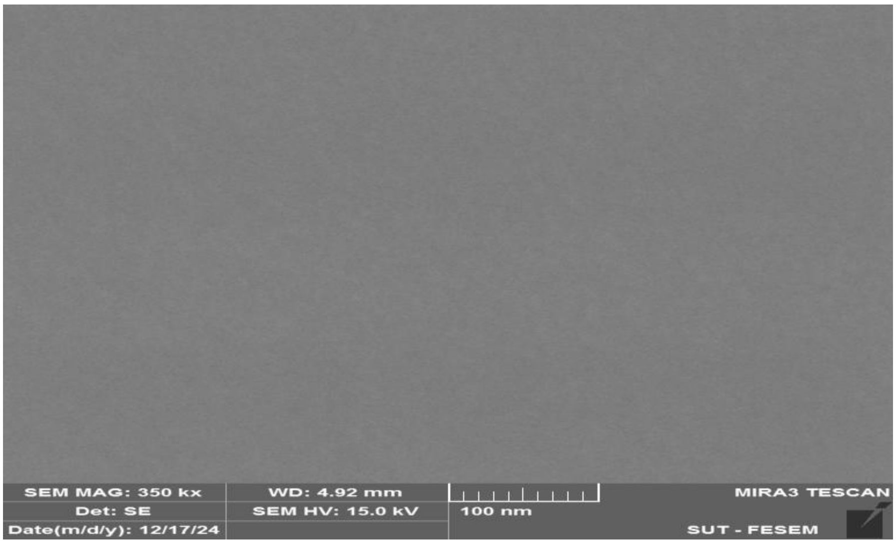

Figure 12. Scanning electron microscope images of Qayyarah asphalt modified with C5.

All the images in the shapes above indicate a high compatibility between both types of petroleum resin and the asphalt of Al Qayyarah, as there are no changes on the surface of the asphalt at the micrometer and nanometer levels. The surface topography is very homogeneous, as if it appears to be a single material.

Conclusion

Petroleum Resins significantly improved the performance and properties of Q asphalt, as observed from its physical characteristics. They had a significant impact on stability performance, as observed in the Marshall parameters. They resulted in a substantial increase in indirect tensile strength for both conditioned and unconditioned samples of the asphalt. The resistance of the asphalt to moisture and heat has become significantly high due to the petroleum resins.

1. It is recommended to use them to improve asphalt properties in temperate countries.
2. C9 is more effective in enhancing the performance of Qayyarah asphalt compared to C5.

Acknowledgments

To everyone who contributed to the completion of this work, to my esteemed professors, I offer my sincere thanks and appreciation.

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