Validation of Fourier Transform Infrared Radiation (FTIR) Spectrometer Test Results using Indirect Tensile Strength and Rut Analysis for Modified Bituminous Binders

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Abstract

Bituminous pavements play extensive part of roadway system in India, building and maintaining them is an expensive task. Hence, it is vital that a quality binder and mix be produced consistently and economically. Performance of a flexible pavement mainly depends on bituminous mix design done in laboratory, mix design procedure followed in laboratory should be a true indicator of field conditions. Further modified bituminous binders are used to enhance the performance of bituminous mix as well as bituminous pavements. Since modified bituminous binders are expensive and exhibit less shelf life, it is observed that, the adulteration of modified bituminous binder's increases due to its cost in market, which reduces the performance of binder, leading to decline in pavement performance during service. Adulteration of binders cannot be easily traced through conventional testing/evaluation procedures; therefore, it requires a specialized, quick, efficient and fool proof testing practice of binder. In this direction FTIR Spectroscopy plays an important role in identification of modified binders characteristics. In current studies modified bituminous binders such as Polymer Modified Binder (PMB), Crumb Rubber Modified (CRM) binder, Natural Asphalt modified binder are prepared and used. Similarly adulterants such as Charcoal Powder and Photo Copier Powder were used to modify the binder. The modified binders were subjected to tests as prescribed by Bureau of Indian Standards (BIS). Apart from BIS tests, binders were subject to FTIR spectroscopy scanning and results of FTIR scanning indicated the clear difference between the modifiers and adulterants modified bituminous binders characteristics. Further the same was validated by performing Indirect Tensile Strength (ITS) test and Rutting Test on Bituminous Concrete (BC) Grade-1 mix, the test results of ITS and Rutting Test validated FTIR test results. Performance of adulterated binder mix was less compared to modified binder mix.

Keywords: Polymer Modified Bituminous Binder, Fourier Transform Infrared Spectrometer, Flexible Pavement, Bituminous Concrete Grade-1

Introduction

Over the last decade, percentage of heavy trucks have increased enormously in the total traffic volume plying on Indian Highways, demanding stronger and long lasting pavements. This is a challenging task since most traditional methods of selecting bituminous binder, aggregate, mixture specifications and its evaluation were developed over fifty years ago, largely based on empirical correlations with traffic conditions which have transformed over the years. India has the second largest road network in world with a total length of 6,215,797 kilometers. Nearly 90 percent of the total road length are flexible pavements and consists of bituminous layers. Construction and maintenance of such large network requires a stringent and fool proof quality control system. Indian Roads Congress (IRC) provides the guidelines with regard to road design, construction and maintenance. In this process modified bituminous binder characterization is governed by IRC's IRC SP 53-2010 specifications along with BIS 15462-2009 specifications. Modified bituminous materials can bring real benefits to highway maintenance and construction, in terms of better and longer lasting roads, and savings in total road life costs. Manufacturing and use of modified bituminous binder has to go through several processes before it is used in pavement layer. During this process bituminous binder undergoes adulteration most common reason for bitumen adulteration is the price of modified binder and demand in market.

Current research project involves testing of modified binders using FTIR spectrometer and evaluate the test results by performing performance tests such as ITS and rutting on the BC Grade-1 mixes, prepared using modified binders and adulterants.

Formulation of Problem

Flexible pavement failures due to bituminous binder are mainly due to wrong choice of binder or adulteration of binder. The second is very predominant, in case of modified binders being tempered performance of the pavement comes down. The demand for modified binders pushes adulterated binders in the market, based on the survey carried in market it was found that the cost of modified binders also contributes to increase in adulterated binders, further two common types of adulterants which are used in binder adulteration are identified as charcoal powder and photo copier powder. Further the common types of modifiers used in modification of binders are Styrene Butadiene Styrene (SBS), Crumb Rubber Modifier (CRM) and Natural Asphalt (NA). It was also established that, Viscosity Grade (VG) binder-10 is used as the base binder in binder modification processes.

The cost of modifiers and modification process is tedious, time consuming, demand of modified binders in market is high, binder manufacturers other than refineries would push the adulterated binders in market. The current binder evaluation procedure prescribed by BIS and IRC cannot detect the adulterated binders from that off modified binders, therefore the modified and adulterated binder's samples prepared were subjected to FTIR spectroscopy test and results were recorded. It would not be sufficient if the results are recorded further the performance of bituminous mix needs to be cross verified with the results of FTIR and validate the results. In this direction samples of modified binders and adulterants required for testing was prepared in laboratory and tests on binders were carried out, mix design as per Marshall method of bituminous mixes was carried out for BC Grade-1 mix satisfying the requirements of IRC. The mix thus prepared at design binder content was subjected to ITS and Rutting test. Using the results obtained by ITS and Rutting are compared with that of FTIR and results of FTIR are validated.

Scope and Objectives of Study

The investigation needs to address the issue of binder being modified and produce enough evidence to ascertain the reason following study was envisaged. Samples of virgin bitumen used for manufacturing modified bitumen to be collected and subject it to test as per BIS and IRC code provisions. To manufacture modified as well as adulterant binders using the modifiers and adulterants collected. Subject the modified binder and adulterated binders to FTIR test and to obtain the test results. By performing Marshall method of bituminous mix and later determine the Optimum Binder Content (OBC) of the mix with different binders. To prepare the specimen at OBC's and subject it to performance tests such as ITS and Rutting.

Highlights

- FTIR Spectrometer identifies chemical bond using infrared absorption spectrum
- Polymer Modified Bituminous Binder is produced by adding different polymer additive then subjected to test as per BIS.
- Similarly, to understand the effectiveness of the test prescribed by BIS, samples of virgin bituminous binders blended with adulterants were prepared and tested as per BIS. BIS tests were not able to distinguish polymer additive blend with that of adulterants blend, instead the test results cleared the limits prescribed by BIS to be used in road construction.
- FTIR scan results clearly distinguished binders with polymer and that of adulterants.
- Performance tests on bituminous mixes validates the results of FTIR results.

Materials and Methods

Test on prepared samples were conducted to characterize the properties of Virgin Bitumen and Polymerized Bitumen. Different percentage of polymer concentration provides a wider range of results which helps in analyzing each type of the polymer blend at particular concentration. Following tests were carried out on the samples;

- Penetration test (In accordance with BIS 1203-1978)
- Softening test (In accordance with BIS 1205-1978)
- Viscosity test (In accordance with BIS 1206-1978-Part-3)
- Specific gravity test (In accordance with BIS 1202-1978)
- Flash and Fire point test (In accordance with BIS 1209-1978)
- Solubility in Trichloroethylene test (In accordance with BIS 1216-1978)
- Ductility test (In accordance with BIS 1208-1978)

Materials

The material used in this study includes Viscosity Grade-10 (VG-10) bitumen obtained from refinery.

The materials used for modification are as follows the same can be viewed at Figure-(1) and (2).

- Finoprene
- Finoprene (High Quality)
- Natural asphalt
- Crumb Rubber Modifier (CRM) and

Adulterants used for current studies

- Copier toner powder
- Charcoal powder

Figure-1 Modifiers

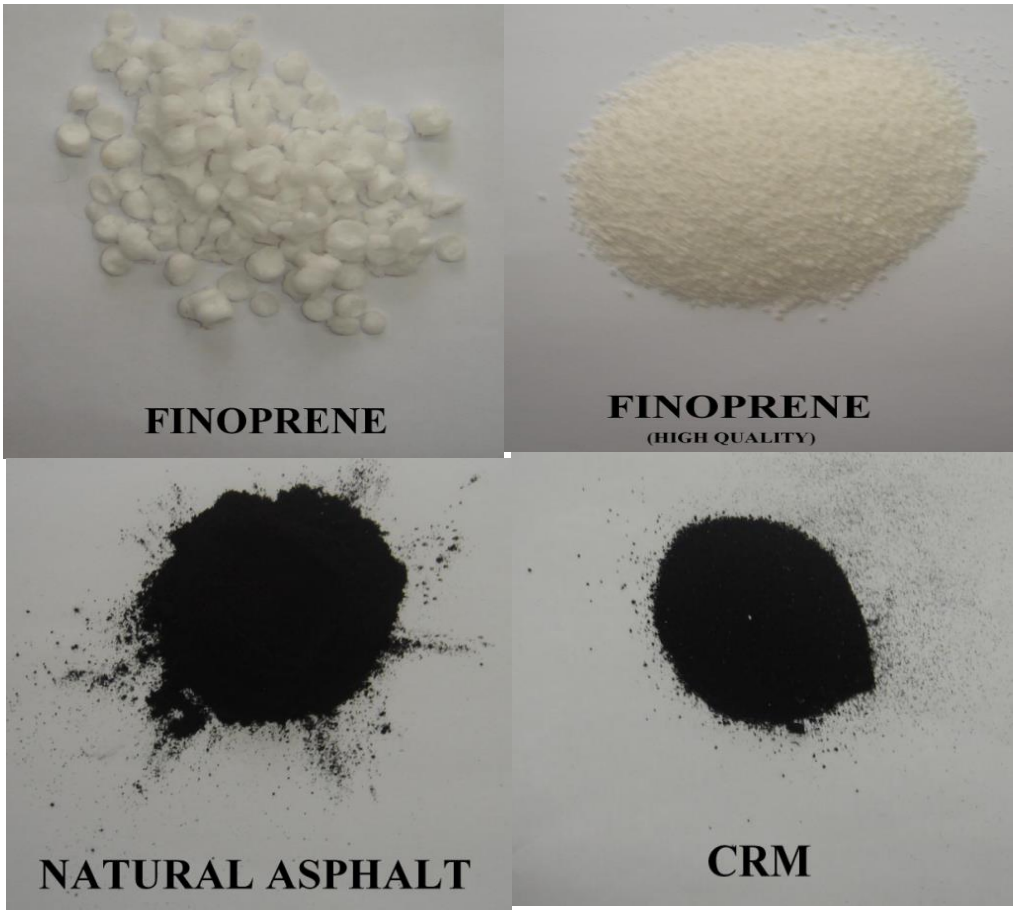
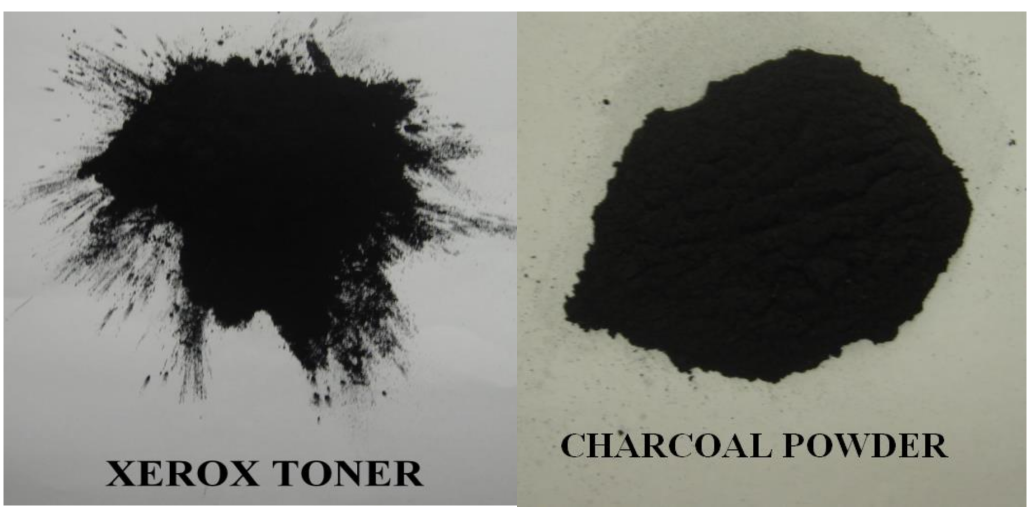


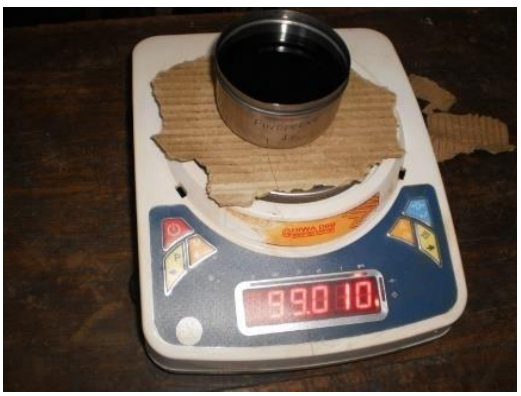
Figure-2 Adulterants



Preparation of Polymer Modified Bitumen sample

The preparation of PMB involves softening of bitumen to a point where it comes to pouring consistency, the temperature range is between 75º and 100º C or above the approximate temperature at which bitumen softens (flow state). Further bitumen samples are weighed by reducing their weight equal to weight of additives (Polymer Modifier) added to produce PMB, modifiers were used in incremental value of 1% by weight. Further samples with adulterants were also prepared as indicated above. PMB samples were prepared using melt blending technique, bitumen was heated in an oven and was continuously stirred at temperature range of 160°C and 170°C, here modifier in desired quantity was added and blended continuously to obtain a homogenous mixture. After blending process, the modified bitumen was cooled and sealed in containers covered with aluminum foil and stored for further testing. Table (1) and (2) indicates the test results, and Figure (3) indicates the preparation process.

Figure-3 Preparation of Polymer Modified Bitumen



Step 1: Weight of Bitumen

Step 2: Addition of Polymer Modifier

Step 3: Blending and Production of Polymer Modified Bitumen

Table-1 Test Results of Viscosity Grade-10 Bitumen

TEST DESCRIPTION & CODE	RESULTS	REQUIRMENT OF VG-10 AS PER BIS 73-2006
Penetration at 25ºC, 100 g, 5 s, 0.1 mm. (BIS 1203-1978)	90	80-100
Softening Point (R&B), ºC, Min (BIS 1205-1978)	43	40
Viscosity at 135 ^{0}C in CPS (ASTM 4402-2006)	287	250
Specific Gravity (BIS 1202-1978)	0.98	0.9 – 1.02
Flash Point (º C) (BIS 1209-1978)	258	220
Solubility in Trichloroethylene in Percent, Min. (BIS 1216-1978)	99.0	99.0
Viscosity Ratio on residue of Thin Film Oven Test (TFOT) Sample 60 ^{0}C, Max (BIS 1206 (Part-2)-1978 & 9382-1979)	2.7	4.0
Ductility at 25 ^{0}C, in cm, Min, on residue of TFOT Sample (BIS 1208-1978 & 9382-1979)	85	75

Table-2 Test Results of PMB and Adulterant Binders

Test	Minimum Criteria IRC:53	Finoprene	Finowax	Natural Asphalt	Crumb Rubber Modifier	Copier Toner Powder	Charcoal Powder
IS:1203-1978,Penetration Test	<60	36	52	43	42	43	49
IS:1202-1978 Specific Gravity Test	0.99-1.02	1.020	1.040	0.980	1.020	1.050	0.990
IS:1205-1978, Softening Point Test	60^{0}C Min	70	67	59	57	56	59
IS:1208-1978, Ductility Test	-	30	22	21	50	19	16
IS:1209-1978, Flash and fire Point Test	Flash Point 220 ^{0}C MIn	Flash Point -260 ^{0}C Min	Flash Point -256 ^{0}C Min	Flash Point -235 ^{0}C Min	Flash Point -250 ^{0}C Min Fire Point-290^{0}C	Flash Point - 254 ^{0}C Min Fire Point-269^{0}C	Flash Point - 256 ^{0}C Min Fire Point-265^{0}C

		Fire Point- 276^0C	Fire Point- 282^0C	Fire Point- 270^0C			
Viscosity Test- Brook's Field Viscometer	2-6 Poise at 150^0C	@135^0C- 7.110 P @150^0C- 3.50 P	@135^0C- 6.903 P @150^0C- 3.460 P	@135^0C- 6.018 P @150^0C- 2.032 P	@135^0C-6.370 P @150^0C-2.436 P	@135^0C-3.32 P @150^0C-2.236 P	@135^0C- 3.47 P @150^0C- 2.789 P
IS:15462-2004, Elastic Recovery Test, ANNEX-A	50%	59%	68%	65%	51%	49%	48%
IS 9382 Mass, Percent, max	1.0	0.56	0.53	0.64	0.72	0.63	0.75
Reduction in Penetration of residue at 25^0c percent, max	40	28	25	31	46	52	43
Increase in Softening Point ^{0}c, max	6	2	2	2	2	3	2
Elastic Recovery in Percent, min	35%	39%	54%	48%	42%	23%	28%

After comparing the test results of PMB and adulterant modified bitumen, test results in accordance to BIS or IRC provisions could not distinguish PMB products with that of adulterant modified bitumen. Test results of PMB's as well as adulterant binders were within permissible vales specified by code. Further test results have pushed researchers to bring out a testing procedure which can identify and clarify the end users whether the product obtained by them is genuine or not. Hence the modified binders both PMB as well as adulterants were subjected to Infrared Spectroscopy, where the infrared wave when passed through these binder samples, depending upon the materials present in the binder certain change in the waves of infrared was observed, where the change in wave frequency was mainly dependent on the percentage or quantum of wave length absorption and emittance which could clear indicate the type of materials present in the binder.

Working Principle of Infrared Spectroscopy

FTIR Spectroscopy was used for the current studies wherein, FTIR is a type of spectroscopy IR analysis that uses infrared radiation to record molecule movements via computer-based programs. It uses a formula called Fournier Transform and a scheme of conversion called Michelson Interferometer. FTIR spectroscopy is a multiplexing technique, where all optical frequencies from the source are observed simultaneously over a period of time known as ***scan time***.

Fourier Transform is a mathematical equation that converts the spectral signal performed by a computer from a time domain towards a frequency domain. Its mechanism of action is based on the Michelson Interferometer modulation.

Spectrum is the graph that shows movement of compounds when they undergo a reaction involving infrared radiation. The movement of compound is actually a vibration, which results to the emission of energy measured in wavelength or wave numbers. Thus, the absorption spectrum is a plot of wavelength vs. a beam of radiation.

Binder's Testing Using FTIR

The modifier or adulterants when added to bitumen exhibit their own wave number. The polymer modifiers used for modification has particular wave number 1540 cm^{-1}. The change in wave number indicates addition of other materials. The wave number 1252 cm^{-1} indicates the adulterants such as charcoal powder or copier toner which contains high percentage of carbon. By observing the wave number one can clearly distinguish the material blended with binder, this was not possible by performing the tests specified by BIS material test procedure.

Based on test results it can be concluded that, FTIR is capable of differentiating between polymer and non-polymer-modified binders. Further the results obtained using FTIR can be verified by preparing samples of Bituminous Concrete Grade-1 mix and subjecting it to, Indirect Tensile Strength and Rut Analysis, so that the results obtained from the above test can be used for validation.

Mix Design of Bituminous Concrete Grade-1by Marshall Method.

The following steps are involved in Marshall Method of Mix Design as per ASTM D 6927-2006

1. **Select a bituminous binder suitable for the climate.**
2. **Select aggregates that meet the suitability criteria.**
3. **Create an aggregate blend that meets the gradation criteria to BC Grade-1.**
4. **Establish specimen mixing and compaction temperatures from the viscosity-temperature chart for the bituminous binder.**
5. **Compact three specimens at each of five binder contents 0.5% apart spanning the expected optimum bituminous content.**
6. **Determine the mix volumetrics (G_{mb}, G each specimen. Mm, VMA, VFB)**
7. **Measure the performance properties of each specimen at the high service temperature of 60°C.**

Figure (4) indicates the gradation curve of BC Grade-1which falls within the limits prescribed by Ministry of Road Transport and Highways (MoRT&H) section 500. Table (3) indicates the Marshall test properties.

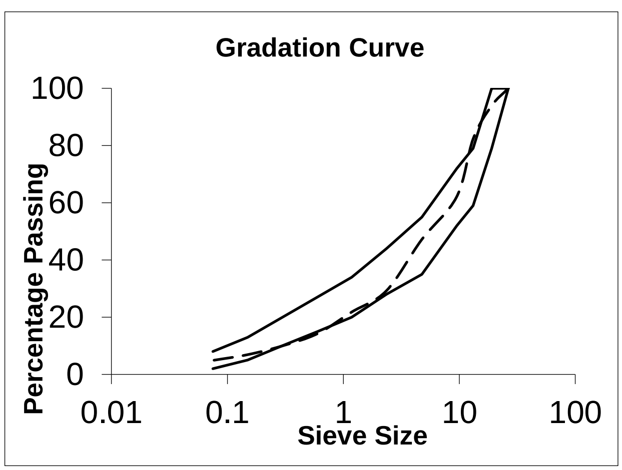


Figure (4) Gradation Curve BC Grade-1

Table-3 Marshall Test Results of VG-10 binder along with Various Modifiers and Adulterants

Properties	Standard Values	VG-10 Binder	Finoprene with VG-10 Binder	Finoprene (High Quality) with VG-10 Binder	Natural Asphalt with VG-10 Binder	Crumb Rubber Modifier with VG-10 Binder	Charcoal Powder with VG-10 Binder	Photo Copier Powder with VG-10 Binder
Optimum Binder Content (%)	5 -- 6	5.1	5.5	5.6	5.4	5.6	5.3	5.2
Stability (Kg)	900	1590	2078	2100	1780	1987	1780	1690
Flow (mm)	2--4	3.6	3	3.5	3.2	4	3.5	3
Density (g/cc)		2.38	2.39	2.38	2.36	2.38	2.36	2.37
Air Voids (%)	3 -- 6	4.02	4	4	4.2	4.5	4.5	4
Void in Mineral Aggregate (%)	15 min	15.87	16.43	16.34	15.90	16.32	16.32	15.89
Void Filled Bitumen (%)	65 -- 75	74.7	72	73	73	72	72	70
Marshall Quotient	400	441.67	692.70	600	556.25	496.75	496.75	563

Evaluation of Moisture Sensitivity

The specimens for BC Grade-1 mix with design binder contents for seven binders are cast. As per AASTHO T-283 moisture evaluation test, one subset of three specimens were compacted to approximately 7 percent air voids at design binder content and named as controlled specimens. One more subset of three specimens were cast with design binder content at 4 percent air voids. The evaluation for moisture sensitivity was done by thawing cycle only. The freezing cycle was not considered, since the minimum pavement temperatures does not fall below zero degree. The controlled subsets of three specimens were conditioned by subjecting the specimens to partial vacuum saturation that is, the specimens were kept at 60^{o}C for 24 hours in water bath followed by 2 hours in temperature-controlled chamber at 25^{o}C. The other subset of three specimens were kept in a temperature-controlled chamber at 25^{0}C for 2 hours and these specimens were called as unconditioned specimens. All the specimens were subjected to Indirect Tensile Strengths and the ratio of ITS conditioned to that of unconditioned specimens are represented as Tensile Strength Ratio (TSR). The results of ITS are indicated in the figure (5).

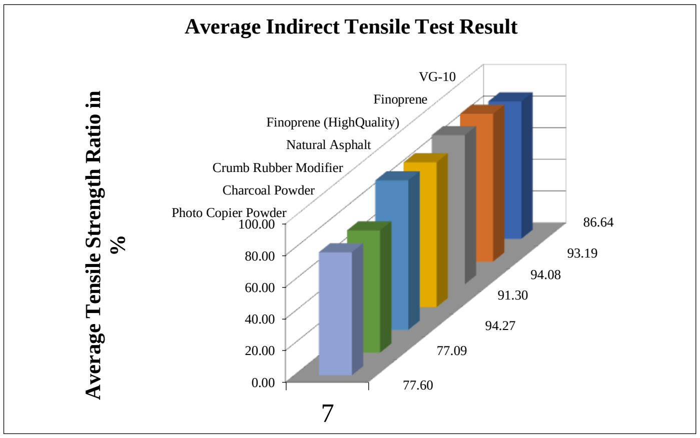


Figure (5) Results of AASHTO-T-283 Test

Steps involved in Rutting Potential of the Bituminous Mixes prepared with various Binders at their respective OBC's

1. The complete composition of pavement layers was built in laboratory using a Wheel tracking equipment which basically accommodates various thickness of Pavements and capable of loading different tyre pressure while testing.
2. Currently the pavement composition as per IRC: 37-2018 is constructed and the pavement is subjected to a tyre pressure of 7 Kg/cm^2, later the data in terms of number of passes required to cause 20 mm thick depression (rutting) on the surface of pavement is noted.

Figure (6) indicates the rutting test results of all the seven binders, were number of passes required to cause 20 mm depression on the surface caused due to a tyre pressure of 7 kg/cm^2.

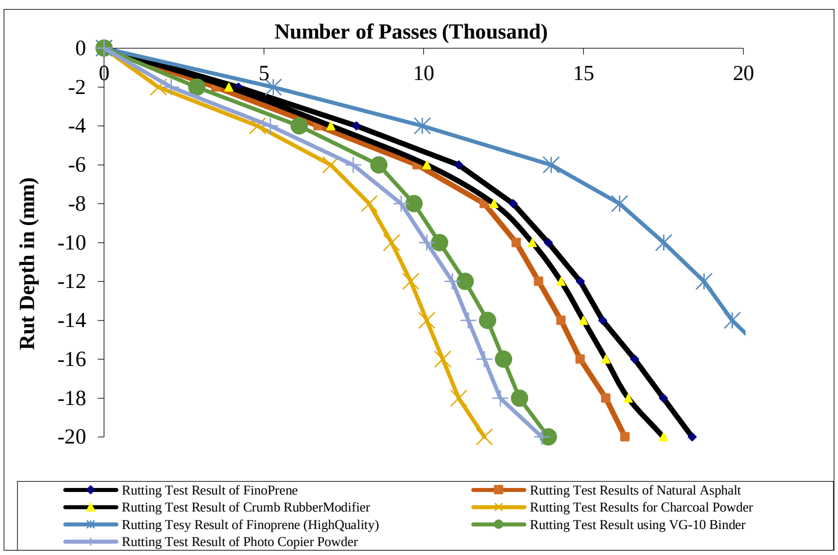


Figure (6) Rutting Test Results

Discussion and Conclusion

Based on obtained results from FTIR Scanner, it can be observed that, parameters like absorption height, wave number, band width for virgin bitumen, modified bitumen & bitumen with adulterants are completely independent of each other. This gives the idea of what material is blended along virgin binder. By observing the wave number and absorption height of particular polymer, there is increase in absorption height by increasing polymer concentration there by plastomaric or elastomeric properties of modified bitumen also increases.

1. From the above studies it is evident that the Performance tests such as ITS and Rutting Test have supported the test results obtained from FTIR test results.
2. It is very difficult and time consuming to establish the adulterations of Bituminous Binder through the conventional testing process.
3. Even the Marshall test results could not distinguish the adulterants from the modifiers.
4. Performing the tests such as ITS and Rutting for every batch of mix design causes delay in completing the mix design process.
5. Therefore it would be convenient to perform FTIR scan on bituminous binder before subjecting it to mix design. The number of samples and tests can be done with ease.

BIS tests are not able to trace the quality & quantity of polymer used in preparation of PMB, hence the chance of bitumen adulteration is high. FTIR Scanner testing process provides procedure to identify the quality & quantity of PMB. Protocols are developed for FTIR analysis of polymer quantification in bitumen binders.

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