

Article

Extraction and Characterisation of Maltene from Virgin Asphalt as a Potential Rejuvenating Agent

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Abstract: The wide application of reclaimed asphalt pavement (RAP) is hindered due to the highly brittle nature of the material, which contributes a major factor towards cracking-related distresses. While the utilisation of rejuvenating agents has been shown to enhance the flexibility of RAP, they also trigger certain negative effects on the performance of asphalt mixtures. In view of this, potential rejuvenators should be able to alter the rheological properties of asphalts to limit fatigue issues and enhance the potential of low-temperature cracking. Therefore, this study aimed to investigate the possibility of extraction and characterisation of maltene from virgin asphalt (VA) as a potential rejuvenating agent in RAP. Several physicochemical characteristics were examined, including density, viscosity, gas chromatography–mass spectrometry (GC–MS), Fourier-transform infrared (FTIR) spectroscopy, CHNS elemental analysis, and energy dispersive X-ray (EDX) analysis. Finally, the stiffness modulus characteristics of the different types of asphalt binders were evaluated at low and high temperatures. The results demonstrated that maltene was successfully extracted from VA using petroleum ether. In addition, the GC–MS showed that the extracted maltene contained polar and non-polar compounds with low molecular weights compared to VA. Furthermore, the spectra curve of maltene was very similar to that of asphalt, indicating its compatibility with asphalt binder and prospective use. Finally, adding maltene to aged asphalt decreased stiffness values to 0.0063, 0.0499, and 0.0108 MPa, which are equivalent to VA values (0.0061, 0.0481, and 0.0104 MPa) at loading times of 1.0, 0.1, and 0.55 s, respectively. Meanwhile, the stiffness modulus characteristics at low temperature were restored with the addition of maltene.

Keywords: maltene; asphaltene; rejuvenating agent; compatibility; solvent extraction; stiffness

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1. Introduction

The recycling of road asphalt materials serves an essential function towards achieving a sustainable road construction industry [1], particularly due to the regular maintenance and periodic rehabilitation/reconstruction of asphalt pavements via effective approaches under persistent traffic loads and harsh environments [2]. Consequently, a constant supply of asphalt binder and mineral aggregate, which both originate from non-

renewable resources, is required for the maintenance and rehabilitation/reconstruction process that, in turn, generates a huge amount of waste asphalt materials [2,3]. Thus, various waste materials, such as RAP, have been frequently used as a component in pavement mixtures [4,5]. Given that RAP materials comprise 4–6% of asphalt binder (by weight of aggregate), the use of 20–50% RAP content could lower construction expenses by 34% [6]. Apart from economic advantages, the utilisation of recycled asphalt materials minimises the overuse of raw materials, thereby reducing the consumption of energy and the emission of greenhouse gases (GHGs) through a sustainable disposal method [7]. Moreover, the addition of RAP in asphalt mixtures enhances the performance of asphalt pavements in terms of the rutting resistance due to improved stiffness of pavements.

On the contrary, the use of excessive or unbalanced RAP proportions leads to certain disadvantages [7]. This is due to the presence of organic materials in RAP-mixed asphalts, which becomes hard and brittle over time when exposed to water, oxygen and ozone in the air, bacteria, and dynamic loads from vehicles, as well as numerous factors during the maintenance process that worsen the pavement performance and even result in pavement failures, such as cracking, reducing the lifespan of asphalt pavements [8–10]. Specifically, certain factors, including solar and thermal radiation, and mechanical and chemical effects, lead to the degradation and establishment of new and existing physical and chemical bonds in the asphalt binder molecules [11]. In other words, the chemical and physical properties of asphalt can be affected by the change in chemical structure, composition, and binding form of the asphalt mixture [12]. For example, the ageing of asphalt causes physical alterations over time, such as phase homogeneity, relaxation ability, and stiffness. Hence, the ageing of asphalt binder is among the main factors that affect the durable properties of flexible pavement and ultimately its environmental footprint and cost of the life cycle [11].

Moreover, the gradual decrease of the aromatic portion in asphalt due to the ageing process corresponds to a higher proportion of asphaltene components in the asphalt mixture [13]. This phenomenon is linked to the structural changes in aged asphalts during their lifespan, where lightweight elements are converted to heavyweight elements, such as the following: Aromatic constituents → resin constituents → asphaltene constituents → carbenes/carboids [14]. With reference to the theory of regenerative component regulation [15], the constituents and characteristics of the asphalt mixture significantly determine the overall performance of the asphalt.

Currently, the highway agencies in many countries permit the use of RAP at a percentage range of 15–45% for the preparation of asphalt mixtures. The properties of the pavement depend greatly on the amount of RAP in the asphalt mixture [16]. In view of this, a low percentage of RAP is utilised to produce new asphalt pavements due to the presence of aged binder in RAP, which is stiff and inelastic. Therefore, a higher amount of RAP in asphalt mixtures would produce asphalt pavements that are extremely vulnerable to thermal, fatigue, and reflection cracking due to the highly brittle texture compared to the virgin asphalt (VA) binder [17]. Therefore, as the percentage of RAP binder is increased in the mixture, the percentage of aged binder utilised in the mix is also increased and may in turn reduce the performance characteristics (mainly fatigue and thermal cracking) of the resulting asphalt mixtures [18]. Thus, it is necessary to consider the negative effects of RAP in asphalt mixtures on the performance of asphalt pavements [19]. Numerous approaches have been employed to tackle the low durability of asphalt pavements with the addition of RAP. Although several researchers and engineers suggested the utilisation of a softer VA to restore the high durability of RAP that resembles that of VA, this method is ineffective when a more aged or higher RAP amount is used in the asphalt mixture [20]. To date, various research studies have considered the use of softening and rejuvenating agents to recover the essential properties of RAP, such as waste-derived, commercially viable, plant-based, and refinery-based oils [21]. Comparatively, softening or fluxing agents, such as flux oil, slurry oil, and lube stock, could reduce the viscosity of RAP binder [22,23], while rejuvenating agents could be used to retain the balanced

composition of aged asphalt due to the sufficient amount of maltene components that revive the internal structure of aged asphalt [23]. Over the last decade, rejuvenators have been used as the conventional material due to their rapid action in improving the viscosity and elasticity of aged binders [24] in asphalt mixtures, even under high RAP composition [25,26], by restoring the viscosity and elasticity of the aged binder.

Nevertheless, Zaumanis et al. [27] noted that the rejuvenating agents should be carefully selected in order to achieve the desired short- and long-term properties of asphalt pavement performance. An example of a short-term criterion is the rapid diffusion into RAP which triggers asphalt mobilisation [28], while one of the long-term criteria is the alteration of asphalt rheology to avoid fatigue and thermal cracking (to prevent any rutting or softening issues) [28,29].

Given the strong demand for rejuvenating agents and materials with similar molecular structures to petroleum-derived substances, such as waste refinery crude oil with asphalt, several studies have reported the integration of recycled asphalt with refinery crude oil, such as waste engine oil (WEO) [30,31]. In principle, recycled asphalt can be blended well with WEO, which serves as a rejuvenating agent to reduce the viscosity and soften the recycled asphalt [32]. Apart from enhancing the properties of recycled asphalts [33,34], the use of WEO reduces the excessive dependency on non-renewable and GHG-related materials, thus preserving already limited natural resources and lowering the rate of pollution [34,35]. On the contrary, the overuse of WEO could contribute to the binder softening effect that decreases the rutting resistance of the asphalt [36]. It was also reported that the excessive addition of WEO reduced the cohesive strength, particularly in high-temperature conditions [37]. The deteriorating properties of the asphalt at high temperatures are due to the absence of resin in WEO [38]. Another concern over the durability of rejuvenating agents is the presence of volatile compounds in certain softening agents that reduce the stiffness of asphalt to undergo compaction and further improve the asphalt [39]. The softening agents are unable to improve the mixture once the compounds become volatilised. Thus, rejuvenating agents should provide a long-term improvement in the characteristics of asphalt mixtures.

The chemical interaction between rejuvenating agents and asphalt is also a crucial parameter, since the rejuvenating agents accelerate the ageing process and make the asphalt obsolete [39]. The performance of various oils as rejuvenating agents has also been explored, such as used lubrication oils, flux oils, lube oil extracts, slurry oils, extender oils, waste vegetable oils, and bio-based oils. From a chemical perspective, used oils, such as binder additives, are considered complex mixtures due to the various unknown constituents [19]. However, the effect of exposure to the varying unknown compositions of used oils on the health of workers, the environment, and the general public has not been assessed [17]. Therefore, a comprehensive analysis of the environmental impact and sustainability of potential rejuvenators at higher temperatures and the modified RAP during the production and paving process should be performed prior to large-scale commercialisation.

Realising such constraints, several studies have recently evaluated the performance of maltenes as a rejuvenating agent due to their unique characteristics, such as suitable viscosity, and good blending and dissolution within the asphalt structure that ensures uniformity and compatibility compared to other rejuvenating agents [40,41]. Furthermore, maltene can be retrieved from asphaltene separation units in refinery systems, making it a practical rejuvenating candidate. Maltene is composed of saturates, aromatics, and resins [42]. These fractions present different properties, as they interact with each other with the presence of asphaltene, which dictates the complicated behaviour of asphalt. Functionally, resins promote anti-rutting ability, whereas aromatics offer flexibility to asphalt. Such traits promote maltene to serve as a rejuvenator to restore the attributes of aged asphalt. Hussein et al. [43] assessed the use of maltene-derived asphalt as a rejuvenating agent to restore the physical properties of very old asphalt. Their findings recorded the recovery of aged asphalt properties using 15% maltene. In addition, Al-Saffar et al. [40]

investigated the potential utilisation of maltene as a rejuvenating agent to improve the properties of RAP mixtures. Their results verified that up to 30% and 50% of the physical properties of recycled asphalt were restored using 8% and 16% maltene, respectively. In addition, the performance of recycled asphalt was similar to that of VA in low and high temperature conditions. Accordingly, the mechanical features of rejuvenated asphalt mixtures were enhanced compared to the VA mixture. In a separate study, Al-Saffar et al. [41] applied 12% maltene as a rejuvenator to determine the engineering properties of a 40% reclaimed hot mix asphalt (HMA) mixture. Based on the findings, maltene was found to efficiently reduce the effect of ageing of RAP asphalt and significantly improved the RAP–maltene blend compared to the VA and RAP mixture without maltene. Moreover, both moisture damage and cracking resistance of the rejuvenated RAP after long-term ageing (LTA) surpassed those of VA, implying the efficient use of maltene as a rejuvenator to restore the properties of aged asphalts.

Despite the various reports on the potential use of maltene, none of the studies has adequately characterised the properties of maltene. Based on these premises, the present study aims to perform a comprehensive characterisation of maltene as a promising rejuvenating agent in RAP. The testing programme included viscosity, density, GC–MS, FTIR, CHNS and EDX. Finally, to study the effect of maltene on the flexibility and stiffness properties of aged asphalt, the stiffness modulus characteristics of the different types of asphalt binders (virgin, aged, and rejuvenated asphalt by maltene) as well as the complex modulus and phase angle were evaluated.

2. Methodology

This section describes the techniques and standards used for the experimental work as shown in Figure 1.

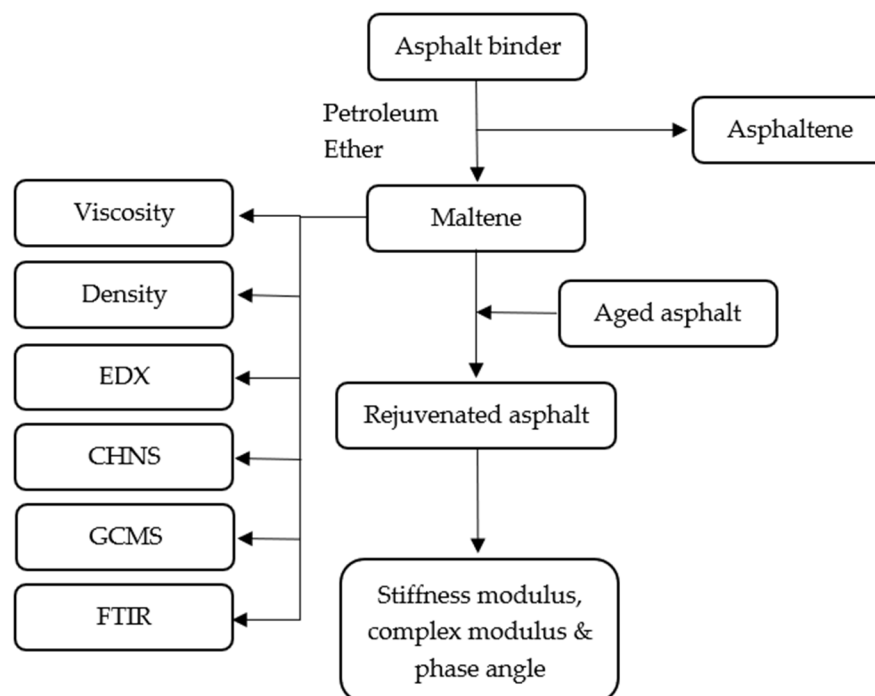


Figure 1. Flowchart of the experimental work.

2.1. Maltene Extraction Standard Methods

The conventional approaches for the physical fractionation of maltene are based on the physicochemical reactions, namely solvent extraction, distillation, and surface-active adsorption [44]. The extraction using non-polar solvents of similar nature but with different boiling points is similar to the fractional distillation method that produces a complex

mix of fractions. This is achieved through the use of a wide range of boiling points and molecular weights [45]. The total yield of extracted fractions depends on several parameters, including the temperature, nature of the solvent extraction, dilution range, and the equilibrium of the system [44].

Maltene can be extracted from petroleum asphalt using non-polar solvents with a low surface tension of less than 25 dyne cm^{-1} at 25 °C, such as low molecular weight paraffinic hydrocarbons, including low boiling point petroleum naphtha, *n*-heptane, *n*-pentane, petroleum ether, isopentane, and liquefied petroleum gases [14]. The various techniques that have been used for the separation of asphaltene from maltene following the ASTM standards are presented in Table 1.

Table 1. Different methods for asphaltene and maltene separation [14,46].

Method	Precipitant	Volume Precipitant per Gram of Sample
ASTM D893	<i>n</i> -Pentane	10 mL
ASTM D2007	<i>n</i> -Pentane	10 mL
ASTM D3279	<i>n</i> -Heptane	100 mL
ASTM D4124	<i>n</i> -Heptane	100 mL
ASTM D6560	<i>n</i> -Heptane	30 mL

It should be noted that various hydrocarbons (*n*-pentane or *n*-heptane) yield varied asphaltene fractions. Inaccurate yield is gained when a solvent is not compensated by extra hydrocarbon [46]. However, excessive hydrocarbon causes varying asphaltene fraction yields that are inaccurate [46]. In determining the accurate content of asphaltene and maltene, crude oil (e.g., asphalt) should be blended with excess (>30 volumes hydrocarbon per volume of sample) low boiling point hydrocarbons (e.g., *n*-heptane or *n*-pentane) [46]. Solvent (e.g., toluene) may be used prior to incorporating low boiling point hydrocarbon for an extremely highly viscous sample. In order to compensate for the solvent, extra hydrocarbon (>30 volumes of hydrocarbon per volume of solvent) should be included [46]. After some time, the filtration process separates the insoluble material (asphaltene fraction), and it is later left to dry.

In the present study, petroleum ether was selected as a solvent to separate asphaltene from maltene. Petroleum ether exhibits a low density, a boiling point range of 60–80 °C, and contains light compounds with aliphatic hydrocarbons. Hence, a given amount of petroleum ether was used to extract maltene from asphaltene. On the contrary, *n*-heptane (as an example) exhibits a higher density compared to petroleum ether and has a boiling point of over 98 °C, making it difficult to use as a solvent for extraction of maltene from VA

2.1.1. Determination of the Asphalt to Petroleum Ether Ratio

Five concentrations of petroleum ether (X1, X2, X3, X4, and X5 mL) were added to 1 g of asphalt to identify the optimum petroleum ether dose that should be added to the asphalt (petroleum ether: asphalt) in order to separate asphaltene from maltene. The maltene content increased and the asphaltene content decreased with the increment in solvent content. The dose at which the percentages of maltene and asphaltene are stable without any change in the weight reflects the optimum dose of petroleum ether. The results were compared with ASTM D4124 [47], which assumed mixing 100 mL of *n*-heptane as solvent to 1 g of asphalt, to ensure that the process was successfully performed.

2.1.2. Extraction and Recovery of Maltene

In this study, VA was acquired from Kemaman Bitumen Company (KBC), Malaysia for the extraction of maltene. The solvent extraction method was performed based on that described by Al-Saffar et al. [41], which consists of several stages, beginning with the

mixing of VA with an optimum dose of petroleum ether in a container, as shown in Figure 2. The mixture was then transferred into a water bath at 50 °C for 2 h with continuous stirring until the soluble fraction was completely dissolved. The mixture was left in the water bath for another 60 min to allow the asphaltene to separate from the dissolved VA and settle to the bottom of the container and subsequently filtered from the mixture using filter paper. The maltene was recovered from the petroleum ether using a rotary evaporator at 50–70 °C. Depending on the extraction conditions, the ability to recycle the petroleum ether for repeated extraction makes this approach more economical. Eventually, the extracted maltene was heated in an oven at 80 °C (which corresponds to the boiling point of petroleum ether) for a maximum of 30 min to completely remove any presence of chemical solvents. Finally, the maltene was weighed several times to ensure that the material was devoid of petroleum ether. The value obtained from this procedure was also compared with ASTM D4124 [47] standard to prove that this process was successful.

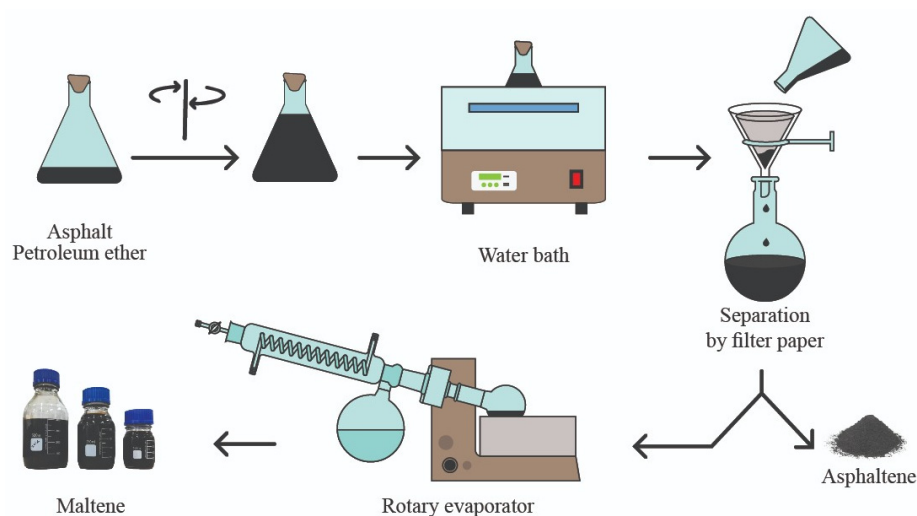


Figure 2. The procedure for extracting maltene [41].

2.2. Characterisation of Maltene

The following tests were performed on the maltene-derived asphalt to characterise the material:

2.2.1. Viscosity

A Brookfield Rheometer V3.3 (Model LV, Spindle CP41) was used to measure maltene's resistance to flow according to ASTM D7042 [48], as well as calculating both shear rate and shear stress of the material. The test was conducted at 95 °C by driving a spindle (immersed in a maltene sample) through a calibrated spring. The spring deflection measured the viscous drag of the maltene against the spindle. Spring deflection was measured with a rotary transducer. The measuring range (in centipoise) was based on the rotational speed of the spindle, the size and shape of both the spindle and the container the spindle rotated in, as well as the full-scale torque of the calibrated spring.

2.2.2. Density

A density meter (DMA 4100 M model) was used to determine the density of maltene at 20 °C, according to ASTM D7042 [48]. The procedure was performed by injecting the material inside the U-tube of the instrument. The U-View camera feature displayed live images of the oscillating U-tube sensor to ascertain that the entire filled-in sample was captured. Next, "Filling Check" was used to monitor the filling defects, such as gas bubbles and particles in the sample. The result was obtained automatically after 30 s.

2.2.3. Elemental Analysis Using Energy Dispersive X-ray (EDX)

The chemical elements of the maltene were measured using EDX, which was connected to a field emission scanning electron microscope (FESEM) (Hitachi SU8020 model). The process involved exposure of the sample surface to an electron beam, which triggered a collision between the bombarding electrons and the electrons of the sample atoms. As a result, a few of the electron sample atoms were eliminated. External shell electrons then filled the gap left by the eliminated electrons from the internal shell with higher energy. The external electrons were supposed to emit X-rays and relinquish a portion of their energy for this replacement. Upon transfer, X-rays were released by the atom of each element with singular energy quantities. Hence, the same atom responsible for the X-ray emission was determined by quantifying the energy stored during exposure to the electron beam in the sample-emitted X-rays.

2.2.4. Elemental Analysis Using Elemental Analyser

Elemental analysis was conducted using a CHNS elemental analyser to provide the details of maltene chemical composition. The elemental analysis gives information about the percentages of the material's carbon, hydrogen, nitrogen, and sulphur (CHNS) elements. Typically, carbon is the principal element present in asphalt, followed by hydrogen and sulphur, whereas nitrogen is commonly found in small amounts.

2.2.5. Gas Chromatography–Mass Spectrometry (GC–MS)

American Agilent (6890N) equipment that combined gas chromatography (GC) and mass spectrometry (MS) was employed to analyse the chemical composition of maltene quantitatively. This method has the advantages of fast analysis speed, high accuracy, and high sensitivity. The technique allows the isolation, quantification, and differentiation of complex chemical compounds associated with a low detection limit. As for the GC analysis, the original sample was maintained for 2 min at a temperature of 50 °C. Afterwards, the temperature was elevated to 325 °C. The running time was 59 min. The selected carrier gas was helium, with a 1.0 mL/min flow rate. The nominal initial pressure was 8.8 psi, while the average velocity was 37 cm/s. Meanwhile, the MS analysis was carried out with an ion source, in the following conditions: solvent delay of 2.00 min, gain factor EMV Mode, resulting EM voltage of 1023.5 V, and mass scanning rate of 50–550 *m/z*.

2.2.6. Fourier-Transform Infrared (FTIR)

FTIR is a convenient analytical method to detect the presence of functional groups in asphaltic materials [49]. In this study, an IRTracer-100 instrument was employed to determine the presence of functional groups in maltene at a resolution of 0.25 cm^{-1} and rapid scanning of 20 spectra/s. After the sample was placed in an infrared IR beam, IR radiation was emitted and passed through the interferometer for spectral encoding. Subsequently, the beam penetrated the sample, where the sample absorbed some IR, while the others were transmitted. Finally, the beam reached the detector for the final measurement. The intensity and frequency of light absorbed, which represents the absorption of various bonds, were used to identify the functional groups in the sample. The peak height was also determined from the captured chemical functional group in the form of absorbance and the band area in the FTIR spectrum.

2.2.7. Aged Binder's Properties Restoration

The possibility of aged asphalt's rejuvenation using maltene-derived asphalt was evaluated according to the stiffness modulus characteristics using the nomographs for asphalt characteristics developed by Van der Poel [50]. In this study, the stiffness of the virgin asphalt, aged asphalt and rejuvenated asphalt (maltene + aged asphalt) were calculated at low and high temperatures over a wide range of loading conditions, according to the penetration, softening point and penetration index (PI) of the asphalt binders. The

high temperature represents the mean of the hottest seven-day period in the last five years in Mosul, Iraq, while the low temperature represents the average of the minimum one-day air temperature for each year. Furthermore, the complex modulus and phase angle of asphalt binder were also determined according to the mean of the hottest seven-day period in the last five years in Mosul, Iraq, using BitProps V2 software developed by G. Rowe and M. Sharrock.

3. Results and Discussion

3.1. Determining the Asphalt to Petroleum Ether Ratio

During the separation process of asphaltene from maltene, it was essential to select the most suitable percentage of petroleum ether that should be added to the asphalt. Figure 3A shows that adding X1 mL petroleum ether to 1 g asphalt failed to separate asphaltene from maltene, primarily because X1 mL of petroleum ether did not dissolve the asphalt. Nonetheless, the addition of X2 mL petroleum ether to 1 g asphalt resulted in partial success. In particular, specific percentages of maltene were dissolved by solvent and passed through the filter, while some other stuck to the filter paper, as shown in Figure 3B. Conversely, the separation process was successful after adding X3 mL of petroleum ether. The maltene wholly dissolved into the solvent, and the asphaltene was filtered entirely out of the mixture (see Figure 3C). Figure 4 shows that adding X2 mL of petroleum ether to 1 g asphalt gave 60.21% of maltene and 39.79% of asphaltene. At the same time, adding X3 mL of petroleum ether to 1 g of asphalt yielded 80.83% of maltene and 19.17% of asphaltene. Adding X4 and X5 mL of petroleum ether approximately attained the same values retrieved for X3 mL of petroleum ether, which means the optimum dose of petroleum ether that should be added to extract maltene was X3 mL petroleum ether to 1 g asphalt. The value obtained from this procedure was compared with ASTM D4124 [47] standard, thus verifying that this process was highly successful.

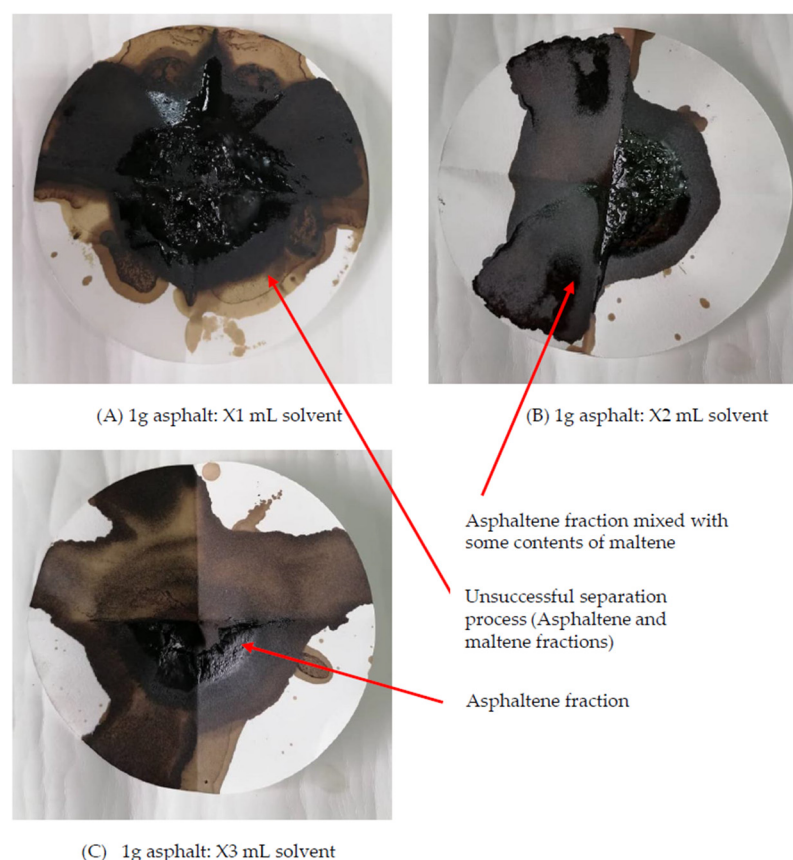


Figure 3. Separation of asphaltene from maltene using different volumetric percentages.

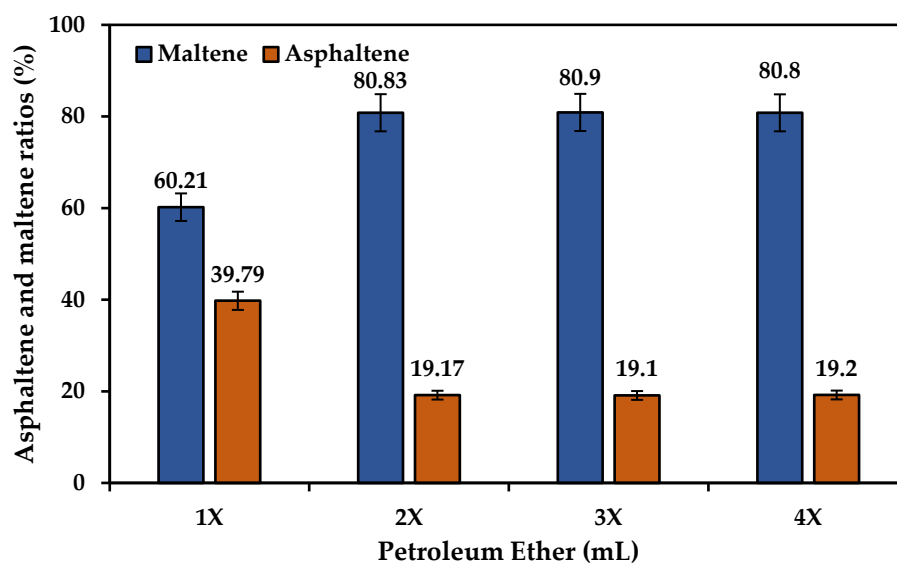


Figure 4. The relationship between the solvent content and asphalt fraction.

3.2. Viscosity and Density of Maltene

Table 2 lists four readings of the viscosity values obtained from the Brookfield Rheometer V3.3 of the maltene sample at 95 °C. In addition, the density meter apparatus indicated that maltene density was 0.955 g/cm³ at 20 °C.

Table 2. Results of viscosity test for maltene sample.

Number	Viscosity (mPa.s)	Torque (%)	Shear Stress (N/m ²)
1	59.39	25.8	0.59
2	61.00	31.8	0.73
3	52.62	32.0	0.74
4	47.48	33.0	0.76

3.3. Chemical Compounds Analysis

Outcomes retrieved from the GC–MS test showed that maltene had over 70 peaks, indicating its complex material. Table 3, however, only lists 13 components that exceed 1.0% content in the maltene. The rest of the components were regarded as minor components and were therefore not listed. If a substance is present in high amounts it will have a high peak area, so the more the functional groups in the material, the higher the peak area [51]. As mentioned above, maltene is made up of numerous chemical compounds, with molecular weights of more than 200 g/mol. Therefore, maltene is made up of heavy molecular weight compounds. According to Table 4, the molecular weight of the compounds ranged from 192.25–342.49 g/mol, and almost all the molecules were C₆–C₁₇ carbons with a maximum peak area of C₁₂. Nevertheless, the weight of these compounds is much lower than the 700 g/mol average molecular weight of petroleum–asphalt [52]. This implies that maltene can be used to reduce the viscosity of aged asphalt. Determining the boiling point of maltene compounds is essential to estimate the evaporation of the volatiles in the rejuvenated asphalt while producing the asphalt mixtures via a mixing and compaction processes. Hydrocarbon compounds with C₆–C₁₂ had boiling points of 184–309.6 °C. In comparison, HMA has a lower production temperature. The same is true for hydrocarbon with C₁₄–C₁₇, which has boiling points ranging from 195–300.5 °C. Hence, the boiling point of maltene compounds is higher than the mixing and compaction temperature of the asphalt mixture. Furthermore, crude oil refineries produce petroleum asphalt as residue. The refinery works at a very high temperature of 400 °C to distil oil [53]. As

can be seen in Table 3, this distillation temperature is higher than all the maltene compounds. Therefore, petroleum asphalt has shallow mass losses during evaporation [53].

Table 3 also shows the formulas of the maltene compounds. The results show both non-polar and polar ends groups in the maltene molecules. Therefore, maltene is both hydrophobic (material that can repel water molecules) and hydrophilic (material that can absorb the water) [54]. The first most dominant component in maltene was 1,4-Bis(trimethylsilyl)benzene ($C_{12}H_{22}Si_2$), which is non-polar. The presence of high percentages of non-polar groups in maltene means that this material can be used to decrease the polarity and restore the properties of aged asphalt. The second component was Methyltris-trimethylsiloxy-silane ($C_{10}H_{30}O_3Si_4$), which is a polar end group with methyl (CH_3) components. It was followed by silane, 1,4-phenylenebis[trimethyl- ($C_{14}H_{26}Si_2$), which is also considered a non-polar group, followed by a polar group, which is benzene, 2-[(tert-butyl dimethylsilyl) oxy]s-1-isopropyl-4-methyl- ($C_{16}H_{28}OSi$). Added to the above, maltene also has a ketone polar group, i.e., acetamide, N-[4-(trimethylsilyl) phenyl ($C_{11}H_{17}NOSi$), with a $C=O$ bond. In addition, another polar group was identified, namely the ($C_{16}H_{28}OSi$) compound. The presence of silicon groups linked to carbon atoms increases the adhesion work and reduces the susceptibility of asphalt to water and various weather conditions, which results in improved stripping resistance (i.e., better adhesion between asphalt and aggregate) [55].

Table 3. Chemical compounds of maltene.

N	Compound	Area (%)	Formula	Molecular Weight (g/mol) [56]	Boiling Point (°C) [56,57]
1	1,4-Bis(trimethylsilyl)benzene	43.08	$C_{12}H_{22}Si_2$	222.48	194.0
2	Methyltris(trimethylsiloxy)silane	8.67	$C_{10}H_{30}O_3Si_4$	310.68	212.3
3	Silane, 1,4-phenylenebis [trimethyl-	5.12	$C_{14}H_{26}Si_2$	222.47	195.0
4	Acetamide, N-[4-(trimethylsilyl)phenyl-	4.22	$C_{11}H_{17}NOSi$	207.34	221.0
5	Benzene, 2-[(tert-butyl dimethylsilyl) oxy-1-isopropyl-4-methyl-	3.46	$C_{16}H_{28}OSi$	264.48	300.5
6	Cyclotrisiloxane, hexamethyl-	3.38	$C_6H_{18}O_3Si_3$	222.46	184.0
7	Arsenous acid, tris(trimethylsilyl) ester	3.15	$C_9H_{27}AsO_3Si_3$	342.49	244.2
8	Silicic acid, diethyl bis(trimethylsilyl) ester	3.07	$C_{10}H_{28}O_4Si_3$	296.58	231.0
9	2,4,6-Cycloheptatrien-1-one, 3,5-bis-trimethylsilyl-	1.80	$C_{13}H_{22}OSi_2$	250.48	214.8
10	benzenamine, 4-[2-(4-methoxyphenyl) ethenc-N,N-dimethyl-	1.56	$C_{17}H_{19}NO$	253.34	N/A
11	1,2-Bis(trimethylsilyl)benzene	1.41	$C_{12}H_{22}Si_2$	222.47	249.9
12	4-(4-Hydroxyphenyl)-4-methyl-2-pentanone	1.19	$C_{12}H_{16}O_2$	192.25	309.6
13	4,5-Dibromo-6-chloro-2-benzoxazolinone	0.96	$C_7H_2Br_2ClNO_2$	327.36	N/A

3.4. Elemental Analysis

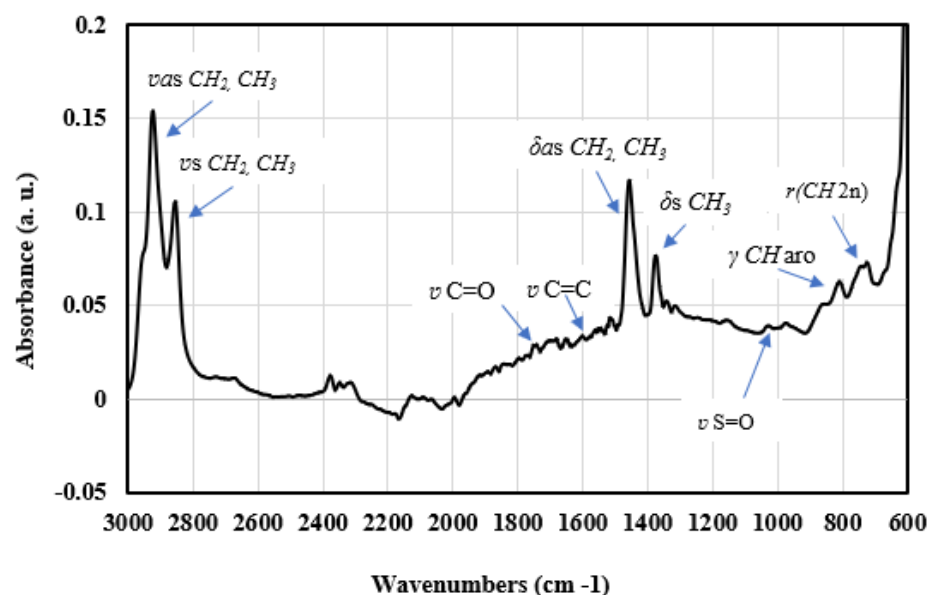
EDX is a useful tool used to determine the elements contained in the asphalt binder [58]. Table 4 illustrates the chemical elements of maltene, as observed based on EDX using FESEM. The percentages of the chemical elements were detected on the surface of the maltene only. As a result, the sample recorded high content of the carbon (C) element at 93.6%, followed by sulphur (S) (5.5%) and oxygen (O) (0.8%). The EDX, nonetheless, could not detect other elements, such as hydrogen and nitrogen. Therefore, the elemental composition of maltene was also analysed using the CHNS analyser. The results are tabulated in Table 4. It can be seen that the C element recorded the highest value (82.8%), followed by H (11.61%), N (0.48%), and S (5.1%). Notably, the C value examined using EDX was higher than the value retrieved from the CHNS analyser, mainly because EDX only detected elements present on the material surface [59]. Hence, it differed from the elements' percentages from the elemental analysis.

Table 4. CHNS and EDX analysis of maltene.

Test	C (%)	H (%)	N (%)	S (%)	O (%)
EDX	93.6	-	-	5.5	0.8
CHNS	82.80	11.61	0.48	5.10	-

3.5. Fourier-Transform Infrared (FTIR)

Figure 5 presents the FTIR spectra of maltene. The different bonds or functional groups in the form of wavenumber correspond to the intensity of spectral peaks linked to maltene. For instance, the bending vibration at 722 cm^{-1} signifies the long-chain methylene $-(\text{CH}_2)_n-$ of maltene, while the small bending frequencies at approximately 742 cm^{-1} and 807.6 cm^{-1} are assigned to the presence of the C-C functional group in maltene. In addition, the very small absorption peak at 1030 cm^{-1} reflects the presence of the sulfoxide group. These findings correlate with the CHNS analysis and EDX results, which detected the presence of S in the chemical structure of maltene. Furthermore, the adsorption peaks at 1371.97 cm^{-1} , 1454.63 cm^{-1} , and 1700 cm^{-1} correspond to the symmetric deformation vibration of CH_3 asymmetric vibration of CH_2 , CH_3 , and the carboxylic group, respectively. This suggests that maltene could be utilised to reduce the oxygenated group in aged asphalts that contain extremely high amounts of $\text{S}=\text{O}$ and $\text{C}=\text{O}$. Meanwhile, the 2851 cm^{-1} and 2919.7 cm^{-1} absorption bands indicate the C-H stretching vibration. Based on the results, it can be perceived that the maltene spectra curve is very near to the normal asphalt binder. This undoubtedly indicates the potential for good compatibility of maltene in the binders [40].

**Figure 5.** FTIR spectra of maltene.

3.6. Stiffness at High and Low Temperatures

Table 5 shows that maltene diminished the ageing effect of aged asphalt by decreasing the stiffness to a level similar to that of virgin asphalt. This change means that the penetration increased as the amount of maltene was raised, while the softening point gradually decreased, and was restored to an almost similar level to that of the virgin asphalt. The reason is associated with the asphaltene content in the binders, where its content in the aged asphalt decreases with the addition of maltene. More specifically, it can be seen that the stiffness values of aged asphalt were 0.00193, 0.1339, and 0.3207 MPa at 1.0, 0.1, and 0.55 s loading time at a high temperature, respectively. However, adding maltene led to a decrease in the values to 0.0063, 0.0499, and 0.0108 MPa, which are

approximately similar to that of virgin asphalt values (0.0061, 0.0481, and 0.0104 MPa). The same changes happened at a low temperature, where the stiffness values of aged asphalt were 74.68, 169.3, and 93.01 MPa, and restored to 72.37, 174.9, and 92.48 MPa with the addition of maltene. Similarly, Zhang et al. [60] and Zhang et al. [61] asserted that increased stiffness of asphalt can deteriorate low temperature cracking resistance, while the addition of rejuvenator can improve the low temperature cracking resistance of aged asphalt.

Table 5. Stiffness modulus of asphalt binders.

Type of Asphalt	Loading Time: 1 s	
	Stiffness at High Temperature (MPa)	Stiffness at Low Temperature (MPa)
Virgin asphalt	0.0061	72.12
Aged asphalt	0.0193	74.68
Rejuvenated asphalt	0.0063	72.37
Type of asphalt	Loading time: 0.1 s	
	Stiffness at high temperature	Stiffness at low temperature
Virgin asphalt	0.0481	175.5
Aged asphalt	0.1339	169.3
Rejuvenated asphalt	0.0499	174.9
Type of asphalt	Loading time: 0.55 s	
	Stiffness at high temperature	Stiffness at low temperature
Virgin asphalt	0.0104	92.43
Aged asphalt	0.3207	93.01
Rejuvenated asphalt	0.0108	92.48

The complex modulus and phase angles of asphalt binder samples at 0.1 Hz, 1 Hz, and 10 Hz are shown in Figures 6 and 7. The complex modulus of asphalt represents its resistance to deformation under shear stress, while the phase angle represents the viscous response of the asphalt to shear deformation [62]. From the figures, it can be seen that the complex modulus values of the aged asphalt were higher than the values of virgin and rejuvenated asphalts. This means that the aging process made the asphalt binder become stiffer, to a large extent. Meanwhile, the addition of maltene rejuvenator softened the aged asphalt for all frequencies. Thus, the values of virgin and rejuvenated asphalt binders appear relatively close to each other in the table, indicating an improvement in rheological properties after rejuvenation.

In contrast, the aged asphalt exhibited the lowest phase angle values at the different frequencies, compared with virgin and rejuvenated asphalt binders. A lower phase angle asphalt binder typically has higher elasticity and performs better in recovering shear deformation. Meanwhile, the asphalt binder experienced a substantial increase in the phase angle after including maltene. This indicated that the addition of maltene improved the viscous components and rejuvenated the viscoelastic properties of the aged asphalt binders to be close to the original level. The conclusion is consistent with Cavalli et al. [63] who reported that aged asphalt with rejuvenating agent exhibited lower rutting resistance relative to aged asphalt without rejuvenating agent.

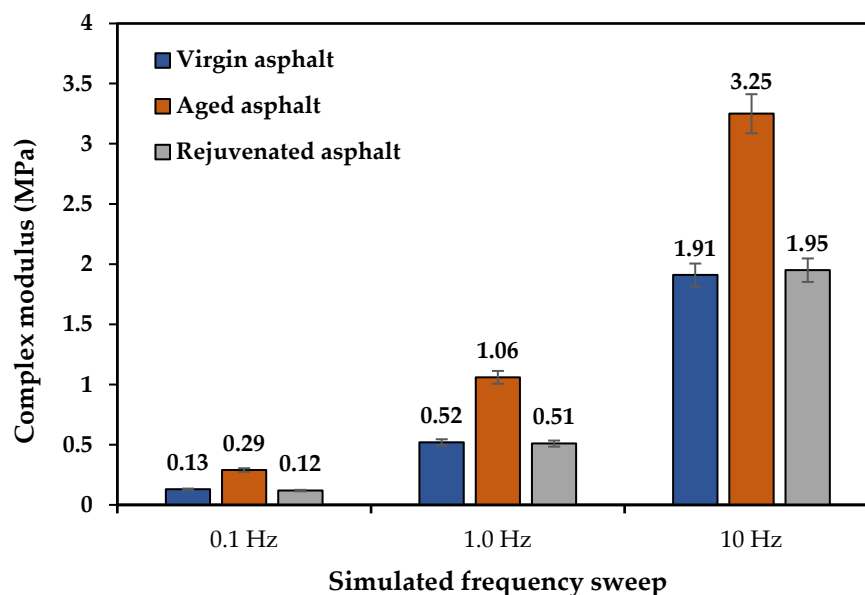


Figure 6. Complex modulus for different asphalt samples.

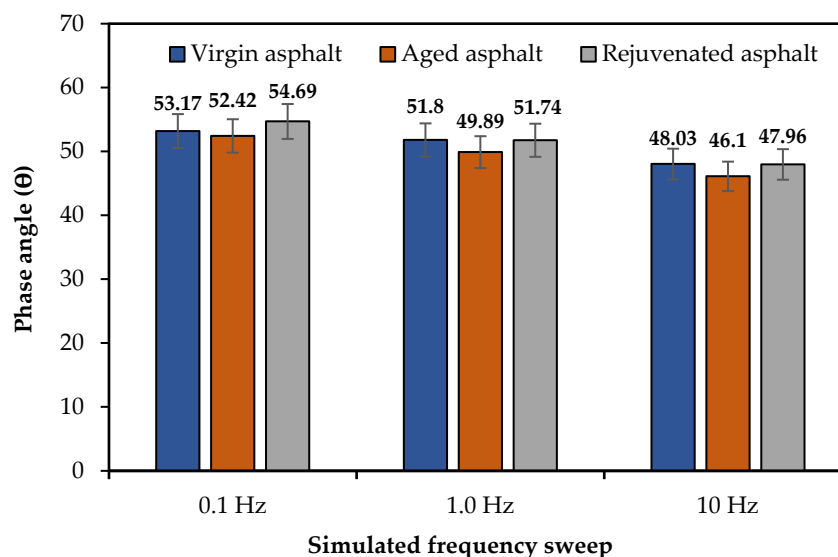


Figure 7. Phase angle for different asphalt samples.

4. Conclusions

This present study aimed to examine the feasibility of extraction and characterisation of maltene to be used as a rejuvenating agent. The significant outcomes obtained from this study are summarised below:

Maltene was successfully separated from VA using petroleum ether. The GC–MS showed that the maltene contained polar and non-polar compounds, and the molecular weights of these compounds were much lower than that of asphalt binder. The maltene's spectra curve was very similar to that of asphalt, which indicated the potential of good compatibility between maltene and asphalt. Meanwhile, the addition of maltene to aged asphalt caused an increase in the aromatics and saturates content and a consequential decrease in the stiffness modulus. Moreover, the stiffness modulus of rejuvenated asphalt at high and low temperatures was restored with the addition of maltene, signifying an improvement in features of aged asphalt after rejuvenation. Meanwhile, the addition of maltene enhanced the viscous components, and rejuvenated the viscoelastic characteristics of

the aged asphalt binders to be close to the original level. These outcomes were verified using the complex modulus and phase angle measurements.

Based on the laboratory tests, several recommendations are suggested for extensive research work in the future:

- a. Extraction and characterisation of maltene from more than one resource of asphalt is suggested.
- b. Research on the effect of short- and long-term ageing on the physical, rheological and microstructure characteristics of aged asphalt incorporating maltene is recommended.
- c. Future research should examine the surface-free energy (SFE), the binder bond strength (BBS) and compression pull-off test (CPOT) tests before and after ageing to understand the bonding and interactions between the aggregate and rejuvenated asphalt.

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