PROJECT REPORT ON

"FEASIBILITY STUDY FOR USE OF BOTTOM RESIDUE TO MIX WITH BITUMEN/BITUMINOUS MIXES/RAP MATERIAL THROUGH LABORATORY EVALUATION"

FLEXIBLE PAVEMENT DIVISION

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Central Road Research Institute (CRRI), New Delhi can accept no responsibility for the inappropriate use of this report. Engineering judgment and experiences must be used to properly utilize the principles and guidelines contained in the report, taking into account available equipment, materials provided by client and conditions. All reasonable care has been taken in the preparation of this report; however, the CRRI can accept no responsibility for the consequences of any inaccuracies, which it may contain. The report pertains to the laboratory evaluation of bitumen and bituminous mixes by using the samples of bottom residue supplied by client. However, fresh evaluation may be needed for various types of bituminous layers and different sources of bottom residue, bitumen and aggregate.

ACKNOWLEDGEMENT

The project entitled **"Feasibility study for use of Bottom Residue to mix with bitumen/bituminous mixes/RAP material etc., through laboratory evaluation"** was sponsored by Petroleum Re- Refiners Association of India(PRAI) to Central Road Research Institute (CRRI), New Delhi. We are grateful to the sponsor. The support rendered by all staff members of FP division for completion of this project is acknowledged.

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1. INTRODUCTION

The construction of road surfaces is associated with a significant consumption of natural resources, including mineral aggregates and crude oil, as well as the emission of hazardous compounds into the atmosphere during the process of asphalt mix production. Through the use of waste materials and innovative technologies, there is a chance to make road construction not only environmentally friendly but also less cost-intensive, while ensuring the appropriate durability of the pavement as well as comfort and safety for users. Despite asphalt being an integral part of human society for over 6,000 years and modern society for over a century, many complexities of asphalt pavement still elude researchers. Increase of road traffic has proportionately increased the amount of engine oil waste. The use of vacuum distilled residues from the re-refining of used motor oils as blend stock in paving grade bitumen has occurred in isolated markets in North America for more than 30 years. Recently in the United States the increasing need for low stiffness bitumen for use in high binder replacement mixtures, coupled with economic considerations, has led to an expanding market for these products. Re-refined vacuum tower bottoms (RVTBs) are the non-distillable fraction from the re-refining of used engine oils. RVTBs have been blended with paving grade binders to improve the low temperature properties for over 20 years. In the past few years, there has been increased interest in the properties of asphalt binders containing RVTBs and the effect on HMA performance. In India used waste engine oil is available from two major sources, generator or transformer oils and road motor vehicles. The residue after re-refining these oils is called as Bottom Residue in this study.

Rejuvenating agents, which restore the chemical structure of aged asphalt by providing lost aromatic constituents and reducing the overall viscosity of the binder, are used for improving the properties of mix asphalt with RAP. One of the potential municipal waste materials that can be used to rejuvenate RAP is waste engine oil (WEO) from cars. The structure of WEO resembles the molecular structures of asphalt with sufficient aromatic content, which leads to coherent bonding by altering the constituents and rejuvenating the aged asphalt Thus more used motor oil is being re-refined creating more of the residual by-product. In India, adequate research has not been done on its use in bituminous mixes. Also by incinerating the bottom residue will result in high level air pollution. Hence there is an urgent need to find its use in road construction. Several research has been going on in various countries for use of the distillate residue by re refining of waste engine oil. This feasibility study is carried out for usage of bottom residue of waste engine oil in the bituminous mixtures. The General secretary, Petroleum Re-Refiners Association of India, Delhi, had earlier approached CRRI, through his letter by mail September, 2019, requesting to take up a study on feasibility of usage bottom residues in the bituminous mixes. Further correspondences and follow up by both Petroleum Re-Refiners Association of India and CRRI led to arrive at a mutual consent for taking up a technical assignment by CRRI as research study. CRRI vide its letter No. CRRI/FPD/PRAI/846, dated 30thSeptember, 2019 communicated its acceptance and accordingly proceeded with the requirements. Petroleum Re-Refiners Association of India confirmed the assignment in the first week of December, 2021 and accordingly subsequent course of action was planned. The Project was awarded by Petroleum Re-Refiners Association of India to CSIR-CRRI with a requirement of providing the feasibility of usage bottom residues in the bituminous mixes.

2. OBJECTIVES and SCOPE OF WORK

The main objective of this study is to find the feasibility study for use of bottom residue to mix with bitumen mixture through laboratory evaluation.

Scope:

- Rheological/Physical properties of bitumen
- Chemical Characterization of bitumen by using SARA Analyzer
- Optimization of bottom residue in bitumen
- Evaluation of the bituminous mix (Both controlled and bottom residue modified mix)

3. LABORATORY TESTS

3.1 Aggregates: The Quartzite aggregates were procured and characterized in the lab. Coarse and fine aggregates procured are shown in **Photos-1**. The properties of aggregates used in this study were tested as per BIS specifications and confirmed to MoRTH specification. Test results are given in **Table-1**.

Photo-1: Aggregate used for this study

Table-1 Properties of Aggregates used for the study

Property	Test	Specification as per IS:2386 ,1997	Limits as per MoRTH specification	Value Obtained
Particle Shape	Combined Flakiness and Elongation Indices	IS:2386 ,1997part I	Max 35%	46%
Impact Test	Aggregate Impact Value	IS:2386 ,1997part III	Max 30%	9%
Water Absorption	Combined Water absorption	IS:2386 ,1997part III	Max 2%	0. 5%
Specific gravity	Bulk Specific gravity (coarse)	IS:2386 ,1997part III		2.790
Specific gravity	Bulk Specific gravity (fine)	IS:2386 ,1997part III		2.830
Stripping of aggregates	Stripping of aggregates	IS:6241,1998	Min. retained coating, 95%	98%

3.2 Bitumen: Viscosity grade bitumen was used for this study. VG10, VG30 and VG40 which have been tested in the lab as per BIS codes. Some basic tests were conducted and rheological properties of binder were also studied. To know the suitability of the bitumen, various tests has been carried out.

The specific gravity of the bitumen is the ratio of the mass of the substance given to the same volume of water, the temperature of the two being 27°C.In general, pure bitumen's specific gravity is between 0.97 and 1.02. Penetration test has been carried out at 25°C temperature. The Ductility test is used to evaluate the bitumen's adhesive property and stretching capacity. The ductility is described as the range to which a normal bitumen briquette can be extended in centimeters before the thread breaks. The test is performed at a pull rate of 50 mm per minute at 27 ° C. Kinematic viscosity is carried for different binders at 135°C, cSt. Testing of bitumen for specific gravity, penetration, ductility, viscosity and softening point are shown in **Photos 2,3,4,5&6** respectively. Results of test conducted on different bitumen are given in **Table-2.**

Photo-2: Specific gravity testing of bitumen

Photo-3: Penetration testing of bitumen

Photo-4: Ductility testing of bitumen

Photo-5: Viscosity testing of bitumen

The softening point is the temperature at which the bitumen reaches a specific degree of softening under the given condition.The softening point used in pavements for different bitumen grades varies between 35 ° C and 70 ° C. Higher softening point indicates lesser temperature susceptibility which can be used in warmer places.Some Basic properties of neat bitumen which were conducted are given in the table below:

Photo-6: Softening point testing of bitumen

Table 2: Results of Tests Conducted on Neat Bitumen

Property Evaluated	VG10		VG30		VG40	
	Result	Spec*	Result	Spec*	Result	Spec*
Penetration @ 25°C, 100 g, 5 s, 0.1 mm	85	Min 80	51	Min 45	37	Min 35
Softening Point, °C	47	Min 40	59	Min 47	61	Min 50
Absolute viscosity at 60°C, Poises	1145	1000±20 %	3191	3000±20 %	4210	4000±20 %
Kinematic viscosity at 135°C, cSt,	250	Min 250	500	Min 350	800	Min 400

3.3 Bottom Residue: The oil removed from the vehicle during a routine oil change is the used engine oil or waste engine oil. This oil is then subject to further refining to obtain good quality lubricants. The process of distillation is called Re-Refining. The structure of used engine oil or waste engine oil resembles the molecular structures of asphalt with sufficient aromatic content, which leads to coherent bonding by altering the constituents and rejuvenating the aged asphalt The non distillable fraction from re-refining of such used/waste engine oils is called Bottom Residue. This study was taken up to find whether bottom residue can be used in bitumen and bituminous mixes. For these aggregates and bitumen were procured and tested as per specifications in the IS standards in the laboratory. The bottom residue sample was supplied by the client. Some tests were conducted on this material and on the blends of bitumen with bottom residue. The sample bottom residue shown in the **Photo-7** was similar to bitumen. It was semi-solid(just not liquid) at room temperature, soft and black in colour,

Photo-7: Bottom Residue Sample

3.4 Chemical Characterization for bitumen and bottom residue

SARA analysis is caaried out for the different type of binders and bottom residue.SARA is an analysis method that divides crude oil components according to their polarizability and polarity.

Procedure

1. Firstly, prepare the solution by dissolving 100mg of bitumen sample in 10ml DCM solution (3 solutions samples were prepared for reference i.e.(VG10, VG-30, VG-40). The prepared solution is injected into the chromarods which consists of 10 rods.
2. Then take the chroma roads carefully and put it in oven for 3mins at 60˚C, this time is known as oven dry time.
3. After that take out the chroma roads and put it in a development jar with 70ml Hexane solution for 30mins, this time is known as development time.
4. After 30 mins take out the sample and thoroughly dry the development jar and repeat the step
5. Then place the chroma roads in a development jar containing 56 ml toluene and 14ml Hexane (80% and 20% of 70 ml respectively) for 10 mins.
6. Take out the chroma roads and dry the development jar and repeat the step 4.
7. Then place the sample in a development jar containing 66.5ml DCM and 3.5ml Methanol (95% and 5% respectively) for 2 mins.
8. After this again put the chroma roads in oven for 3 mins at 60˚C.
9. Now scan the roads in Fire ionization detector (FID) iatroscan MK-5.

Photo-8: SARA Analyzer

SARA analyzer is shown in **Photo-8** above.
The four major components were evaluated using SARA analyzer. The results are given in the **Table-3** below:

TABLE-3: SARA Analysis of Neat Bitumen and Bottom Residue

Material	Saturates	Aromatics	Resins	Asphaltenes
Bottom Residue (neat)	14.94	61.76	18.37	4.91
Vg 10	12.96	50.48	30.23	6.31
Vg 30	10.31	61.06	21.23	7.39

The above table indicates that the Bottom Residue is having similar proportion of the four major components; (SATURATES, AROMATICS, RESINS, ASPHALTENES).

4. BITUMEN BLEND WITH BOTTOM RESIDUE – METHODOLOGY AND PROPERTIES

Neat bitumen VG30 and the bottom residue provided by client were taken for the study. Bitumen was heated up to 150^{o}C in hot air oven till it became pourable. Four beakers of one liter capacity were taken. After tare weight of these beakers 500g of hot bitumen was weighed in each. The bottom residue at ambient temperature was then added to hot bitumen at 2%, 4%, 6% by weight of bitumen in three beakers. That is; 10g, 20g and 30g respectively. Each of these mix was slowly stirred manually for approximately five minutes to form uniform blend and left overnight undisturbed. Next day all four beakers were heated up to 150^{o}C in hot air oven till molten stage and stirred well. The pure bitumen and the blends were ready for testing and moulds filled for penetration, softening point, viscosity and DSR and tested as per standards. The blending is shown in **Photo-9** and method of preparation of blend is shown in flow chart in **Figure-1**

Photo-9: Bitumen and addition of Bottom Residue

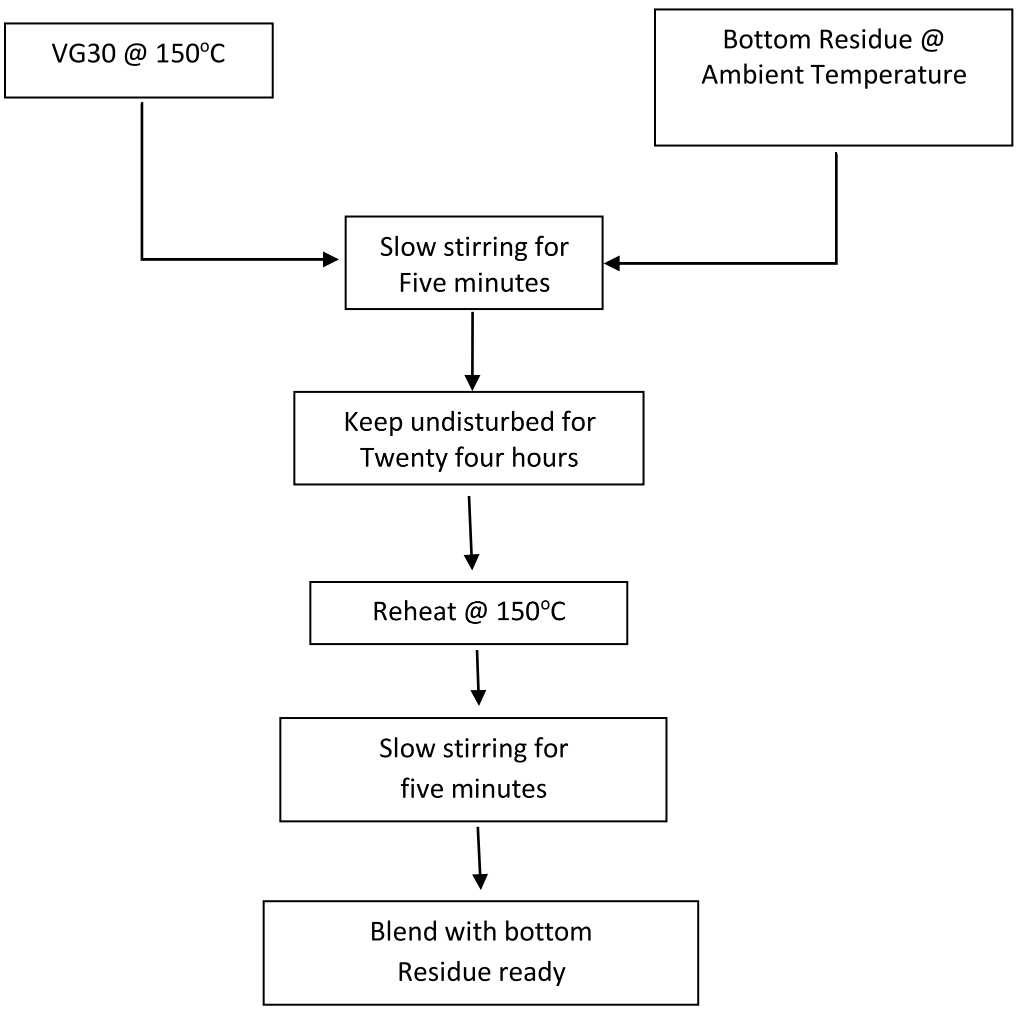

Figure 1: Flow Chart for Preparation of Blend

The blends were subjected to basic tests and the test results are in the **Table-4** below.

Table-4: Properties of Blend with VG 30

Sample ⇨ Properties ⇩	Vg 30	Vg 30+2%	Vg 30+4%	Vg 30+6%	Vg 30+8%	Vg 30+10%
Penetration at 25°C, 100 g, 5 s, 0.1 mm, Min	51	62	70	82.3	89	102
viscosity at 135°C, cP, Min	500	475	450	400	310	250
Softening point (R&B), °C, Min	59.6	54	52.6	48.4	43.1	38

For obtaining VG10 from VG30 7.5% to 8% Bottom Residue can be added. Trend shows increase in penetration with increase in the dosage of bottom residue in **Figure-2**.Viscosity is decreasing with increasing dosage of addition of Bottom Residue. Graphical representation in **Figure-3** shows that on adding near about 10% Bottom Residue to VG30 it softens down to VG10.

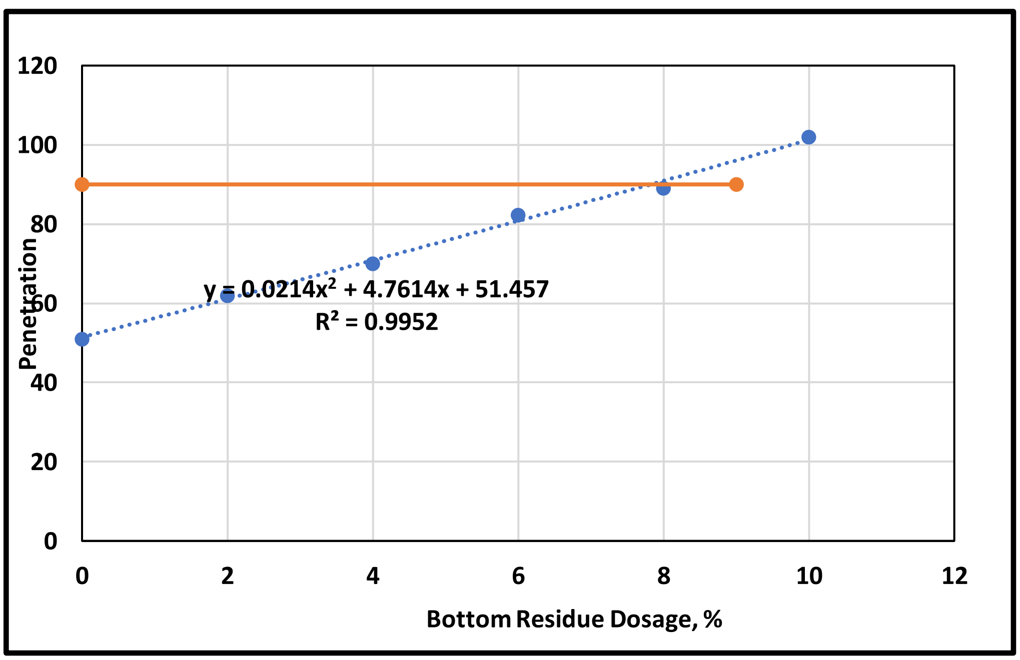

Figure -2 : Penetration Vs Dosage of Bottom Residue

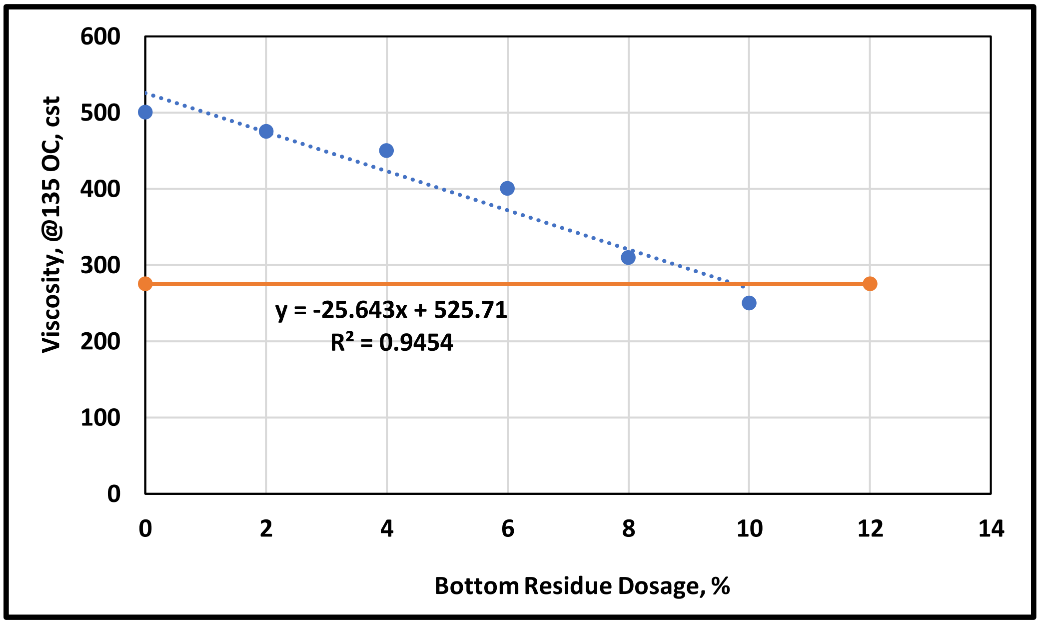

Figure -3 : Viscosity Vs Dosage of Bottom Residue

Increasing dose of Bottom Residue in VG30 bitumen there is decrease in softening point of blend. The graph in **Figure-4** shows that VG10 grade is obtained by addition of 8% to 10% (approx. 9%) of Bottom Residue to VG30

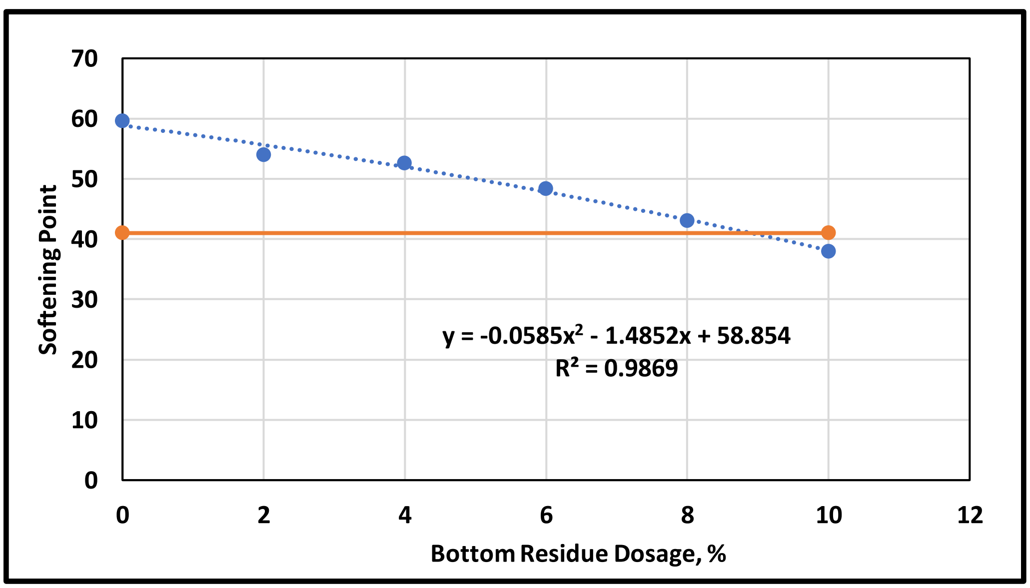

Figure -4 : Softening Point Vs Dosage of Bottom Residue

5. BINDER RHEOLOGICAL PARAMETERS

Dynamic shear rheometer (DSR) is used for evaluating bituminous binders whose performance depends on temperature and loading frequency. **Photo-10** shows the dynamic shear rheometer used in the current study.A thin bitumen film sandwiched between two parallel round plates is subjected to oscillatory stress at selected temperature and frequency. The bottom plate is fixed and the top plate oscillates at the particular frequency. For a given set of frequency and temperature, two key parameters: complex shear modulus (G*) and phase angle (δ), are measured. Complex modulus is a measure of the total resistance of a visco-elastic material to deformation when continuously sheared. Complex modulus is computed using Eq (1). Phase angle (δ) is the lag between the applied shear stress and the resulting shear strain.

Photo-10 : Dynamic Shear Rheometer

$$\text{ComplexModulus, } G^* = (\tau_{max} - \tau_{min}) / (\gamma_{max} - \gamma_{min}) \quad (1)$$

Where, τmax and τmin are the maximum and minimum shear stresses respectively, γ_{max} and γ_{min} are the maximum and minimum shear strains respectively. Test was carried out as per ASTM D7175-08 (2008) on the neat VG 10, VG 30 and blends prepared with VG 30 and Bottom Residue at different percentage. Test result for complex modulus is given in **Table-5**.

Table 5: Complex Modulus of Neat VG 10, VG 30and Blends with Bottom Residue

Sample	|G*|/sin(delta), kPa
VG 30	6.654
VG 10	2.351
vg 30+2%	4.597
vg 30+4%	4.415
vg 30+6%	3.833
vg 30+8%	2.64
vg 30+10%	2.286

Complex Modulus has been observed to have reduced from 6.6 MPa for neat VG30 to 2.6 MPa by increasing dosage of Bottom Residue which is almost the same as that of VG10 neat bitumen. This indicates the softening of hard binder/ bitumen to a softer grade as shown in **Figure-5**.

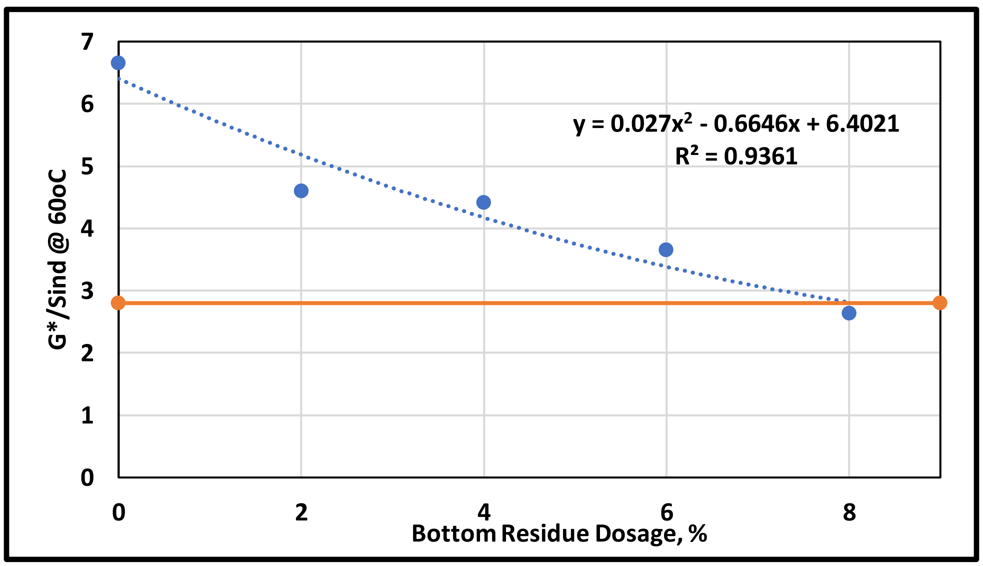

Figure-5: Complex Modulus Vs Dosages Of Bottom Residue

For rutting resistance of binders, high complex modulus (G*) value and low phase angle (δ) values are desirable. Higher G* values represent stiffer binders that are more resistant to rutting and lower δ values suggest greater elasticity of the binders with small plastic deformation. Thus, higher G*/sinδ signifies more resistance to rutting by the binder (SP-1, 2003). The recommendations of superpave binder specifications (SP-1, 2003) for satisfactory rutting performance of mixes are given in **Table 6**.

Table 6: Performance Grading Specifications for G*/sinδ(SP-1, 2003)

Binder Condition	Parameter	Specification
Unaged binder	G*/sinδ	≥ 1.0 kPa
RTFO residue	G*/sinδ	≥ 2.2 kPa

6. SEPARATION TEST

The separation of modifier and bitumen during hot storage is evaluated by comparing the ring and ball softening point of the top and bottom samples taken from a conditioned, sealed tube of polymer modified bitumen. The conditioning consist of placing a sealed tube of modified bitumen in a vertical position at 163±5°C in an oven for a period of 48 hours. It provides a reference for determining the relative separation properties between different types of bitumen modifiers and their respective bitumen. Such modified bitumen's relative stability to separation under storage in static condition is determined in heated oven storage without agitation

Photo-11: Separation Test Set up

Separation test set up is shown in **Photo-11** and test results are given in **Table-7**.

Table -7: Result of Separation Test

Sample	Softening Point, °C		Difference	Limit Max.
	Top	Bottom		IS 15462
VG 30 + 2% Bottom Residue	54.4	54.5	0.1	3
VG 30 + 4% Bottom Residue	53.1	53.4	0.3	3
VG 30 + 6% Bottom Residue	48.8	49.0	0.2	3

The above table shows that there is no separation of bottom residue from the blend as the results are with in the range as per IS 15462 -2019.

7. BITUMINOUS CONCRETE MIX – PREPARATION AND PROPERTIES

The bituminous mix for bituminous concrete mixes were prepared by Marshall method. BC grade I as per MoRTH specification was followed. The mid gradation was used for aggregates as shown in **Table-8**. Bitumen of viscosity grade VG 10 and VG30 were used. Bitumen content for BC was 5.2% by mass of mix. Cylindrical specimens of 100 mm diameter were prepared by mixing aggregates of different fractions and binder in appropriate quantities. The specimens were compacted by applying 75-blow Marshall compaction on both faces of the specimen and kept aside for 24 hours after which it is de- moulded.

Table-8: Aggregate gradation for BC grade I as per MoRTH, V Edition

	Cumulative % by weight of total aggregate passing			
Sieve size	Lower limit	Upper limit	Mid value	% passing
mm	%	%	%	%
26.5	100	100	100	100
19.0	90	100	95	95
13.2	59	79	69	69
9.5	52	72	62	62
4.75	35	55	45	45
2.36	28	44	36	36
1.18	20	34	27	27
0.600	15	27	21	21
0.300	10	20	15	15
0.150	5	13	9	9
0.075	2	8	5	5
Bitumen content % by mass of total mix, minimum				5.2

Selection of Binders: In India, VG30 binder is generally used for different bituminous layers (MoRTH 2013). Indian Roads Congress (IRC:111, 2009) suggests VG30 binder for dense bituminous mixes for climatic conditions defined by "minimum daily average air temperature more than – 10^0C and maximum daily average air temperature in excess of 30°C". IRC:111 (2009) recommends VG40 binder or modified bitumen of equivalent stiffness for project locations for which the commercial traffic is in excess of 2000 commercial vehicles per day per lane and with the maximum daily average temperature in excess of 40°C. Properties of Mix: The Marshallspecimen after de-mouldingare ready for testing. Before subjecting to test height and diameter of the specimen are measured. For each test three specimen are prepared. Tests to measure Density, Stability and Indirect tensile strength were conducted.

Indirect Tensile Strength

The indirect tensile strength test was performed as per ASTM D 6931-12 (2012) on cylindrical samples compacted at 4% and 7% air voids. The test was conducted by loading the specimen along its diametral axis at a fixed deformation rate of 51 mm/min until failure. Failure is defined as the condition after which there is no increase in load. The maximum load sustained by the specimen was used to calculate the indirect tensile strength using the Eq. (2).

$$ITS = 2P/(\pi dt) \quad (2)$$

Where, P is the failure load, d and t are the diameter and thickness of the Marshall specimen respectively. For evaluating the moisture sensitivity of mixes, ITS samples were tested at 7% air voids in both dry and wet conditions. Dry ITS values of specimens compacted to 4% and 7% air void contents are reported in this section. ITS values of moisture conditioned specimens (wet) and the corresponding tensile strength ratios are presented in chapter 7. Results of indirect tensile strength test conducted on different mixes are given in **Table9**. **Photo-12** show the ITS test set-ups available

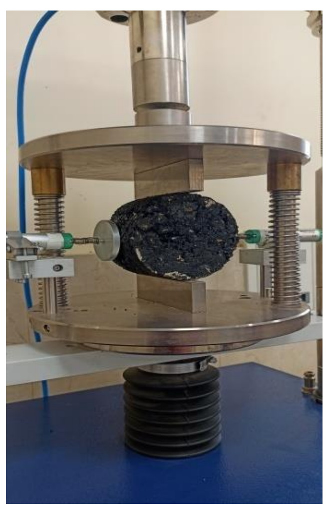

Photo-12: Set **up for ITS**

Testing of Samples

Marshall samples were prepared. three controlled samples of VG 10, VG 30 and three each of VG 30 with 6% and 8% (from the results of blend with bitumen) bottom residueforeach test were prepared and tested. The average value of density, stability, flow, and ITS are given in the table.

TABLE 9: PROPERTIES OF MIX WITH VG 10, VG 30 AND BLENDS WITH VG 30

Sample	Density, g/cc	Stability, kN	Flow, mm	ITS @25ºC, MPa
VG10 Neat	2.575	12.96	3.03	1.004
VG30Neat	2.577	14.58	3.09	1.319
VG30+6% R	2.563	13.01	3.40	1.278
VG30+8%R	2.561	12.85	3.75	1.040

8.RAP Material

Due to the exponential increase in the cost of the bitumen and the government's policy to reduce the aggregate quarrying activities in a number of states in India, the road agencies are actively considering the use of high proportions of the Reclaimed Asphalt Pavement (RAP) material in the new hot mix asphalt (HMA) production. Using high RAP content in HMA mixes may result in reduced crack resistance that often leads to premature pavement failures and consequently, early maintenance and increased rehabilitation costs. To counter this increased cracking potential in high RAP asphalt mixes the most widely adopted approach is to use rejuvenators with or without softer grade bitumen.RAP material was collected from Rashtrapati Bhavan roads, New Delhi, India. Aggregate extraction from RAP was carried out as per ASTM D2172. Distillation method was used to recover the binder from the solvent. Rotavapor was used for recovering the binder from the solvent. The binder contents in the RAP material was found to be 4.8%. **Photos 13 and 14** show the centrifuge extractor and Rotavapor respectively. Marshall cylindrical samples of 100 mm diameter were prepared at 40 % RAP. It is seen in **Table-11** that the ITS values decreases as the percentage of bottom residue in the mixture increases. This is because softer binder is replacing the virgin binder as residue content increased. Gradation of washed aggregates of RAP is given in **Table-10.**

Photo-13: Centrifuge Extractor **Photo-14: Rotavapor for Binder Recovery**

Table-10: Extracted Aggregate gradation of RAP

Sieve size	% Passing
mm	%
26.5	100
19.0	96.7
13.2	88.6
9.5	75.2
4.75	40.9
2.36	27.4
1.18	17.2
0.600	12.2
0.300	8.4
0.150	5.4
0.075	3.4

Table-11: properties of RAP, RAP with VG-30 and different dosages of bottom residue

	DENSITY	ITS
SAMPLE		MPa
RAP+VG30Neat	2.569	1.893
RAP+VG30+4%R	2.616	1.372
RAP+VG30+8%R	2.595	1.162

9 CONCLUSIONS

Bottom Residue is miscible in Bitumen and no separation has been observed. From the laboratory tests, addition of bottom residue to the bitumen it softens the base bitumen significantly. So, it can be used as bitumen extenders thus reducing cost of bitumen. Also, VG 10 can be achieved by mixing in right proportion (around 8 to 9% bottom residue can be replaced in the bitumen) with VG 30.Bituminous mixes with Bottom Residue modified binder is showing equivalent results. Bottom Residue has a potential usage in bitumen and bituminous mixes. For RAP mixes, it is seen that the ITS values decrease as the percentage of bottom residue in the mixture increases. This is because softer binder is replacing the virgin binder as residue content increased. However, the study had some limitations as it was a preliminary feasibility study in the laboratory. Detailed performance studies (Rutting and fatigue characteristics) were required for the full-scale usage. Further detailed study is to be done for various other aspects specially its usage in RAP Mixes which will have a large effect on **Circular Economy** Also, a uniform specification for manufacturing process and of Bottom Residue has to be taken up in near future.