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Evaluation of ageing impact on rheological properties and surface free energy of asphalt binder

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Abstract
The empirical test methods of asphalt cement like softening point and penetration can only give limited insight into the property of the binder, advanced test methods are used in the present investigation to characterize the effects of ageing on the rheological and physical properties of asphalt cement. In this study, a specific combination of physical, rheological, and surface free energy testing has been implemented to categorize the influence of ageing on binder properties. Asphalt cement obtained from Doura oil refinery was subjected to ageing with the aid of Thin Film Oven Test (TFOT) apparatus, the binder experienced a temperature of 163 °C for 5 hours. All the testing was conducted on samples before and after ageing. It was observed that surface free energy decreased by (76 and 7.8) % after practicing ageing when tested by Sessile Drop and Wilhelmy plate methods respectively. However, the stiffness modulus increases by 26.6 % after ageing. The penetration and ductility decreased by (13 and 12) % respectively while the softening point increased by 11 % after ageing. On the other hand, the penetration index (PI) and the penetration viscosity number (PVN) have improved by (74.6 and 10.8) % respectively after ageing. It was recommended that asphalt cement should practice the physical, rheological, and surface free energy testing before a decision can be made of its suitability to be used for pavement under a specified environmental condition.

Keywords: Asphalt Cement, Surface Free Energy, Rheology, Physical Properties, Ageing

1. Introduction
Ageing of asphalt binder significantly affects the performance of asphalt pavements and is believed to be one of the main reasons for substantial pavements failures, such as loss of bonding, cracking or fatigue as reported by Li *et al.*, [1]. However, in special cases, ageing can also be the positive reason for an improved ability to resist distress, like permanent deformation as addressed by Little *et al.*, [2] because of the increase in stiffness. Therefore, many authors, Sarsam, [3] and Alisov *et al.*,[4] consider better knowledge on ageing behavior and ageing susceptibility of asphalt binders of utmost importance for designing more durable asphalt pavements. Zeng *et al.*, [5] stated that asphalt binder being an organic polymer, it is susceptible to aging during long-term exposure to oxygen, UV radiation and vehicle loading. Aged bitumen has a higher stiffness, which is beneficial to rutting resistance, but makes the binder prone to fatigue and low temperature cracking. Yang *et al.*,[6] explored the aging mechanism of bitumen and stated that the aging of bitumen comes from three aspects: Loss of volatiles, dehydrogenation, and oxidation. It was concluded that due to chemical variation, aged bitumen has higher modulus and lower phase angle, showing a solid-like rheological behavior, which is observed by increasing viscosity and brittleness as well as decreasing flexibility. Wang *et al.*,[7] reported that aging significantly increased the spatial variations in the sample properties and appeared to increase the sample's adhesive and/or cohesive strength. It was concluded that asphalt binder ageing leads to significant changes in mechanical and physicochemical properties and often premature cracking in asphalt pavements. Consecutive ageing by means of the Rotating Thin Film Oven Test RTFOT leads to a significant change in Dynamic Shear Rheometer (DSR) parameters. It was found by Koyun *et al.*, [8] that after ageing, equivalent modulus temperature increases, and the corresponding phase angle decreases. This means that ageing increases stiffness and elasticity. The test results indicate that rheological and binder properties follow an approximately linear trend for increasing ageing condition of the material. Interestingly, the highest impact of oxidation happens during the first ageing step, whereas all subsequent ageing conditions have approximately similar but reduced impact. The ageing phenomena is as a combined result of oxidation Soenen *et al.*, [9], UV radiation Aguiar-Moya *et al.*, [10] and loss of volatile molecules and physical hardening Eberhardsteiner *et al.*, [11]; and Leite *et al.*, [12]. Principally, it can be subdivided into short-term ageing during asphalt mixture production, and long-term ageing during the in-service life of the pavement. In both cases, the chemical asphalt binder composition changes, leading to an increased polarity. Taris *et al.*, [13] compared the effects of different laboratory ageing methods on the chemistry and rheology of bitumen types, (Pen 40/60, and Pen 70/100).

Four ageing protocols were applied: ageing at room temperature, oven ageing, pressure ageing vessel (PAV), and rolling thin-film oven test (RTFOT) combined with PAV ageing. The effects of temperature, pressure, and ageing time were studied using dynamic shear tests. The results highlight the relationship between chemistry and rheology of bitumen. Bitumen hardening, which was revealed by an increase in complex modulus and a decrease in phase angle, was reflected in the growth of specific chemical functional groups. Among all materials, soft bitumen showed the greater tendency to oxidize. Molenaar *et al*.,[14] studied the effects of well-known short-term and long-term laboratory aging procedures on the rheological characteristics and chemical composition of binders. Test results are compared with the rheological characteristics and chemical composition of field aged binders. The results obtained on field aged binders are also compared with the results of laboratory aging protocols in which a special weatherometer was used. The results clearly show that none of the laboratory procedures is capable of simulating long-term field aging. At best 2 years field aging could be simulated. Fatigue testing showed that aging initially increases the fatigue resistance of the binder. However long-term aging occurring in the field after a period of more than 7 years results in a significant decrease of the fatigue resistance.

The aim of the present investigation is to assess the influence of ageing by implementing the (TFOT) apparatus of asphalt binder on the physical, rheological, and surface free energy properties.

2. Materials and Methods

2.1. Asphalt cement

Asphalt cement with a penetration grade of (40-50) was obtained from Doura oil refinery and implemented in the present investigation. Table 1 exhibit the physical properties of the binder.

Table 1: Physical Properties of Asphalt Binder

Property	Test conditions	ASTM, [15] Designation	Value	SCRB, [16] Specification
Penetration	25˚c, 100gm, 5 sec	D5-06	40	40-50
Softening point	Ring &Ball	D36-95	54	-
Ductility	25˚c,5cm/min	D113-99	167	>100
Specific Gravity	25˚c	D 70 – 03	1.04	-
After Thin Film Oven Test (ASTM D-1754, [15])				
Retained Penetration of Residue (%)	25˚c, 100gm, 5 sec	D5-06	87	>55%
Ductility of Residue	25˚c,5cm/min	D113-99	74	>25
Loss on Weight %	163˚c,50gm,5hr		0.32	------

2.2. Thin film oven test

The thin film oven test as per ASTM D 1754,[15] is implemented to evaluate the changes in physical properties of asphalt cement when its exposure to short-term aging process (heat and air) during conventional hot mixing in asphalt plants, The penetration, softening point, ductility, viscosity and loss in weight after thin film oven test were determined. A set of 50-gm of binder samples resulting in a thin film thickness of 3.2 mm of asphalt cement was placed in a flat container with (140 mm in diameter). The containers were placed on the rotating shelf oven for 5 h at 163 °C.

2.3. Surface Free Energy Components by Sessile Drop Method and Wilhelmy plate method

The surface free energy of asphalt binder is defined as an amount of work required to create unit area of the material in vacuum. It is related to moisture sensitivity and adhesive fracture properties of the binder. The three components of which the surface free energy is composed for asphalt binder are the Lifshitz-van der Waals component, the Lewis base component, and the Lewis acid component as addressed by Little and Bhasin, [17]. The contact angle and the surface free energy are implemented to compute work of adhesion between aggregate and asphalt binder.

2.4. Sessile Drop Method

A probe liquid is dispensed over a smooth horizontal surface coated with asphalt binder. The image of the drop of liquid formed over the surface of the binder is captured by using a digital camera. Contact angles are obtained by analyzing the image using image processing software (Comef 4.3) software. A static Contact angles measured with different probe liquids are used with equations of work of adhesion to determine the three surface energy components of the asphalt binder as recommended by Lytton *et al*., [18]. Figure 1 exhibit the contact angle according to Sessile drop method.

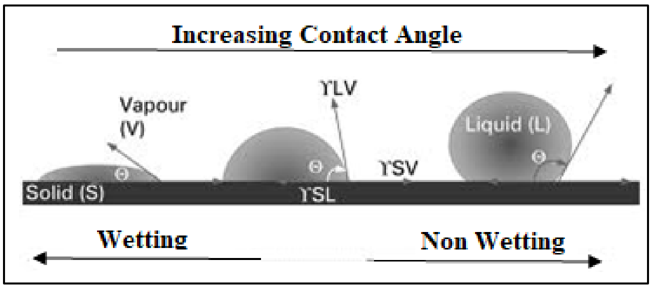

Fig 1: Static contact angle according to sessile drop method

2.5 Wilhelmy plate method

This method is used to measure dynamic contact angles of the asphalt binder with various probe liquids and to determine surface energy components of the binder. A glass slide (25.4mm× 76.2mm × 1mm) coated with the asphalt binder and suspended from a microbalance is immersed in a probe liquid. From simple force equilibrium conditions, the contact angle of the probe liquid with the surface of the asphalt binder can be determined. Contact angles are obtained by analyzing the image using image processing software (Comef 4.3) software. Figure 2 exhibit the contact angle according to Wilhelmy plate method. The test was conducted following the procedure by Hefer *et al*.,[19].

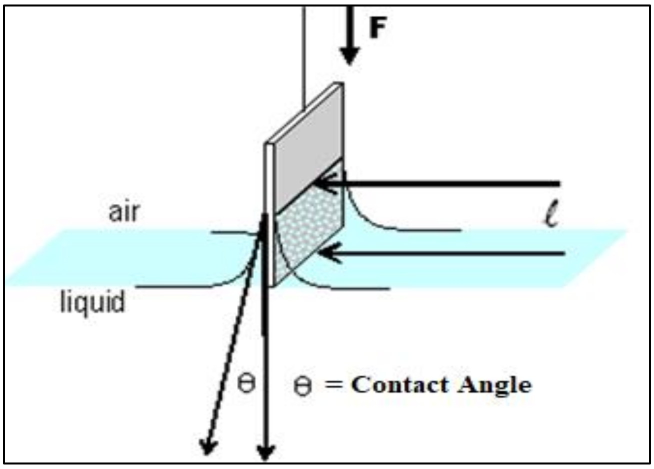

Fig. 2. Dynamic Contact Angle According to Wilhelmy Plate Method

3. Results and Discussions

3.1 Influence of ageing on physical properties of asphalt cement

Figure 3 demonstrate the influence of aging on physical properties of asphalt cement, the consistency of the binder as indicated by the penetration value was reduced by 13 % after ageing and a harder binder consistency was obtained. This could be attributed to the loss of volatiles during the ageing process. The softening point is a representative of the capability of the binder material to flow at elevated temperatures and it is useful in the classification of asphalt cement. It reflects the reduction in viscosity at elevated temperature. Softening point increased by (11) % after ageing process due to stiffening of binder. The ductility which is a measure of tensile behavior of the binder at a particular speed and temperature decreased by 12 % after ageing process. The viscosity which is also a measure of consistency of the binder increase by 1.3 % after ageing process. Similar findings have been reported by Sarsam, [20].

3.2. Influence of Ageing on Rheological Properties of Asphalt Cement

Figure 4 exhibit the influence of ageing on rheological properties of asphalt cement, it can be noted that the negative value of penetration index (PI) which is a measure of temperature susceptibility of asphalt binder, decreases by 74.6 % after ageing. The rate at which the consistency of asphalt cement changes with the change in temperature is known as temperature susceptibility. Asphalt cement with high negative values of (P.I.) is more susceptible to temperature. This behavior indicates an improvement in the binder property against temperature susceptibility. The Penetration Viscosity Number (P.V.N.) increases by 10.8 % after ageing indicating lower temperature susceptibility. The influence of ageing on Temperature of Equivalent Stiffness (T.E.S.) and Viscosity Temperature Susceptibility (V.T.S.) was not significant. On the other hand, the stiffness modulus increases by 26.6 % after ageing process. This is also a positive behavior for resistance of deformation, while it has a negative impact for initiation of fatigue cracking. Such behavior agrees with Little *et al.* [2].

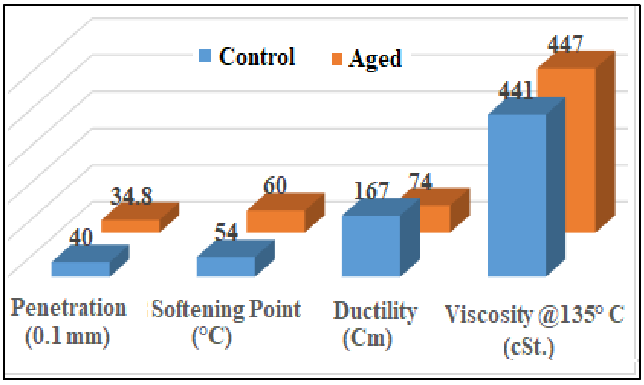

Fig. 3: Influence of Aging on Physical Properties

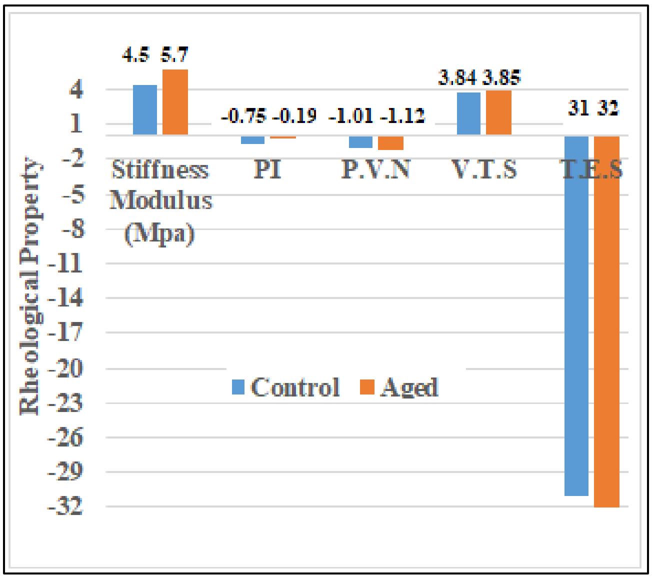

Fig. 4: Influence of Aging on Rheological Properties

3.3 Influence of Ageing on Surface Free Energy of Asphalt Cement

Figure 5 demonstrate the influence of ageing on static contact angle of the binder when implementing Sessile drop method using various prob liquids. It can be noted that the static contact angle increases after ageing process by (68.7, 151.7, and 64.6) % for (water, formamid, and Glycerol) prob liquids respectively. On the other hand, as demonstrated in Figure 6, when implementing the Wilhelmy plate method, the dynamic contact angle also increases after ageing process by (24.2, 8.4, and 3.8) % for (water, formamid, and Glycerol) prob liquids respectively. It can be observed that the static contact angles after ageing are about twofold higher than the dynamic contact angles regardless of the prob liquids implemented. The increment of contact angle indicates the proper wetting adhesiveness which decline the surface free energy. Such finding agrees with the work by Sarsam and Al-Sadik,[21]. Figure 7 exhibit part of the prepared slides for contact angle measurement.

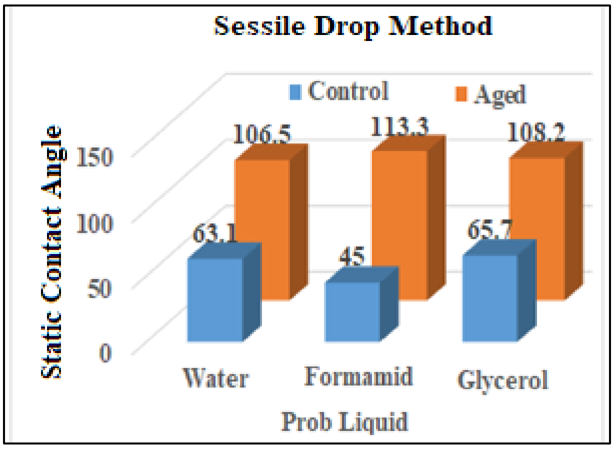

Fig. 5: Impact of Ageing on Static contact Angle

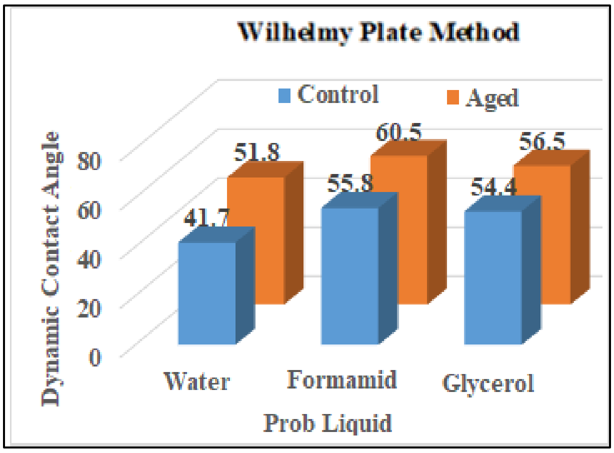

Fig. 6: Impact of Ageing on Dynamic contact Angle

Figure 8 illustrates the surface free energy data of asphalt binder calculated for both testing methods. The surface free energy of asphalt binder decreases by (76 and 7.8) % after practicing ageing when tested by Sessile Drop and Wilhelmy plate methods respectively. This may be attributed to the fact that ageing process conducted at (TFOT) had lowered the flexibility of the binder through the loss of volatiles. This can lower the work of cohesion of asphalt binder, and the fracture resistance of the binder was reduced, leaving the binder to be more susceptible to stiffening and cracking. However, the surface free energy of aged binder is lower by 42.5 % when tested by Sessile drop method as compared to that tested by Wilhelmy plate method.

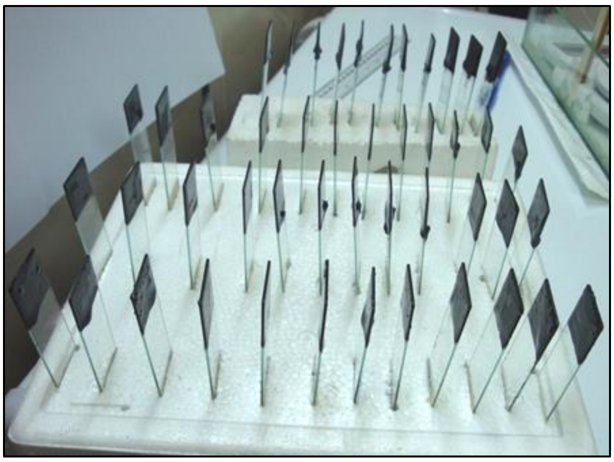

Fig. 7: Part of the slides for contact Angle measurement

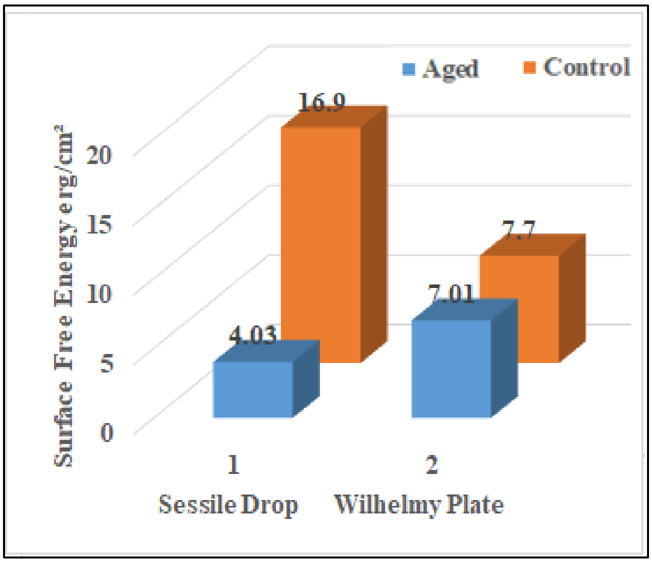

Fig. 8: Surface Free Energy Data

4. Conclusion

Based on the limitations of materials implemented in this investigation and the testing program, the following conclusions could be raised.

1. The penetration, ductility, and viscosity values were reduced by (13, 12 and 1.3) % respectively after ageing and a harder binder consistency was obtained while the softening point increased by (11) % after ageing process.
2. The negative value of penetration index (PI) which is a measure of temperature susceptibility of asphalt binder, decreases by 74.6 % after ageing, while the Penetration Viscosity Number (P.V.N.) and the stiffness modulus increases by (10.8 and 26.6) % respectively after ageing.
3. The static and dynamic contact angles increase after ageing process by (68.7, 151.7, and 64.6) % and (24.2, 8.4, and 3.8) % for (water, formamid, and Glycerol) prob liquids respectively when implementing the Sessile drop and Wilhelmy plate methods.
4. The surface free energy of asphalt binder decreases by (76 and 7.8) % after practicing ageing when tested by Sessile Drop and Wilhelmy plate methods respectively. The surface free energy of aged binder is lower by 42.5 % when tested by Sessile drop method as compared to that tested by Wilhelmy plate method.
5. Static contact angles after ageing are about twofold higher than the dynamic contact angles regardless of the prob liquids implemented.
6. It was recommended that asphalt cement should practice the physical, rheological, and surface free energy testing before a decision can be made of its suitability to be used for pavement under a specified environmental condition.

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