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Physical characterization of asphalt due to weathering using Scanning Electron Microscope

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Abstract —*The main objective of this study is to analyze physical characterization of recovered asphalt binder. Initially, job mix formula was prepared and was laid at site near Wazirabad bypass, Pakistan in 2016. The asphalt core samples were extracted after three years from the same site so that extracted bitumen properties can be compared with virgin bitumen properties. The feasibility of recovered bitumen for the use in flexible pavement was tested according to AASHTO M-20 standardsand SEM analysis. The results indicated a slight decrease in ductility, durability and overall strength of bitumen with varied angularity of threads.*

Keywords **–***Scanning Electron Microscope, Aging, Reclaimed Asphalt Pavements, Asphalt Mix properties, Bitumen, Microstructure, Federal Highway Administration*

I. INTRODUCTION

In today's world it is a common practice to add a small amount of recovered bitumen in construction of flexible pavements without altering properties such as stiffness and low temperature thermal cracking of the mix. The advantage of using recovered bitumen is that not only it is beneficial to the environment as most of the bitumen is recycled but it's also improve the properties such as rutting resistance and fatigue cracking. Also in today's world natural resources are depleting and using recovered bitumen not only is economical, but also it's proving to be very beneficial for preserving the natural resources for the future generation.
In order to determine the feasibility of the recovered bitumen, different tests can be performed to check and compare various properties with that of virgin bitumen. One such test is to do the microstructural characterization of bitumen using scanning electron microscope (SEM) in laboratory.

Scanning electron microscope (SEM) uses beams of electron to produce sharp images of the test sample. The beam of electrons interact with the test sample and as a result numerous signals are produced that contain information about composition of the sample and its topography. SEM uses raster scanning method to detect the signal which is than combined with the intensity of the detected signal and hence a sharp image is produced. Due to focused beam of electrons, secondary electron are emitted which are than detected by Ever Hart thornily detector. The type, nature and topography of the test sample determines how many secondary electrons are to be emitted. Scanning electron microscope can achieve resolution better than 1 nanometer.

SEM micrographs have three-dimensional appearance due to the very narrow beam of electrons which proves to be very useful in fully understanding the surface texture of the test sample.
A wide range of magnification is possible with SEM. The magnification of scanning electron microscope ranges from 10 times to 500,000 times, about 250 times the magnification limit of the best light microscopes. SEM can work entirely without any condenser or objective lenses.

Scanning electron microscope was used by Saeed S. Saliani (et.al,11 may 2019) to differentiate between virgin asphalt and reclaimed asphalt at microscopic level. In the test, virgin bitumen used was PG64-28 from Bitumar (Montreal, QC, Canada). The Rap used was obtained from Bauval (Montreal, QC, Canada).In case of virgin bitumen a fibril microstructure was formed because of presence of lighter components. Reclaimed asphalt, however, showed a much denser structure because of the stiffening of the microstructure of bitumen due to aging.
Peter Mikhailenko (et al, 2016) observed a sample of asphalt of PG-58-28 in scanning electron microscope. Sample was given a 10 to 20 sec time to set properly so that irregularities set and the fibrous structure becomes more visible.
The sample was observed at magnification of 1000x which provided clear results. He compared the images with the microstructure of an unaged asphalt binder and found that microstructure was very similar however the fibers were much more distinct.

In order to validate the results of the SEM analysis an oxidized sample of PG 58-28 was observed under scanning electron microscope and results were compared
Due to oxidation a clear modification of microstructure was observed. Fibers became denser and inter-wind oxidation was carried out by using an electron beam and due to this oxidation change in microstructure resonated with the microstructure of a naturally oxidized asphalt sample.

II. METHDOLOGY

The flow chart of methodology showing various steps involved in the research is given below in figure 1.

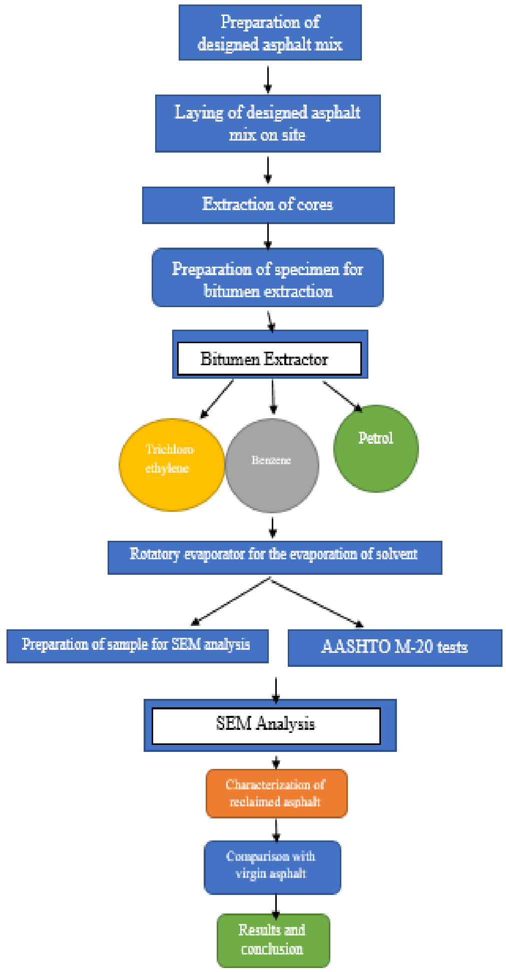


Figure 1. Research methodology flowchart

III. MATERIALS AND METHODS

Following materials and methods were adopted in following sequence for this research project as listed below:

1. Virgin bitumen (60/70 grade) tests were performed according to AASHTO M-20 and M-320standards in laboratory.Thevalues for the properties of bitumen, were also determined for in accordance with ASTM D-7,D-5,D-36,D-113,D-92,D-4 and performance grading tests.
2. Cores were extracted from atrial section of 30m length on north bound of G.T. road (two lane divided highway with TST on edges both sides) as shown in figure 3. The road waslaid in year 2016 up to layer of asphaltic wearing coursein accordance with the specification of AASHTO MS-2.

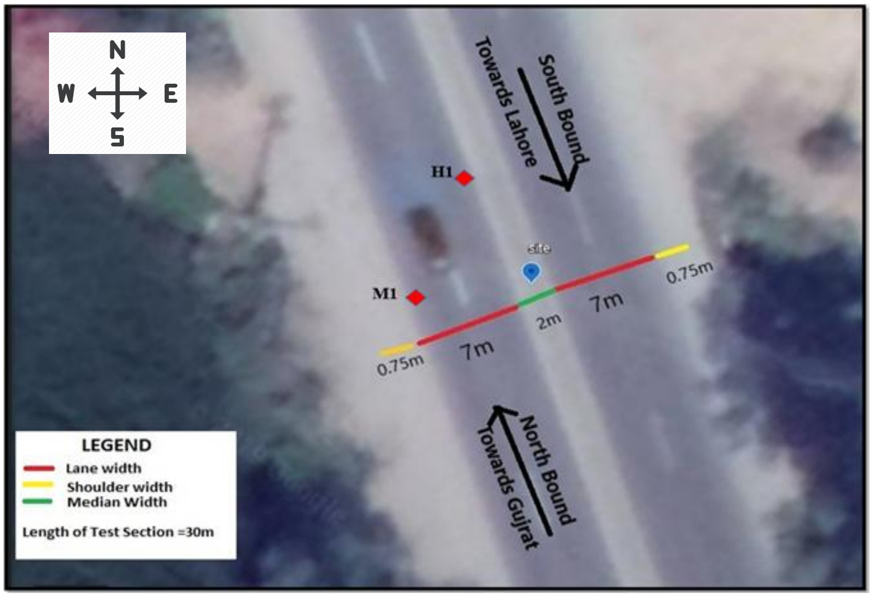


Figure 2. Site Location Map

3. Extracted Core Sample from the site are shown in figure 3 below

Figure 3. Extracted cores

4. The images of site from where cores were extracted are presented in figure 4.

Figure 4. Site pictures

5. The pavement was subjected to environmental changes, traffic loading and weathering effects until 2019. Summary of which is shown in table 1, 2 and 3.

Table 1. Annual daily traffic (Communication and works department Punjab)

Vehicle	No.s (2017)	No.s (2018)	No.s (2019)
AD Vehicles	129	134	140
Motor cycles & Rickshaws	2825	2938	3056
Cars and Pickups	690	718	746
Mini Buses and Wagons	165	172	178
Buses and Flying Coaches	35	36	38
Trucks 2-Axle	93	97	101
Trucks 3-Axle	29	30	31
Tractor	21	22	23
Tractor Trollies 3-Axle	169	176	183
Tractor Trollies 4-Axle	0	0	0
Passenger Car Equivalent (P.C.U.s)	6004	6244	6474

Table 2. Monthly average temperature variation table for site (Wazirabad)

Weather	Month											
	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
Avg. Temperature in 2019 (°C)	12.3	15	20.3	26	31.2	33.9	31.4	30.1	29.5	25.4	18.6	13.5
Avg. Temperature in 2018 (°C)	11.9	13.6	20	27	30.8	34.1	29	32.3	27.3	25.2	19.5	13.4
Avg. Temperature in 2017(°C)	12.4	14.1	21.5	27.5	30.9	36	32.2	34.5	25.1	24.7	17.8	12.9

There is a difference of 176mm in precipitation while in temperature variation throughout the year is 21.6 °C.

Table 3. Annual rainfall data for site (Wazirabad)

Months	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
Rainfall (mm) in 2019	35	34	32	19	18	45	159	183	74	10	7	16
Rainfall (mm) in 2018	34	36	32	20	21	38	170	179	95	9	7	9
Rainfall (mm) in 2017	36	33	33	21	19	47	145	189	98	11	8	13

6. Bitumen was extracted from the aged core samples in the lab using solvent centrifuge extractor according to ASTM D 8159. Rotatory evaporator was used to evaporate solvent (Tri-Chloro-Ethylne) from the extracted bitumen according to ASTM D5404.

7. Specific gravity, penetration, solubility, ductility, flashpoint and softening point of extracted bitumen were determined according to the ASTM D-7, D-5, D-4, D-113, D-92 and D-36 respectively.

8. Both the samples of extracted bitumen and virgin bitumen (Grade 60/70) were analyzed in laboratory using scanning electron microscopy test.

9. Effect of weathering, traffic loading and precipitation on microstructure of binder were determined.

3.1. Scanning Electron Microscope (SEM) Test

It is a type of microscope that uses beams of electron to produce sharp images of the test sample. The beam of electrons interact with the test sample and as a result numerous signals are produced that contain information about composition of the sample and its topography. SEM uses raster scanning method to detect the signal which is than combined with the intensity of the detected signal and hence a sharp image is produced.
Scanning electron microscope can achieve resolution better than 1 nanometer. SEM analysis is performed on the test sample for the characterization of reclaimed asphalt. In characterization of reclaimed asphalt, structure of asphalt, texture of asphalt, angularity of asphalt and surface topology are observed at the microscopic level. The results obtained from SEM analysis of reclaimed asphalt are compared with that of virgin asphalt.
There are two methods to analyze SEM images:

- **Visual inspection**

In visual inspection observer records shape of different patterns, continuity and discontinuity of the patterns, shape of microfibers, topography and visual appearance of the surface. The accuracy of the results obtained from visual inspection of the images depends entirely on the experience of the observer.

- **Image Processing**

In this method an image processing software is used to analyze SEM images. With the help of this software the diameters of different microfibers of both reclaimed asphalt and virgin asphalt are measured and a graph is plotted to compare the diameters of fibers of both the samples.

IV. RESULTS AND DISCUSSIONS

1. The results of AASHTO M-20 and M-320 on virgin bitumen are shown in the table 4 and 5.

Table 4. Specified limits for bitumen used in asphalt mix

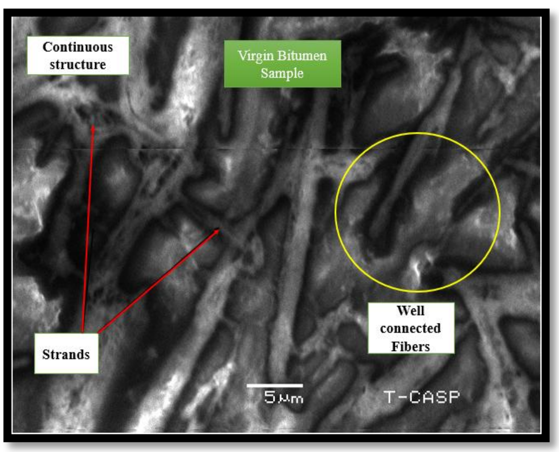
Test	Units	Virgin binder Values	Limits	Methodology
Specific Gravity	N.A.	1.02	1.01 - 1.06	ASTM D-7
Penetration at 25 °C	0.1 mm	65	60-70	ASTM D-5
Softening Point	°C	48	46-56	ASTM D-36
Ductility	Cm	100+	100 min	ASTM D-113
Flash Point	°C	307	230 C min	ASTM D-92
Solubility in Tri Chloro ethylene	%	99.8	99-99.5	ASTM D-4

Table 5. PG grading of virgin bitumen

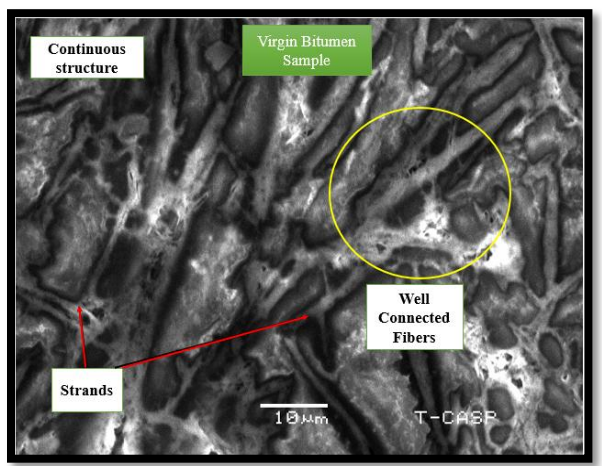
Performance Grading property	Virgin binder values - PG Grading
Original Asphalt Binder (High Temperature)	65.1
RTFO (High Temperature °C)	65.4
BBR (Low Temperature °C)	-22
Performance Grade	64-22
Continuous grade range °C	88.3
Viscosity (Pa s) at 135°C	0.446
VTS	-3.381
Mass Change %	0.056

2. SEM images of virgin bitumen at two different resolutions (2700 and 1500) and wavelengths (5µm and 10µm) are shown below in figures 5.

(a). 2700 and 5µm



(b). 1500 and 10µm



(c). 750 and 20µm

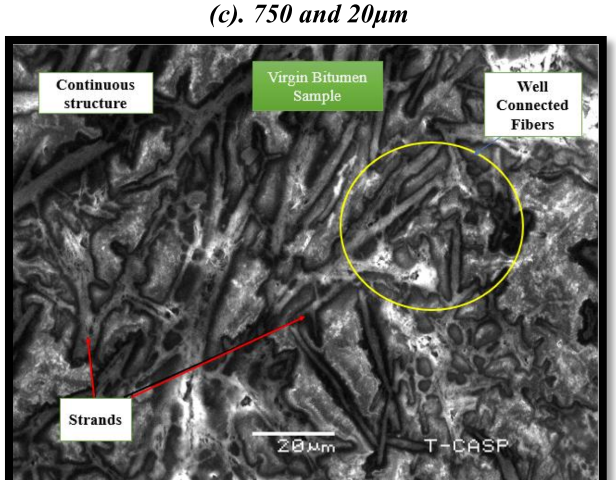


Figure 5. SEM images of virgin bitumen sample

3. In the following histogram shown in figure 6, the variation in diameters of threads of virgin bitumen can be seen. The diameter varied between 1-7µm.

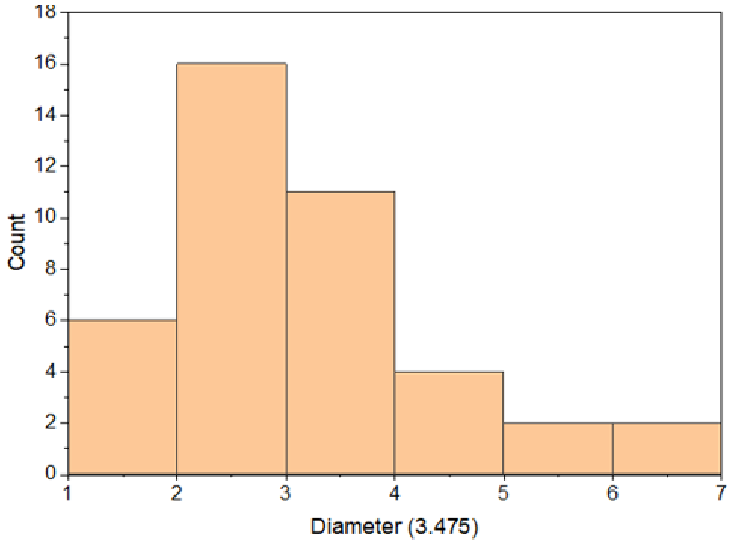


Figure 6. Histogram showing variation in diameters of threads of virgin bitumen

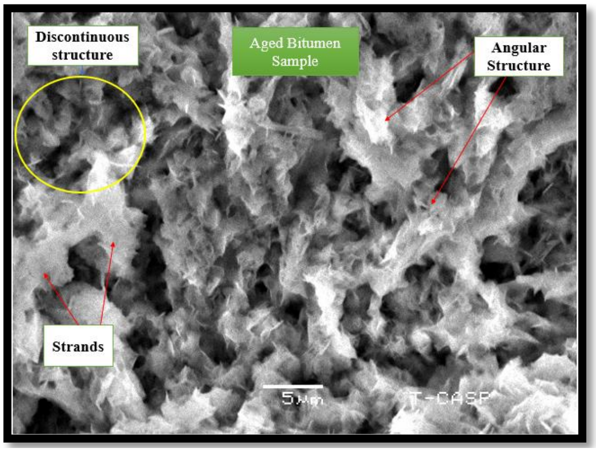
4. Following table 6 presents obtained aged bitumen characteristics.

Table 6. Aged bitumen characteristics

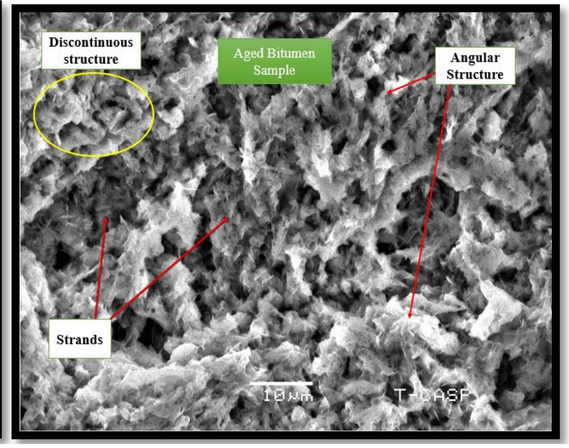
Bitumen characteristics	Aged Bitumen
Specific gravity	1.024
Penetration test	37.7 mm
Softening point	59.5 °C
Ductility	12 (cm)
Flash point	286 °C
Solubility in TCE	99.7%

5. Images obtained as a result of SEM analysis of aged bitumen at different resolutions and wavelengths are shown below in figure 7.

(a). 2700 and 5µm



(b). 1500 and 10µm



(c). 750 and 20µm

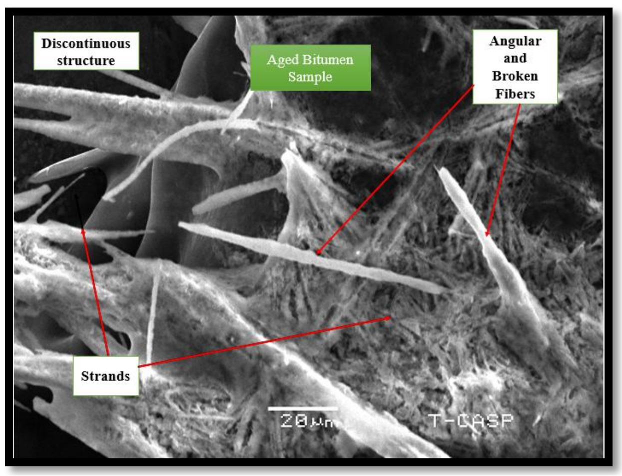


Figure 7. Image of aged bitumen sample

6. From histogram below in figure 8, it can see that in microstructure of aged bitumen, the range of diameter of thread is from 0 to 25µm.

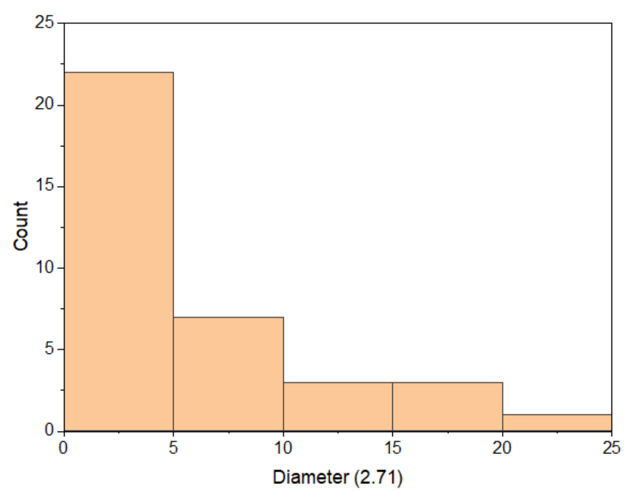


Figure 8. Histogram showing variation in diameters of threads of aged bitumen

7. From the table 6 above, it can be seen that with aging bitumen has become more plastic and rigid because of which the penetration and ductility has decreased, softening point and specific gravity has increased.
9. On comparing the images obtained as a result of SEM analysis of aged and virgin bitumen, it can be seen that there is an evolution in fiber structure of aged bitumen as compared to virgin bitumen. The size of fibers became smaller and more angular in aged bitumen as compared to virgin bitumen.This is due to long term aging under heavy traffic.

In terms of topography, the microstructure of aged bitumen is exhibiting a rough surface with a number of peaks of the micro strands. Whereas, in case of virgin bitumen a smooth surface can clearly be seen with little to no peaks.

An image processing software is used to analyze the microstructures of aged bitumen and virgin bitumen from their respective SEM images.

Following table 14 shows average sizes of threads in micrometers of aged and virgin bitumen. From this it can be seen that average size of threads in aged sample is greater than that of threads in virgin sample.

Table 14. Comparison of average thread size of virgin and aged bitumen

Size	Length(μm)	Virgin	Aged
20 μm	Maximum	28.46	34.56
	Minimum	11.14	10.54
	Average	19.8	22.25

Diameters of their threads are measured at random and following graph in the figure 16 were obtained.

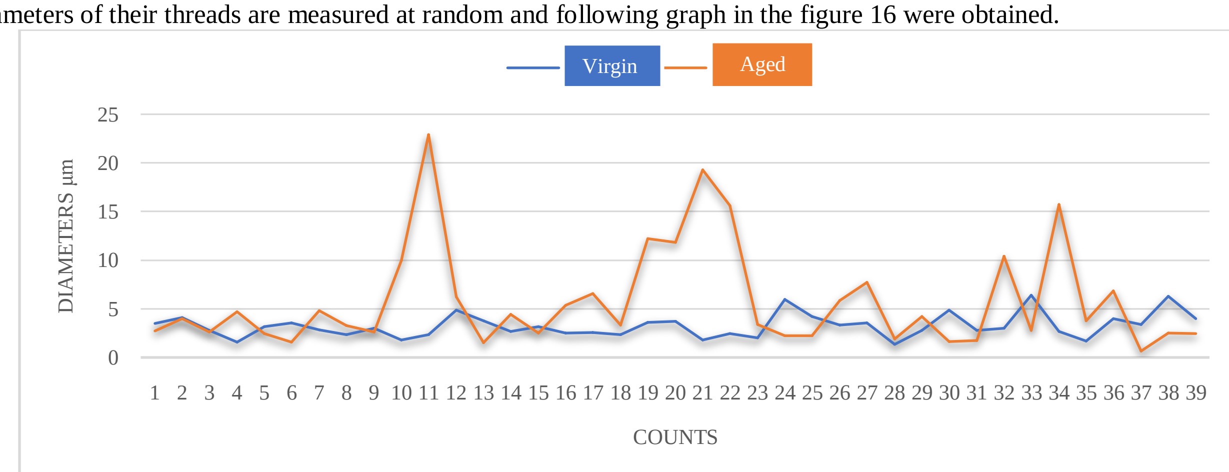


Figure 9. Comparison of diameters of threads of virgin and reclaimed asphalt

In this graph, blue line is representing the diameter of threads of the virgin bitumen and orange line is representing the diameter of threads of the aged bitumen. It can be seen that threads in the virgin bitumen have almost equal diameter with little variation. This is because loading and weathering action. From this it can be seen that the virgin bitumen have uniform and a smooth structure. So, the strength of aged bitumen is comparatively less than that of virgin bitumen.

V. CONCLUSIONS

The Difference between test values of virgin and aged bitumen is clearly visible from this research. It can be concluded that pavement has deteriorated over time due to oxidative aging, moisture effects and traffic loading.

From scanning electron microscope analysis, change in microstructure of reclaimed bitumen as compared to that of virgin bitumen due to long term aging of highway under heavy loading and weathering of the HMA layer can be seen clearly .In microstructure of aged bitumen, strands are all broken which indicates a decrease in elasticity and increase in plasticity of the individual strands, which is due to long term temperature variation and climate changes. However, in virgin bitumen the strands are well connected and have high elasticity.Bitumen has becomes brittle due to aging which leads to cracking. Moreover binding properties of aged bitumen have decreased. But these binding properties can be artificially enhanced by mixing additives and bituminous rejuvenators with aged bitumen.

VI. ACKNOWLEDGEMENTS

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VII. REFERNCES

1. Tarar, A.M., Khan, A.H., Zia, R., Inam, A. (2019). Changes in the rheological characteristics of asphalt binders modified with soybean-derived materials. International Journal of Pavement Engineering. doi: 10.1080/10298436.2019.1600690
2. Stokes, Debbie J. (2008). Principles and Practice of Variable Pressure Environmental Scanning Electron Microscopy (VP-ESEM). Chichester: John Wiley & Sons. ISBN 978-0470758748.
3. McMullan, D. (2006). Scanning electron microscopy 1928–196". Scanning. 17 (3): 175-185. doi:10.1002/sca.4950170309. PMC 2496789.
4. A Noferini, L., Simone, A., Sangiorgi, C., Mazzotta, F. (2017). Investigation on performances of asphalt mixtures made with Reclaimed Asphalt Pavement: Effects of interaction between virgin and RAP bitumen. International Journal of Pavement Research and Technology 10, pp.322–332
5. Omranian, S.R., Golchin, B. (2015). A Review on the Effects of Aging on Properties of Asphalt Binders and Mixtures. Caspian Journal of Applied Sciences Research 4(6), pp.15-34
6. Gao, Y., Gu, F., Zhao, Y. (2013). Thermal oxidative aging characterization of SBS modified asphalt. Journal of Wuhan University of Technology-Mater. Sci. Ed. [J], 28, pp.88-91.
7. Widayanti, A., Soemitro, R.A., Ekaputril, J.J., Suprayitno, H. (2018). Characterization of Reclaimed Asphalt Pavement (RAP) as a road pavement material (National Road Waru, Sidoarjo). MATEC Web of Conferences 181, 05001, ISTSDC 2017
8. Saeed, S.S., Carter, A., Baaj, A., Mikhailenko, P. (2019). Characterization of Recovered Bitumen from Coarse and Fine Reclaimed Asphalt Pavement Particles.
9. Wen, H. Lu, J. and VanReken, T.M.(2014) .Modeling the effects on energy and carbon dioxide from the use of recycled asphalt pavement in hot mix asphalt, International Journal of Sustainable Transportation, vol. 8, no. 4, pp. 249–261.
10. Yu,X. and Li,Y. (2010). Optimal percentage of reclaimed asphalt pavement in central plant hot recycling mixture, Journal of Wuhan University of Technology-Mater. Sci. Ed., vol. 25, no. 4, pp. 659–662.
11. Planche, J.P. (2014). Insights into binder chemistry, microstructure, properties usage in the real world," in Asphalt Pavements, 0 vols., CRC Press, pp. 3713–20.38
12. Yang, Z., Zhuang, G., Wei, X., Wei, J., Yu, H., Xu, W. (2018). Quantitative Analysis of the Blending Degree of Virgin and RAP Binders in Recycled Asphalt Mixtures with a High RAP Content. MDPI Applied Sciences. Appl. Sci. 2018, 8, 2668; doi:10.3390/app8122668

