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Reuse of Aged Asphalt: A Key to Natural Resources Sustainability

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Abstract

Based on Egypt's 2030 vision, road paving becomes the principles of sustainable development and it is a matter of necessity for all national projects as *"Hayah Karema"*. Moreover, the form of re-utilization of paving materials namely asphalts and solid aggregate is economic and environmentally friendly. Aging of asphalt resulting from a series of physical and chemical changes occurring and changing its chemical composition and structure occurring during the application and service life so, it finally hardened and cracking gradually appears. This research aims to reuse aged asphalt using two types of vacuum residues (VR) that obtained from two different petroleum refining companies. To achieve this aim, virgin asphalt of type AC60/70 is aged at lab using Rolling Thin Film Oven Test (RTFOT) and Pressure Aging Vessel (PAV) representing the short and long aging stages. Aged asphalt as well the rejuvenators VR types are characterized for physical and chemical analyses in addition to FTIR and SEM analyses according to ASTM standard and Egyptian code. The needed amount of addition of each VR type to rejuvenate the aged asphalt is determined by establishing the blending chart. Variable mixtures of aged asphalt and VR types are prepared at different percentages and evaluated as previously mentioned in addition to the determination of highest temperature at which the rejuvenated asphalt will stand safe using Dynamic Shear Rheometer (DSR). The results revealed that, the obtained rejuvenated asphalt using the vacuum residue is suitable for reuse in road paving according to Egyptian standard for AC 60/70 and the two residues are successful rejuvenator agents.

Keywords: *Aged asphalt; Short aging; Long aging; Softening agent.*

1. Introduction

Asphalt has been widely used in pavement construction due to its excellent viscoelasticity behavior [1]. Asphalt was firstly used in road construction at King of Babylon in 625–604 B.C [2]. However, the large-scale asphalt pavement construction has led to an increasing shortage of petroleum asphalt binder which is a non-renewable resource [3-4]. Asphalt is primarily composed of a hydrocarbon content of around 90%, heteroatoms and metals. Heteroatoms include nitrogen (0–2%), oxygen (0–2%), and sulphur (0–9%). Metal atoms are vanadium, nickel, and iron, and these atoms are present in trace quantities, typically far less than 1% [5-7]. All chemical elements combine forming the four main fractions of asphalt cement namely: saturates, naphthalene aromatics, polar aromatics (or resins) and asphaltenes, (SARA fractions). In Egypt, AC specification is based on penetration and kinematic viscosity at 135°C established by both " General Authority for Roads, Bridges and Land Transportation in Egypt" as well as the" Egyptian code for Urban & Rural Roads (Part 4: Road materials and their tests)" issued by Ministry of Housing Utilities & Urban Communities .

However, the detrimental effects of hardening in asphalt pavements were first recognized in the 1900s and have been studied extensively during the last 70 years [4]. Asphalt is easy to age as a blended organic compound material in the application and service life (storage, transportation, construction and service, etc.), with effects of temperature, oxygen, ultraviolet radiation [8-9]. Aging and rejuvenating mechanism of asphalt using Fourier Transform Infrared Spectroscopy (FTIR), provide that the aging of bitumen comes from three aspects: Loss of volatiles, dehydrogenation and oxidation in addition to thixotropy polymerization and condensation polymerization [10-12] in two stages namely, short and long terms aging take place at high and low temperatures respectively. The oxidation reactivity of SARA fractions was determined as 1:7:32:40 [13-14]. The functional groups formed by oxidation are mainly ketones, aldehydes, anhydrides, carboxylic acids, and sulfoxides. Sulfoxide is considered the dominant oxidation product in short-term aging, and ketone is considered to be the dominant oxidation product in long-time aging [15-16]. Accordingly, the pavement surface is hardened leading to cracks only in the top inch of the pavement and that below the top inch; the binder is left virtually unaffected by years of use and years of environmental exposure. Finally, as a result of aging; asphaltene fractions generally increase between 1 and 4% and viscosity increases from 150 to 400% [17].

Asphalt aging can be delayed using anti-aging agents and additives and/or it can be recycled in case of complete aging using some types of rejuvenators.

According to the different mechanisms of aging, anti-aging agents can be divided into different types, such as ultraviolet absorbers, antioxidants and hindered amine light stabilizers. [18]. Carbon black, wood lignin, multidimensional nanomaterial, etc are examples of antioxidants. Some antioxidants function by reacting with polar compounds and/or oxidation catalysts such as metals present in asphalts [19-22].

Asphalt recycling has become an important issue used to minimize production costs of new pavement. Furthermore, stricter environmental regulations and depleting resources have led to the use of recycling agents by promoting the application of higher reclaimed asphalt pavement (RAP) in new mixtures. Recycling agents are classified as two types: rejuvenating agents and softening agents. Softening agents lower the viscosity of the aged binder, while rejuvenators are intended to restore the composition of aged asphalt to a stable equilibrium state Flux oil, lube stock and slurry oil are examples of softening agents [23].

The rejuvenator should be easy to disperse in recycled mixtures, compatible with the old asphalt to ensure that synersis will not occur, uniform in properties from batch to batch, have the ability to re-disperse the asphaltenes in the old recycled asphalt, available and cheap.

Generally, asphalt rejuvenators are categorized as paraffinic oils (refined used lubricating oils), aromatic extracts (refined crude oil products with polar aromatic oil components), naphthenic oils (derived from vegetable oils) and tall oils (paper industry by-products-of the same chemical family as liquid antistrip agents and emulsifiers), and waste cooking oil [24-27].

2. Research objective

This research aims to reuse aged asphalt by mixing with two types of rejuvenators separately namely vacuum residues (VR) obtained from two different petroleum refining companies.

3. Materials and experimental work

3.1. Materials

The used materials are Virgin AC 60/70 was obtained from Alexandria Petroleum Company (APC). Two different residues produced by Amerya Petroleum Refining Company (APRCO) and Alexandria Mineral Oils Company (AMOC). VR1 is obtained as a byproduct of the vacuum distillation tower while, the VR2 is produced after subjecting the atmospheric distillation residue produced by APRCO to vacuum distillation followed by deasphalting and dearomatizing processes.

3.2. Experimental program

The experimental program includes the characterization of all raw materials from point of views of physical, chemical, FTIR tests. The virgin asphalt is aged at lab using Rolling Thin

Film Oven (RTFO) followed by Pressure Aging Vessel (PAV). The proper needed amount of each VR type to rejuvenate the aged asphalt is determined using the blending chart. Different blend of each VR type with aged asphalt are prepared, characterized and evaluated according to Egyptian standard of virgin AC60/70. The optimum chosen blends are analyzed as previously mentioned in addition to determination of highest temperature at which rejuvenated stand safely during its service life on road using the Dynamic Shear Rheometer (DSR) and compared to virgin AC sample.

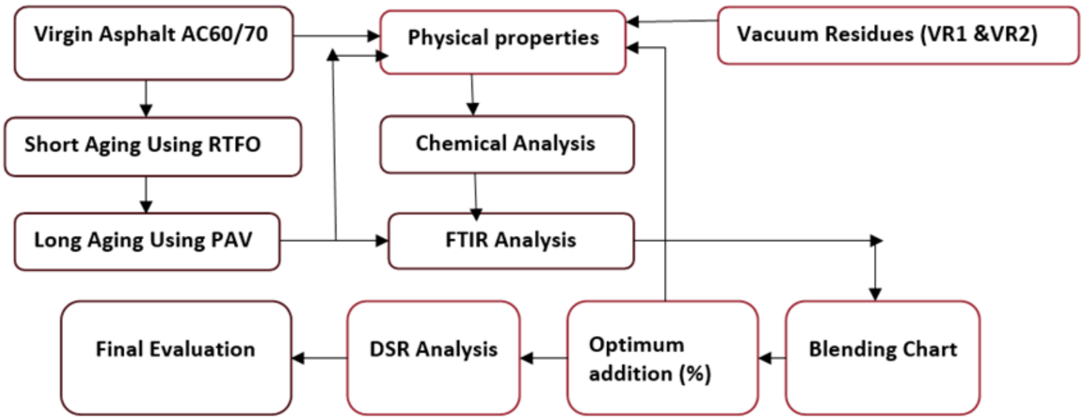

Fig 1. Experimental program.

4. Results and discussion

4.1. Step 1. Laboratory aging of asphalt

4.1.1. Short aging of asphalt using RTFO

In this step, virgin sample AC 60/70 was aged in Rolling Thin Film (RTFO) according to AASHTO T 240 and ASTM 2872. RTFO is a model 81-PVO161, CINTROLS Co.

4.1.2. Long aging of asphalt using PAV

The PAV is adopted for asphalt samples previously aged in RTFO testing by being exposed to PAV according to AASHTO D 5621. It is intended to simulate in-service long-term aging of asphalt binders for 5 to 10 years. The used PAV is of model of PAV s/n: 16-818117-1, Applied Test System ATS Co.,). Figure 2 (a &b) shows RTFO and PAV testers.

Fig. 2 (a): RTFO

Fig.2 (b): PAV tester

4.2. Step 2. Characterization of all asphalt samples

4.2.1. Physical characteristics of all asphalt samples

The physical characteristics of Virgin AC 60/70, RTFO and PAV aged samples are illustrated in Table 1.

Table 1. Physical characteristics of Virgin AC 60/70 and RTFO and PAV aged.

Test	ASTM Test No.	Virgin AC	RTFO Aged	PAV Aged	Egyptian standard (Virgin AC)
Penetration @25ºC, 0.1mm	D5	63	36	23	60/70
Softening point, ºC	D36	49.7	63.2	69.6	45/55
Kinematic viscosity(@135°C), cSt	D2170	357.0	704.0	1310.0	+320
Specific gravity (@ 25ºC)	D70	1.019	1.023	1.028	
Flash point (COC), ºC	D92	336	289	286	+250
Ductility (@ 25ºC,5 cm /sec), cm	D113	+100	60	23	+100
Thin film oven test	D1754		(ND)*	(ND)*	
Loss on heating (%wt.)		0.64			Max 1.0
Retained penetration, (%)		60			Min 54

(*): Not determined

4.2.2. Chemical analysis of all asphalt samples (SARA fractions)

Data in Tables 1& 2 illustrated that comparing to virgin AC, RTFO and PAV aged samples, have chemical constituents completely different from that of virgin AC sample. The aging causes the values of penetration, flash point and ductility to decrease while, the softening point and kinematic viscosity to increase. Also, the oil to convert to resins and then to asphaltene. However, aging causes the saturated and aromatic contents to decrease and asphaltenes and resins contents to increase. All these results due to oxidation, volatilization and polymerization reactions take place during short and long aging staged.

Table 2. Chemical constituents of aged and virgin asphalt samples.

Sample	Virgin AC	RTFO aged	PAV aged
Chemical constituents			
Saturates content, wt%	4.21	2.97	1.37
Aromatics %	50.60	26.84	20.64
Resins content, wt%	21.85	44.40	46.91
Asphaltenes (wt) %	22.79	25.21	30.40
Impurities (non-organics) content, wt%.	0.44	0.58	0.66

(*): Excluded from oil

PAV aged samples are harder than RTFO aged samples. The complete aging of asphalt as presented in PAV simulates the effect of both continued traffic, action of sun, rainfalls and wind action on asphalt. All these factors cause asphalt to lose its flexibility to a big extent and become viscous, fail its function in the asphaltic paving mixture and accordingly it must be removed from the road.

4.2.3. FTIR for virgin and PAV aged asphalt

Figures 3(a-c) show the FTIR analysis of virgin and aged RTFO & PAV samples respectively. The most characteristic peaks of virgin, RTFO and PAV aged asphalt AC samples are illustrated in Table 3.

Table 3. Most characteristic peaks of virgin ACx, RTFO & PAV aged asphalt.

Sample	The most important peaks	The assignments
Virgin ACx	A strong band at 2983 cm^{-1} A strong absorption band at 1375 cm^{-1} A strong absorption band at 1450cm^{-1} A small absorption band at 943cm^{-1} A small absorption band at 674 cm^{-1}	-C-H stretching vibration -Stretching vibration of CH3 -Stretching vibration of CH2 -Non-aromatic C=C double bonds assigned to acid halide appearance

Sample	The most important peaks	The assignments
RTFO aged asphalt	A strong band at 2928cm^{-1} A small absorption band at 935 cm^{-1}	C-H stretching vibration Aromatic C=C double bond
PAV aged asphalt	Signal at 1223 cm^{-1} A strong absorption band at 1373 cm^{-1} A strong absorption band at 1454 cm^{-1} A peak at 1015 cm^{-1} vibration Strong peak at 2850–2960 cm^{-1} region A small signal at 1697 cm^{-1}	The vibration of carbon dioxide, C-O, Stretching vibration of CH_3 Stretching vibration of CH_2 Ascribed to S=O stretching C-H stretching vibrations in aliphatic chains assigned to absorption of aromatic ring

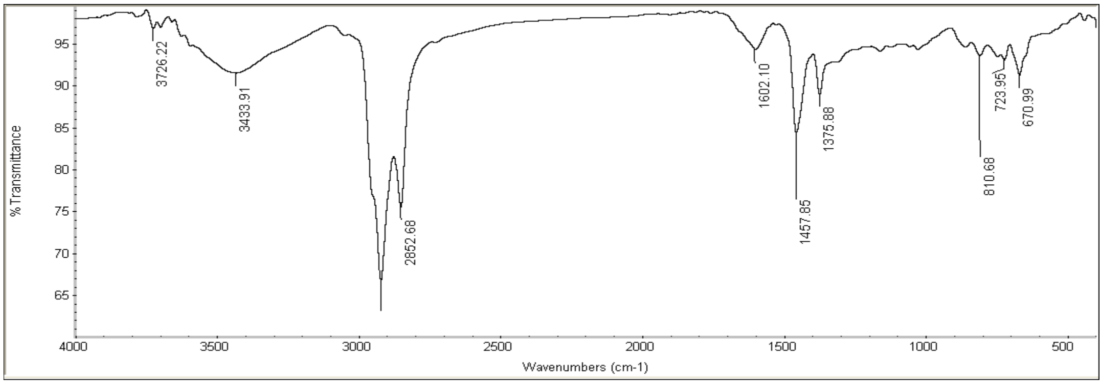

Fig. 3a. FTIR Spectrum of virgin AC sample.

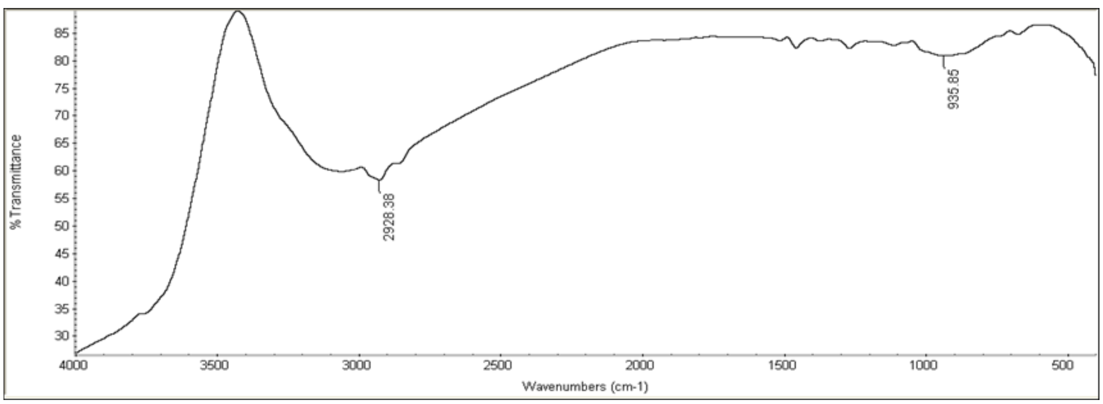

Fig. 3b. FTIR Spectrum of RTFO aged AC sample.

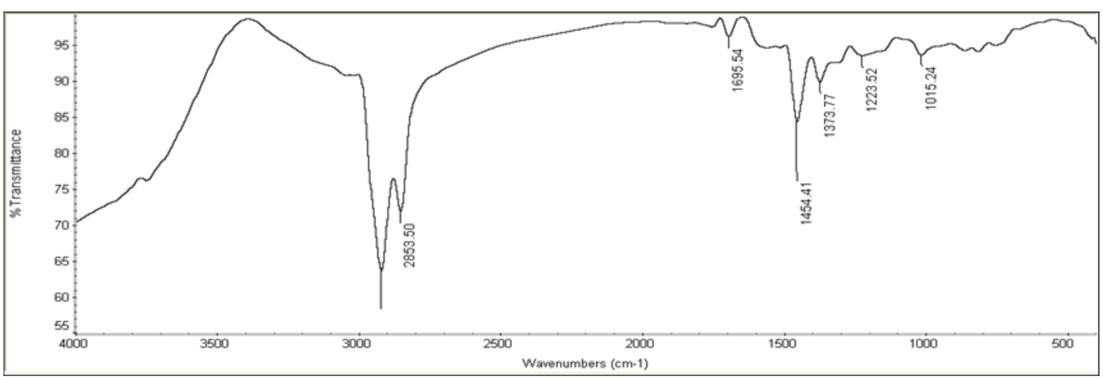

Fig. 3c. FTIR Spectrum of PAV aged AC sample.

From FTIR result, it is obvious that both of RTFO and PAV samples are harder than virgin sample due to aging effects. The appearance of small absorption band at 935 cm^{-1} assigned to the aromatic C=C double bonds in RTFO spectrum and the appearance of small signal at

1697 cm^{-1} assigned to absorption of aromatic ring in PAV aged samples are attributed to the formation of cluster of aromatic rings caused by the aromatization of perhydroaromatic rings. The presence of peaks of CO_2, CO and S=O in PAV aged spectrum confirm the aging of asphalt resulting from the oxidation of hydrocarbons during short and long aging periods of time. The aging causes the compatibility between asphalt components to be reduced and as a result become harder and more viscous.

4.2.4. SEM for virgin and aged asphalt

The surface morphology of virgin and aged asphalt samples was examined by a Field Emission Scanning Electron Microscope (FESEM), model (ZEISS, Gemini, VP, Sigma 300, VP, Germany); the accelerating voltage of electron beam is 30 keV. For better conductivity, the sample were coated with a layer of gold. Photos for the three asphalt samples; virgin, RTFO and PAV aged are shown in Fig.4 (a-c)

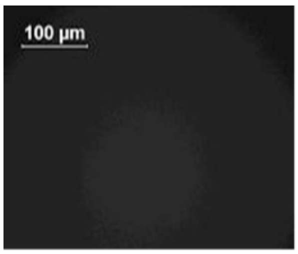

Fig.4a. SEM of virgin AC.

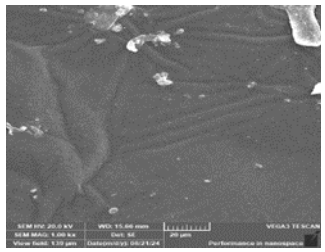
Fig. 4b. SEM of RTFO aged AC.

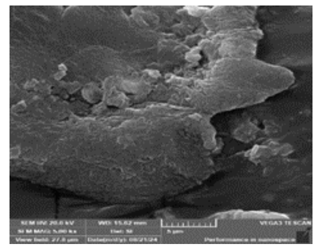
Fig. 4c. SEM of PAV aged AC.

Photo for virgin ACx seems to be homogenous black. However, SEM photos of RTFO is less oxidative (little degraded) compared to PAV photos which appears to be more oxidative. The difference between images of unaged, short-term aged (RTFOT) and long-term aged (PAV) can be clearly seen and can be due to the oxidative ageing of the binder which also affects its viscoelastic behavior of the binder. After ageing, cluster of aromatic rings is formed. This cluster is caused by the aromatization of perhydroaromatic rings. Aromatization takes place (after oxidation and volatilization reactions occur) resulting in attaining more molecular stability by being associated with each other forming a cluster which increase the hardness of aged asphalt.

4.3. Step 3: Characterization of rejuvenator samples

The physical characteristics and the chemical constituents of VR1 and VR2 are illustrated in Tables 4 and 5.

4.3.1. Physical characteristics of all vacuum residue samples

Table 4. Test analysis results of vacuum residue samples.

Test	ASTM	VR1	VR2
Penetration @25ºC,0.1 mm	D5	290	116
Density @15.6ºC, g/mL	D70	0.9549	0.9984
Kinematic viscosity @135°C, cSt	D2170	30.3	104.5
Ductility @ 25 ºC , cm	D113	3.0	32.5
Softening point, ºC	D36	39.8	50.0
Flash point COC, ºC	D92	282	320
Total sulphur, %wt	D2622	3.23	8.2
Pour point, ºC	D97	ND (*)	+67
Water and sediment,% wt		0.05	-
Conradson Carbon Residue, %wt	D189	1.76	15.6
Vanadium content, ppm	D5863	6.5	49.0
Sodium, ppm		3.32	6.45
Potassium ,ppm		2.0	4.6

(*): Not determined

4.3.2. Chemical constituents of all asphalt samples

Table 5. Chemical constituents of Vacuum residue samples.

Sample	VR1	VR2
Saturates content, wt.%	44.78	25.3
Aromatics %	47.77	52.21
Resins content, wt.%	2.04	15.75
Asphaltenes (wt.%.)	5.4	6.72
Impurities (non-organics) content, wt.%	0.01	0.02

The results in tables show that both residues are different in their physical characteristics as a result of the difference in production process route for each sample. VR1 is seems to be softer than VR2 as it has higher penetration, lower kinematic viscosity, ductility, softening point and flash point. All these results are explained as VR1 contains more saturates and lower resins contents comparing to VR2 which contains more resins and aromatic contents.

4.3.3. FTIR analysis of vacuum residue

The constituents of VR1 and VR2 are identified using FTIR analysis as shown in Figure 5 (a & b) while, Table 6 illustrates the most characteristic peaks of residue samples.

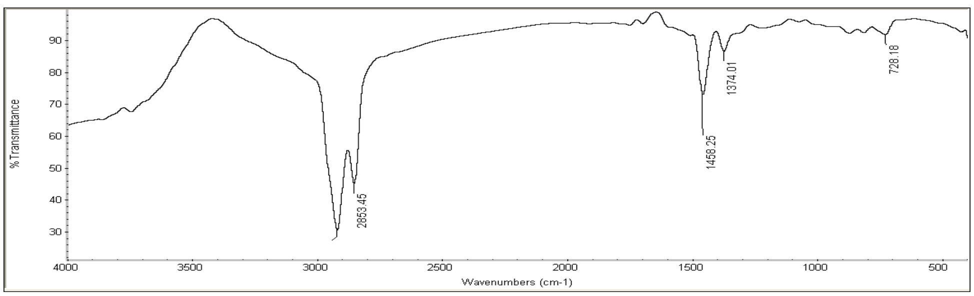

Figure 5a. FTIR Spectrum of VR1 sample.

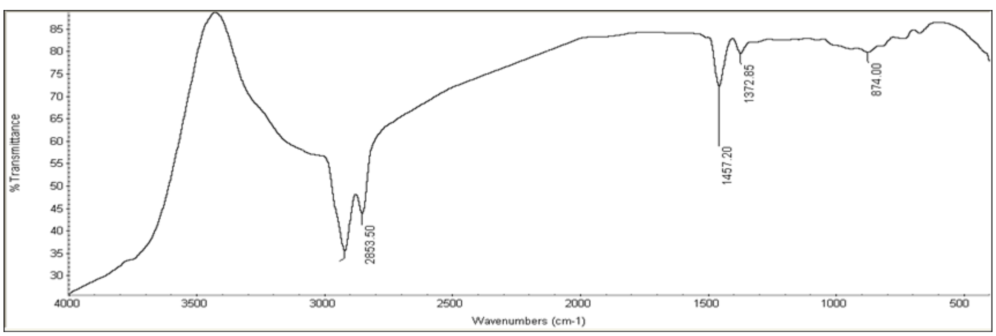

Figure 5b. FTIR Spectrum of VR2 sample.

Table 6. The most characteristic peaks of VR1 and VR2.

Sample	The most important peaks	The assignments
VR1	A strong band at 2853-2921cm^{-1} A strong absorption band at 1375 cm^{-1} A strong absorption band at 1450 cm^{-1} A small absorption band at 728 cm^{-1}	C-H stretching vibration Stretching vibration of CH_3 Stretching vibration of CH_2 C=C bending
VR2	A strong band at 2853-2921cm^{-1} A strong absorption band at 1375 cm^{-1} A strong absorption band at 1450 cm^{-1} A small absorption band at 874 cm^{-1}	C-H stretching vibration Stretching vibration of CH_3 Stretching vibration of CH_2 C=C bending

From all the previous results, it is obvious that both of VR1 and VR2 samples have similar functional groups.

4.4. Step 4. Determination of proper rejuvenators amount needed to soften aged asphalt (blending chart)

The proper suitable content of VR types for rejuvenating aged asphalt sample is determined using ASTM D 4887 entitled "Standard Practice for Preparation of Viscosity Blends for Hot Recycled Asphalt Materials".

4.4.1. Determination of the proper VR contents in the blend

In this step, a chart is established as shown in Figure 6 by determining the kinematic viscosity of both aged asphalt sample (on the left scale) and VR (on the right scale) while the-horizontal axis is the weight percent of VR by weight of a blend of aged binder and the rejuvenator type (VR1 or VR2) knowing as Blend 1 &2 respectively. The values are connected point to point. Based on the Egyptian standards, the lower limit of the kinematic viscosity of virgin AC 60/70 is 320 cSt at 135°C. Moreover, for obtaining a workable hot mix asphalt, the kinematic viscosity of virgin AC 60/70 should not exceed 400 cSt (as recorded for different types of asphalt samples produced from all the Egyptian refineries). Accordingly, by signing the limits on the graph, the highest proper ranges of the percent of each type of VR in the blend are 36 % to 56 % for AC.

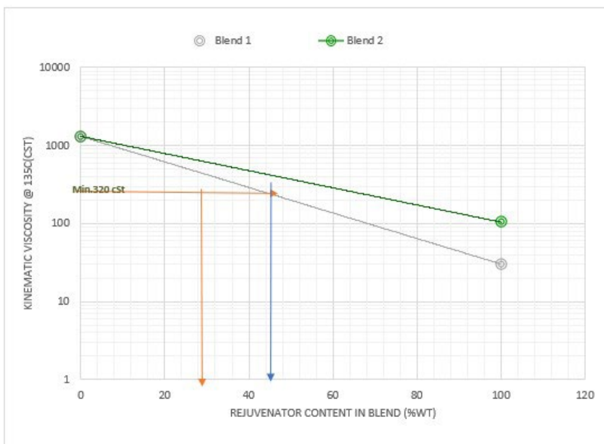

Fig.6. Blending chart

4.4.2. Determination of the optimum VR contents in the blend

To define the optimum percentages of VR needed to rejuvenate aged AC, different mixes of blends containing 32,34 &36% of VR1 and 48,52 and 56% of VR2 separately with aged AC are prepared. The physical characteristics for all the blends are determined as penetration, kinematic viscosity and softening points and compared to standard limits of Egyptian AC 60/70 as illustrated in Table 7.

4.4.2.1. Characteristics of rejuvenated asphalt blends

Table 7. Physical analysis results of VR percentages in blends of AC.

Test	VR1			VR2		
% of VR in blend (wt/wt)	32	34	36	48	52	56
Penetration @25°C,0.1 mm	58	64	71	60	66	72
Kinematic viscosity @135°C,cSt	423	377	318	400	373	290
Softening point, °C	56	53	45	53	51	44

Data in Table 7 illustrated that, 34% and 52% of VR1 and VR2 respectively are the optimum contents of residue types needed to rejuvenate the aged asphalt.

Table 8 illustrated the chemical constituents of the rejuvenated samples containing the optimum contents of VR1 and VR2.

Table 8. Chemical constituents of aged and virgin asphalt samples .

Sample	Virgin AC	Rejuvenated asphalt using VR1	Rejuvenated asphalt using VR2
Chemical constituents			
Saturates content, wt%	4.21	10.06	8.79
Aromatics %	50.60	25.99	30.26
Resins content, wt%	21.85	37.95	37.25
Asphaltenes (wt%)	22.79	25.40	23.06
Impurities (non-organics) content, wt	0.44	0.60	0.64

4.4.2.2. Molecular structure (FTIR) of best rejuvenated asphalt blends

FTIR analysis for molecular structure of the rejuvenated age asphalt are shown in Figures 7(a&b) while Table 9 illustrates the most characteristic peaks of rejuvenated aged asphalt samples.

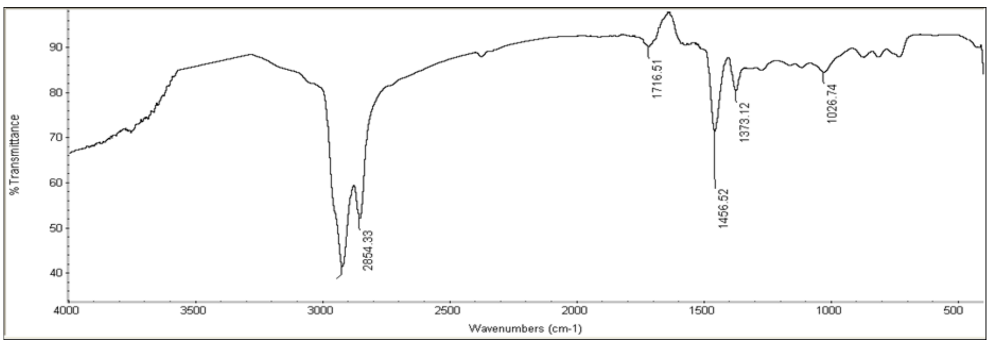

Figure 7a. FTIR spectrum of 34 % of VR1/blend of AC sample.

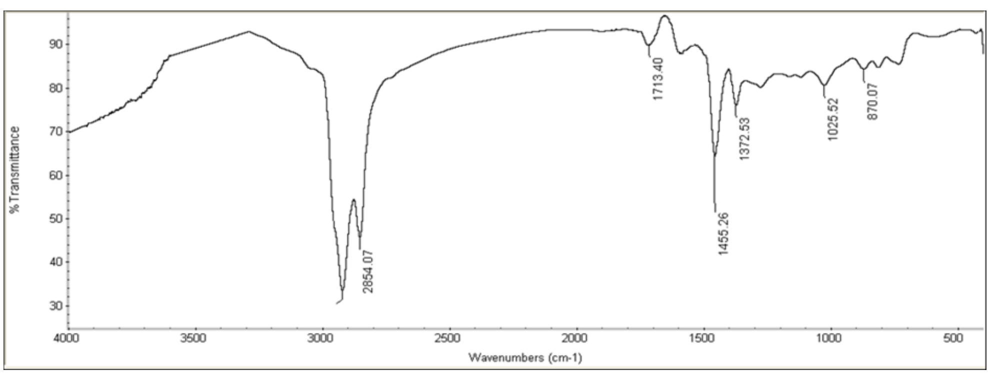

Figure 7b. FTIR Spectrum of 52 % of VR2 in blend of AC sample.

Table 9. The most characteristic peaks of rejuvenated aged asphalt sample.

Sample	The most important peaks	The assignments
34% of VR1 in rejuvenated aged AC	A strong band at 2861-2925 cm^{-1} A strong absorption band at 1379 cm^{-1} A strong absorption band at 1459 cm^{-1} A small absorption band at 874 cm^{-1} The peak at 1019 cm^{-1} vibration The peak at 1723 cm^{-1} vibration A strong band at 1111 cm^{-1}	C-H stretching vibration Stretching vibration of CH_3 Stretching vibration of CH_2 C=C bending ascribed to S=O stretching C=O stretching C-O stretching

Sample	The most important peaks	The assignments
52% of VR2 in rejuvenated aged AC	A strong band at 3436cm−1 A strong band at 2853-2921cm−1 A strong absorption band at 1374cm−1 A strong absorption band at 1455 cm−1 A small absorption band at 729 cm−1 The peak at 1610 cm-1 vibration	O–H stretching of OH group C-H stretching vibration Stretching vibration of CH_3 Stretching vibration of CH_2 C=C bending C=C stretching

From Figures 7(a& b) and Table 9, the analysis revealed that rejuvenated aged asphalt AC samples have nearly the same functional groups as virgin asphalt samples which confirms that VR samples are effective rejuvenators as they restore the FTIR spectrum of aged asphalt to their original states.

4.5. STEP 6. Determination of highest temperature at which the rejuvenated asphalt will stand safe using DSR according to AASHTO T 315

In this step, all asphalt samples are tested using DSR test. This test was conducted by a rheometer of model Smart Pave MCR 201, RheoCompass™, V1.20.496-Release to determine the highest temperature at which the rejuvenated samples will stand safe during their service life time as well the rutting factor (performance Grade). The results are illustrated in Table 10.

Table 10. Highest temperature at which all asphalt will stand safe.

Factor	Rutting factor |G*|/sin(delta)	Highest temperature (°C)
Virgin AC	1.23	64
Rejuvenated asphalt using VR1	1.01	64
Rejuvenated asphalt using VR2	1.16	64

The results revealed that the rejuvenated samples are safe at a temperature up to 64°C as the virgin one. All the obtained results revealed that the vacuum residue samples are successful rejuvenators for aged asphalt.

5. Conclusion

This research aims to help in sustainable development of Egypt by keeping the natural resources as long as possible among which is aged asphalt. Also, the research helps in producing a clean environment and helping in solving the problem created from waste paving disposal. To achieve the research objective, aged asphalt prepared at lab using both of RTFO and PAV representing short and long aging steps respectively. Aged asphalt is rejuvenated using two different types of rejuvenators namely VR.

The aged asphalt sample was characterized for both physical and chemical analysis in addition to FTIR and SEM. Aged asphalt is found to have lower penetration and higher kinematic and softening values. Also, it had lower saturate and aromatic fractions and higher resins and asphaltene contents. The two rejuvenators were characterized for physical and chemical tests. A blending chart was established to find the proper amount of both rejuvenators are required.34 & 52% of VR1 And VR2 respectively (by wt.) in a blend of aged asphalt and rejuvenators were found to be suitable for aged asphalt rejuvenation. The prepared rejuvenated asphalt was found to comply with Egyptian Standard limit for AC60/70.

Conflict of interest: *The authors declare that there is no conflict of interest.*

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