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The Effect of Ageing on Physical and Chemical Properties of Asphalt Cement

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Abstract

Two grades of paving asphalt with penetration of 46 and 65 are studied for determining changes in their physical and chemical properties caused by ageing.

The ageing process has been conducted on two petroleum paving asphalt cement using thin film oven test at 150, 163 and 175 C, and ageing time 5, 10,15, 20, 25 and 30 hours. The effect of ageing time and temperature on penetration, kinematic viscosity, softening point, solubility in trichloroethylene, heat loss and changes in chemical composition are investigated. The results of thin film oven test process indicte that the asphaltenes concentration of all aged asphalt increases with increasing ageing time, while the opposite was observed for polar-aromatic and naphthene-aromatic. The saturate of low penetration grade asphalt is higher than that for high penetration grade asphalt and remained unchanged with ageing time. Th kinematic viscosity and softening point seem to increase with increasing ageing time, while the penetration decreases with increasing ageing time. The DI of aged asphalt inreases with increasing ageing time.

Introduction

Asphaltenes and polar-aromatic play a fundamental role in determining the mechanical and rheological properties of asphalts (penetration index and kinematic viscosity with ageing time and temperatures). 0ther important asphalt properties such as temperature susceptibility coefficient and characterizing factor were depended on the quantity of the asphaltenes and polar-aromatics. Asphalt separation procedure is based on solubility in normal heptane followed by adsorption chromatography of the soluble portion [1].

The term age hardening is used to describe the phenomenon of hardening. Hardening is primarily associated with loss of volatile components in asphalt ageing during the services. This factor causes an increase in viscosity of the asphalt and an increase in Marshal Stability.

Durability of asphalts is a prime consideration in the economics of all asphalts contractions. Asphalts highway systems present the more obvious and perhaps the most important area in which asphalt performance is observed. The term durability during the time connotes a pavement life of about 75 years. The hardening show by an asphalt and service conditions, was for a long time taken as the best measure of its economic value. Resistance to deterioration is a term used to some extent, but it does not seem appropriate since durability under service conditions is frequently affected by changes that, in the strictest sense [2].

Traxler [3] measures age hardening with a falling coaxial cylinder type of viscometer, allowing sample to age. Important researches by this investigator showed that viscosity increases after long time periods and is largely diminished by heating.

Asphalt composition is largely dependent upon the crude from which it was derived. Composition as used in Corbett [4] research is based upon the qualitative determination of the four generic fractions found in all asphalts, using the proposed ASTM procedure. Each fraction differs considerably in color, density and aromatic carbon content. An asphalt meeting a given specification is thus a composite of these quantities, of which the flow properties.

Several asphaltic material and constituent groups (asphaltenes, petroleum resins, asphaltic resins and oils) subjected to oxygen in a closed system under atmospheric pressure at about 204 °C. The volume of oxygen absorbed by each material and fraction was measured. Asphaltenes oxidized but the oils were quite resistant at the temperature used. The oxidized asphaltenes contained considerable amount of carboids (up to 96 %) after 3 hours in the oxygen atmosphere. Carboids are insoluble in carbon disulphide [5].

The durability of asphalt as measured by its hardening in the dark was investigated. Thin films of asphalt on sand grains were used and the volume of oxygen observed by gram of asphalt was determined. Also, 5-mm thick films of asphalts on glass plates were exposed to air in the dark at fixed temperature for many weeks. Samples were removed at intervals and the increase in viscosity caused by the ageing determined by means of the microfilm viscometer. This general method was modified and applied by exposing a 5 mm films of asphalt to ageing on glass plates for 2 hours at 107 C [6] .The hardening which occur is determined by measuring the viscosity of the asphalt before and after ageing. The durability is reported as an ageing index which is the ratio of the viscosity of the aged sample to that of the original, both viscosities having been determined at the same shear rate. A low ageing index signifies more durable asphalt. The original viscosity is obtained from film prepared in the same manner but not aged.

Ishaiet [7] have adopted the durability index to reflect the relationship between ageing and the internal colloidal structure of asphalt. This index is given by the ratio of the sum of the asphaltenes (A) and saturates(S) to the sum of the polar-aromatics (P A) and naphthene-aromatics (N A).

$$DI = (A + S) / (P A + N A) \qquad (1)$$

Rosttler [8] describes the asphaltene fraction as the component of asphalt primarily responsible for asphalt viscosity and colloidal behavior due to its limited solubility in the remaining components. He concluded that the asphaltenes are kept dispersed by the peptizing ability of the nitrogen bases, on other hand, the paraffin tend to reduce solubility, so the increasing amount of this constituent in relation to the nitrogen bases would lead to increasing gel characteristics.

This work deals with the study of factors affeting the quality of different asphalt cements based on ageing time and temperature.

Experimental Work

Raw material

Two grades of petroleum asphalts are supplied from Daura refinery. The physical and chemical properties of these asphalts are presented in table 1.

Chemicals

1. Alumina F-20 (28-200 mesh) chromatographic grade is used for separation of hydrocarbon groups from the petroleum asphalts.
2. Normal heptane.
3. Trichloroethylene (TCE).
4. Benzene.
5. Toluene.

Test methods

1. Durability test

The standard thin film oven test (ASTM-D 1754) has been employed to aged the asphalt film at different temperatures and times for various types of asphalt cements. All aged samples have been tested for penetration, kinematic viscosity, softening point, heat loss and solubility in trichloroethylene. Separation of asphaltenes, polar-aromatics, naphthene-aromatics and saturates were carried on all aged samples.

The changes in physical properties and chemical composition of asphalt cements due to the effect of heat and air are evaluated by thin-film oven test. In all tests 3.2 mm film thickness of asphalt cement is placed in cylindrical pans in an oven maintained at temperatures 150, 163 and 175 C for ageing intervals 5, 10, 15, 20, 25 and 30 hours. Changes in the various physical and chemical properties after oven ageing are then determined. Figure 1 shows the unit of the laboratory durability test Details about the test method were presented elsewhere [9].

2. Fraction composition (ASTM-D 2024).

The precipitation of asphaltenes is applied for all petroleum asphalts and aged samples at temperature range of 150-170 C and time of 5-30 hours. The asphaltenes precipitation carried out by normal heptane.

Ten grams of the sample was dissolved in 150 ml of normal heptane and warmed up to 35 C with stirring for 5 minutes. The mixture of asphalt Sample and solvent was allowed to stand for 30 minutes at room temperature and then filtered with Buchner funnel having a fine porosity filter disk. The precipitate was washed four times with 50 ml of n-haptane. The weight of solid precipitate is calculated as weight percent of asphaltenes. The fraction soluble in n-heptane was evaporated and further fractionated by adsorption chromatography.

Group composition for all raw and aged asphalts is determined by adsorption chromatography. A glass percolation column (20 mm diameter and 400 mm hieght) was filled with 300 g of activated alumina F-20. A 500 ml separating funnel containing the deaspaltened sample was put in the top of the column.

The deaspaltened asphalt was diluted with n-heptane.and allowed to percolate through the alumina bed with n-heptane. Then a defined quantity of effluent (saturates) was collected from the bottom of the column. The separating funnel was removed from the top of the column, then a benzene-toluene mixture (1:1) was charged to the top of the column. The mixture dissolved the polar-aromatics fraction which drawed from the bottom of the column.

The alumina column was then percolated with trichloroethylene for naphthene-aromatics removal from the bottom of the column. The percent weight of each fraction was calculated after solvent stripping.

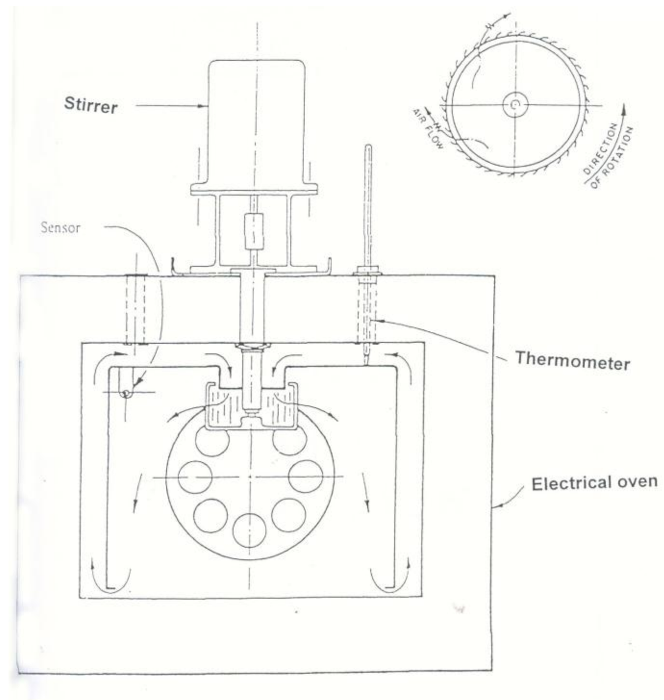

Fig. 1: Scheme of airflow for durability test

3. Penetration

Penetration is a test for the consistency of asphalt. It is based on a standard needle entering asphalt under fixed conditions. The depth of the penetration of the standard needle into the material to be tested within 5 seconds, with a load of 100 g at 25 C is measured in this standard test. The penetration is measured in 0.1 mm.

4. Softening point

Asphalt with high viscosity like solid is soften gradually on heating. The softening temperature determined at described condition is called softening point. The softening point (ring and ball test) is the temperature at which the asphalt layer placed in a standard ring touches the plate under the ring due to the weight of a ball of definite mass and size.

5. Viscosity

The kinematic viscosity test according to the ASTM-D 3120 is measured by using a gravity flow viscometer. The viscometer is putting in a bath at constant temperature. The molten asphalt is charged to the viscometer up to the filling line. The viscometer with the asphalt remains in the bath at test temperature for a defined period of time to make sure that the viscometer with the asphalt is at test temperature.

Results and Discussion

The thin-film oven test has been employed to age the asphalt at a range of temperature 150 to 175 C and a range of time 5 to 30 hours. All aged samples have been tested for penetration, kinematic viscosity, softening point, heat loss and solubility in trichloroethylene. Further more, the aged samples were separated into asphaltenes,naphthene-aromatics, polar-aromatics and saturates.

Figures 2-7 show the influence of ageing time at different temperatures on physical properties of the different grades of petroleum asphalts.

The increase in ageing time from 5 up to 30 hours causes an increase in consistency to flow and loss in volatile. For example, for paving petroleum asphalt grade 60-70 the penetration and softening point at 163 C were 46 and 54 C, respectively for ageing time 5 hours, while they were 43 and 55 C,respectively for ageing time 10 hours. This is due to the conversion of the naphthene-aromatics and polar-aromatics to asphaltenes, which lead to decreasing of penetration and increasing of softening point.

Figures 8-15 show the influence of ageing time at different temperatures on chemical composition change of the asphalts. These figures indicate that the asphaltenes increase with increasing of ageing time, while naphthene-aromatics, polar-aromatics and saturates decrease with increasing ageing time.The increase in asphaltenes is due to the partial conversion of naphthene-aromatics, polar-aromatics and saturates to asphaltenes.

Figures 16 and 17 indicate that durability index increases with ageing time increasing. The increase in durability index is due to increase in the asphaltenes and decrease in naphthene-aromatics and polar-aromatics with ageing time increasing.These results agree with the results obtained by Tuffour for bitumenious materials [2].

Figures 18 and 19 show the effect of ageing time on the percentage of heat loss of the aged samples, these figures indicate that the percentage of heat loss increase with ageing time increasing. This may be due to partial evaporation of the oil from the asphalt during ageing.

Table 2 is summarized the range of durability index of asphalts at different ageing temperatures. This table indicates that low penetration petroleum asphalt cements is less susceptible to ageing time than soft asphalt. This is because the saturates fraction of petroleum asphalt increases the resistance to flow. The heat treatment of petroleum asphalt during vacuum distillation affects its physical and chemical properties, so it become high susceptible to ageing time.

Table 1 Physical and chemical properties of petroleum asphalt cements

No. Test	Grade 40-50	Grade 60-70	Test method
1. Penetration (0.1 mm)	48	68	ASTM-D 5
2. Softening point R&B (C)	51	50	ASTM-D 36
3. Kinematic viscosity at 135 C (Cs)	443	356	ASTM-D 2170
4. Solubility in TCE (wt %)	99.7	99.8	ASTM-D 86
5.Fractional composition (wt%)			ASTM-D 2024
Asphaltenes	21.2	17.9	
Saturates	21.3	16.3	
Naphthene-aromatics	44.5	45.9	
Polar-aromatics	11.5	20.9	
6. Thin-film test			ASTM-D 1754
Loss by heating (wt %)	0.3	0.5	
Penetration (0.1 mm)	36	46	
Softening point (C)	54	55	

Table 2 The change durability index(DI) after ageing time 30 hours at different temperature.

Characteristics	Grade 60-70	Grade 40-50
Range of DI at,		
150 C	0.150-0.811	0.759-1.361
163 C	0.150-0.927	0.759-1.477
175 C	0.150-1.087	0.759-1.660
Increase in DI at,		
150 C	0.661	0.602
163 C	0.777	0.718
175 C	0.937	0.901

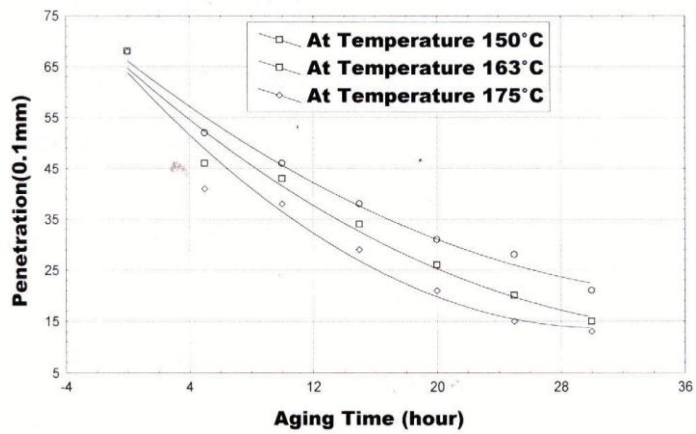

Fig. 2 Influence of aging time on penetration of petroleum asphalt grade 60-70 at different temperature.

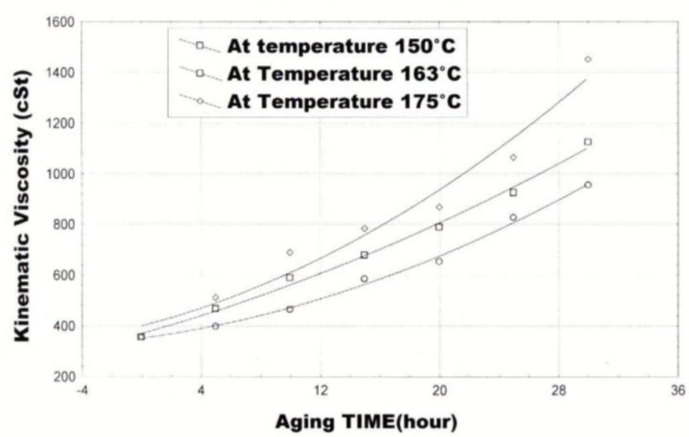

Fig. 3: Influence of aging time on kinematics viscosity of petroleum asphalt grade 60-70 at different temperature.

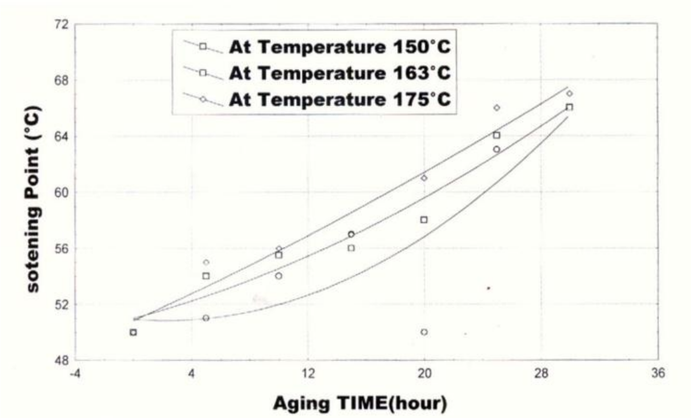

Fig. 4: Influence of aging time on softening point of petroleum asphalt grade 60-70 at different temperature.

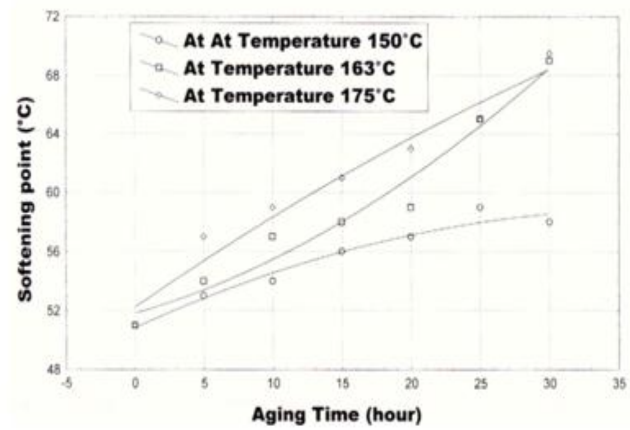

Fig. 7 Influence of aging time on softening point of petroleum asphalt grade 40-50 at different temperature.

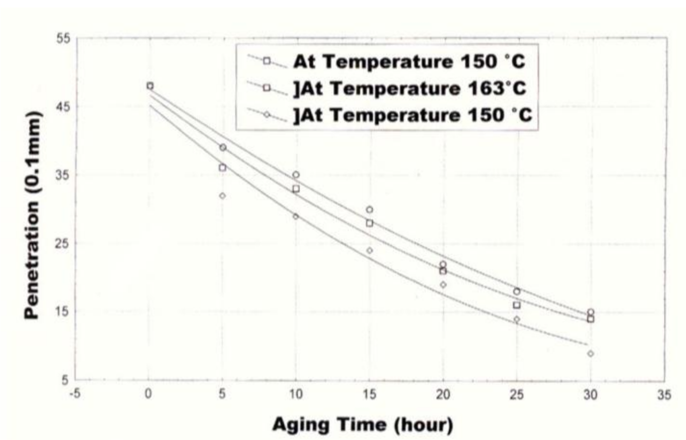

Fig. 5: Influence of aging time on penetration of petroleum asphalt grade 40-50 at different temperature.

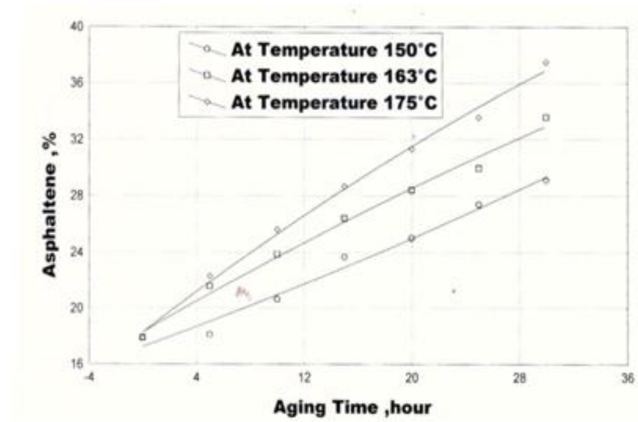

Fig. 8 Influence of aging time on Asphaltene frictions at different Temperature of petroleum asphalt grade 60-70

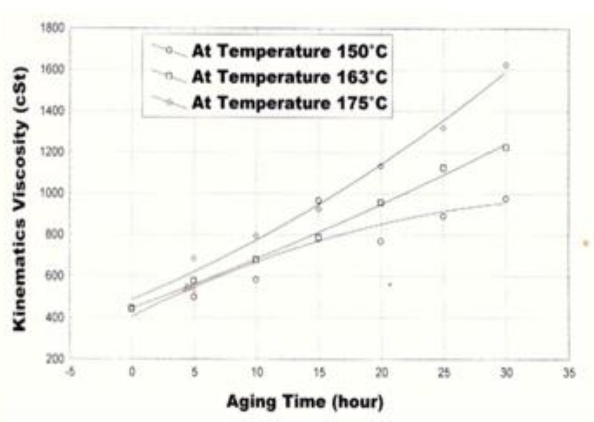

Fig. 6 Influence of aging time on kinematics viscosity of Petroleum asphalt grade 40-50 at different temperature.

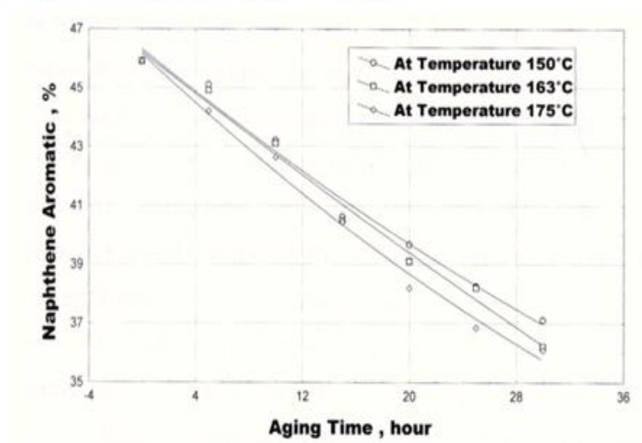

Fig. 9 Influence of aging time on Naphthenic Aromatic frictions at different Temperature of petroleum asphalt grade 60-70

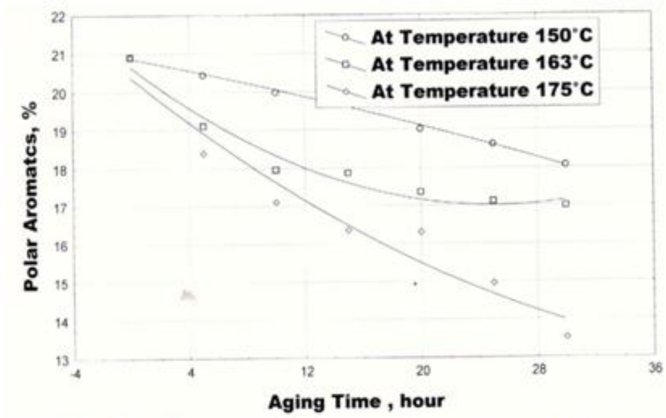

Fig. 10 Influence the aging time on Polar Aromatic frictions at Different Temperature of petroleum asphalt grade 60-70

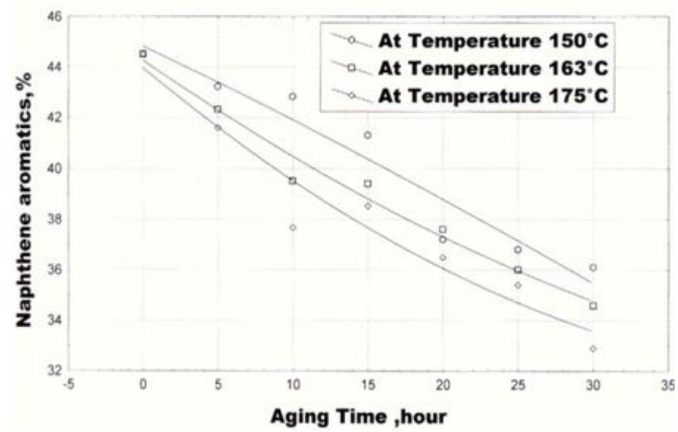

Fig. 13 Influence the aging time on Naphthene aromatic frictions at Different Temperature of petroleum asphalt grade (40-50).

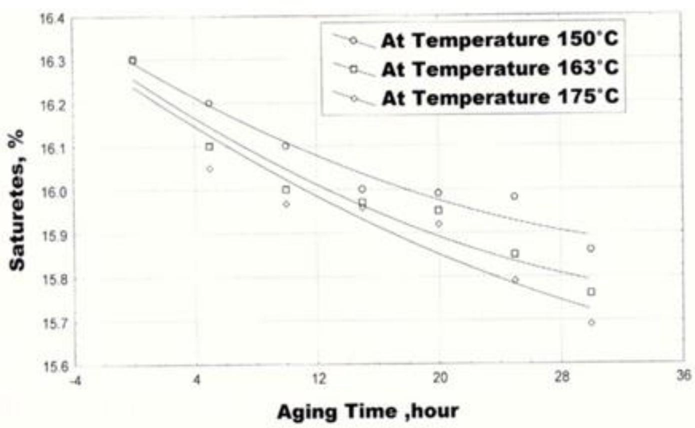

Fig. 11 Influence the aging time on saturate frictions at Different Temperature of petroleum asphalt grade (60-70).

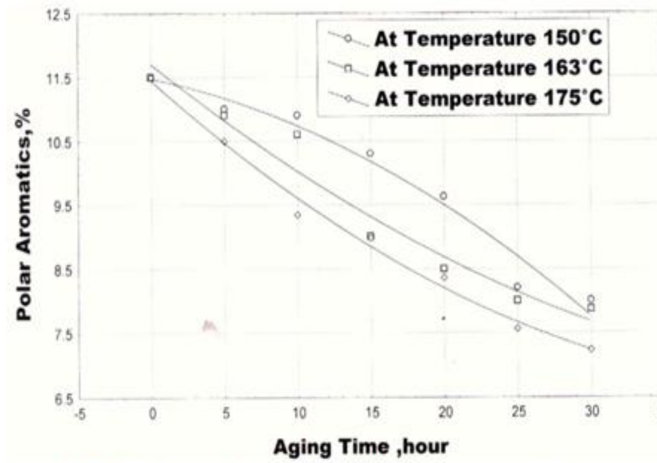

Fig. 14 Influence the aging time on the polar aromatic frictions at Different Temperature of petroleum asphalt grade (40-50).

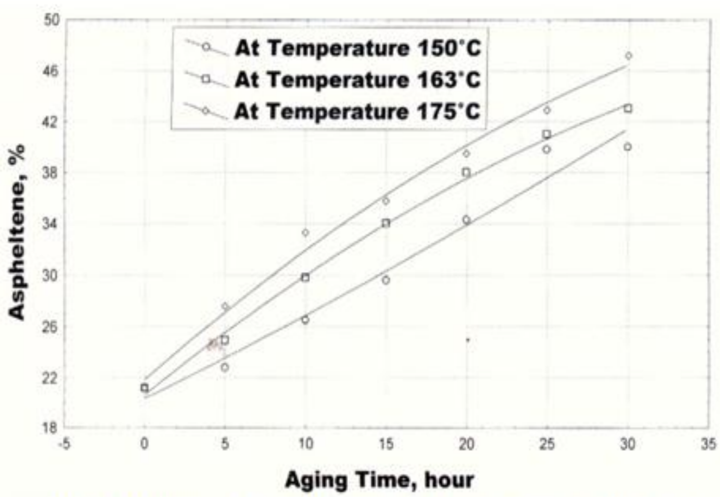

Fig. 12 Influence the aging time on Asphaltene frictions at Different Temperature of petroleum asphalt grade (40-50).

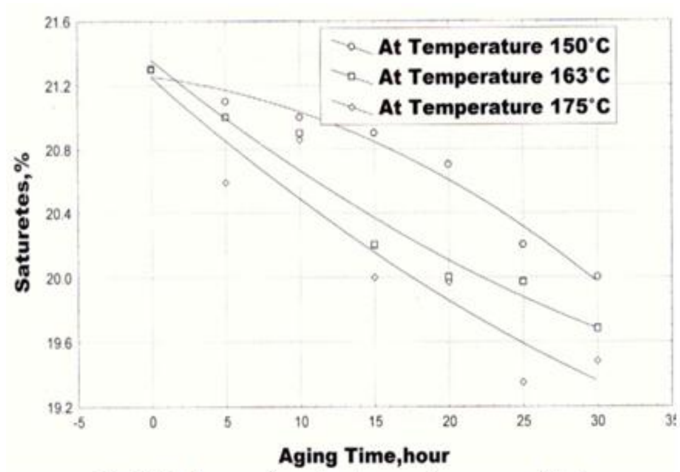

Fig. 15 Influence the aging time on the saturate frictions at Different Temperature of petroleum asphalt grade (40-50).

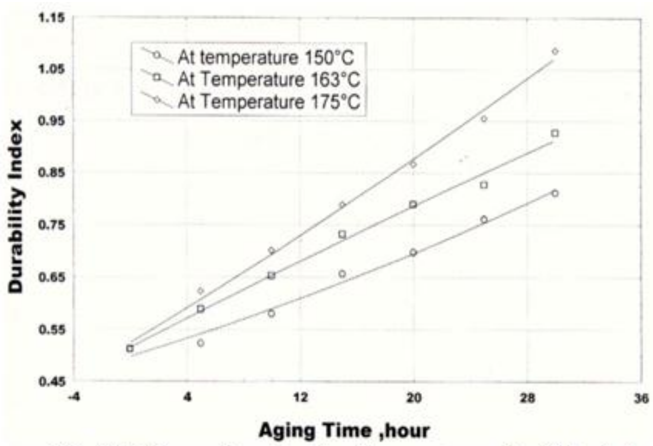

Fig. 16 Influence the aging time Temperature on durability index Of petroleum asphalt grade (60-70)

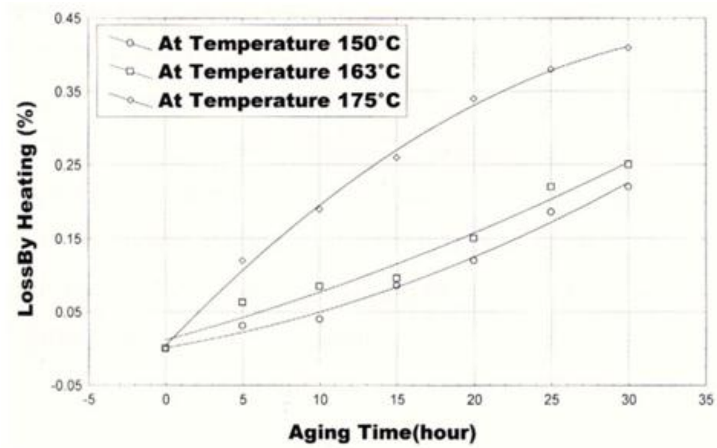

Fig. 19 Influence the aging time on loss by heating of petroleum Asphalt grade (40-50) at different temperature

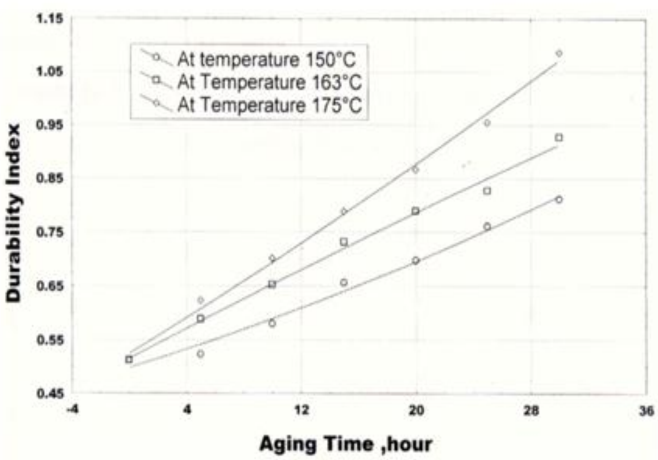

Fig. 17 Influence the aging time Temperature on durability index Of petroleum asphalt grade (40-50)

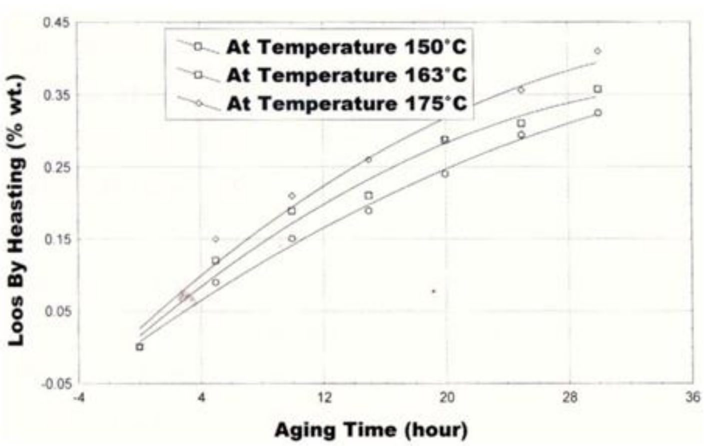

Fig. 18 Influence the aging time on loss by heating of petroleum Asphalt grade (60-70) at different temperature

CONCLUSIONS

1. The asphaltenes content for paving asphalt increase with ageing time at different temperatures. The asphaltenes content is ranged from 17.89 to 40.00 % at ageing time from 5 to 30 hours at different temperatures.
2. Part of the polar-aromatics, naphthene-aromatics and saturates is converted to asphaltenes with ageing time and temperature increasing. This lead to decrease the percentage of polar-aromatics, naphthene-aromatics and saturates in the aged asphalt.
3. The durability index for aged asphalt increases with ageing time and temperature increasing.

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