An Experimental Study to Develop Bituminous Based Crack Sealant Using Rheological Data

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Abstract-Pavement maintenance in hot climatic areas requires a significant annual budget to keep it at a desired serviceability level. Filling of cracks during the pavement maintenance with an appropriate hot-poured crack sealant can save money. However, this cost can further be reduced by utilizing the local modifiers in a relatively higher penetration grade bitumen. This study presents optimizing the percentage rice husk as modifier in 80/100 penetration grade bitumen. A comparison of rheological properties among different crack sealants and bitumen modified with different percentages of modifier has been reported. Intermediate and high temperature performance characteristics of different crack sealants have been compared using stiffness parameters. Temperature and frequency sweep tests were run at different temperatures and frequency levels using a dynamic shear rheometer. The results showed a significant relationship of shear modulus (G*) with percentage addition of modifier and test temperatures. The developed sealant showed elastic behavior at lower temperatures and at higher percentage of modifier. The study recommends utilizing an effective and economical material, which is based on a soft bitumen and 9% rice husk.

Keywords-Flexible Pavements, Bituminous Crack Sealants, Complex Shear Modulus, Time and Temperature Superposition Principle, Yield Test

I. INTRODUCTION

Pavement performance under a specified traffic condition depends on many factors. Cracking associated with the pavement surface commonly reflects climatic variations. Cracks appear at the top surface is one of the signs of flexible pavement failure, associated with a significant temperature variation of that area. In order to achieve a cost-effective crack sealing and filling operation and proper field performance, two factors must be closely controlled: quality of sealant installation and sealant mechanical and rheological properties (such as viscosity, bulk stiffness, and adhesive bonding). Joint and crack sealants prevent the retention of incompressible materials in joints that protects pavement structure from moisture damage. Fend investigated the difference in performance of hot poured crack sealants and cold seal bands through laboratory and field studies. Different laboratory experimentation methods were also developed to characterize the seal bands. Also field investigation methodology was proposed to ascertain field performance and causes behind failures. The study recommended an improved strategies for application of seal bands [i].

A sealant losses its cohesion or adhesion with the passage of time mainly due to weather effects. A sealant fails to take external applied forces, when it losses inter particle cohesion. Such failure types are usually called as cohesion failure. An adhesion failure occur when sealant does not withstand the stress developed at interface between the asphalt surface and the sealant. Such stresses mainly develop due to shrinkage phenomenon during the winter season. The performance of crack sealant in terms of stiffness ability to dissipate tensile stresses also depends on its installation temperature and method of installation [ii].

Rice husk is produced from the rice crop that covers almost 1% of earth surface and is considered as the main food source [iii]. Husk is the rice kernel outer cover having two interlocking halves. Approximately 20% of rice paddy is husk that has been extracted from the two interlocking halves of kernel outer covers. In Pakistan about 1.15 million tons of husk out of about 6.0 million tons of rice has been produced every year [iv]. Rice husk is one of the main source of pozzolanic material that improves the stiffness [v].

Performance evaluation of crack sealants through laboratory experimentation have been proposed in different research studies in the past. The main purpose of such studies were to ascertain the parameters that could affect sealant's field performance at different temperatures [vi]. These parameters could be the mechanical properties of sealant, adhesion to different pavement materials, or chemical composition of sealant. Viscoelastic properties of silicone sealants by using dynamic shear rheometer (DSR) were investigated for six silicone sealants at different frequency levels and a temperature range of -30^{o}C to +50^{o}C with an increment of 10^{o}C. Complex shear modulus (G*) and phase angle (δ), obtained from the DSR tests were used to construct master curves at a reference temperature.

Reference [vii] studied the critical requirement of sealing the cracks using crack sealants and reported that cost or sealing the crack be reduced by adopting the methodology as proposed by this study. The study concluded that some specific cracks, which could not be identified with the help of laser guided tools had no sealing requirement. Different cracks were chosen to complete this study. The selection criteria was based on the type of crack and the dimensions of the crack. Reference [viii] used a cyclic shear and constant horizontal deformations to evaluate the performance of rigid pavement joint sealants. The horizontal deformation was used for simulating temperature loading, while the cyclic shear was used for simulating traffic loading. The scheme [ix] investigated the effect of rout geometry on crack sealants performance using an index and reported that sealants with clean and seal has relatively poor performance. At the same time over band improved the sealant adhesive and plow off strength. Reference [x] studied the linear viscoelastic behavior of hot-poured crack sealant at relatively low serviceability temperatures using bending beam rheometer. The test data was analyzed using a three-dimensional finite-element model and concluded that mechanical behavior of crack sealant can predicted by a linear viscoelastic model.

Several studies in the past have been conducted by using fine aggregate (filler) with bitumen. The main purpose was to stiffen the asphalt mixture and mastic. Such mixtures have been recommended for either seal coats or to improve the rut resistance of asphalt mixtures. Also, to improve the fatigue properties of asphalt mixtures in the cold areas.

The addition of mineral filler to bitumen stiffen the mastic and interrupt the crack growth in the mastic [xi]. Further, the acidic and basic nature of fillers may age the bitumen mastic by oxidative catalysis and chemical adsorption reactions [xii]. The size of mineral filler effect the adhesion properties of mastic as we go on increasing the diameter of filler, adhesion decreases [xiii]. No study as such recommends using a mastic as sealant. Therefore, based on the literature the function of both (rice husk and filler) are different.

Specific research areas depends on the intended functions and utility. As such rice husk modified bitumen as crack sealants have not been used in the cold climatic areas, but it is a common practice in Pakistan. In Pakistan, rice husk or saw dust is a common composition of sealants. It is important to characterize such mixtures.

II. Objectives and Research Methodology

Two phase study was planned to achieve the study objectives. The objectives of present study were to characterize intermediate and high temperature performance of crack sealants and to compare results with laboratory prepared sealant. This sealant was prepared using 80/100 penetration grade bitumen and rice husk. Also, to propose the optimum solution towards the utilization of local modifier such as rice husk. The scope of present study includes selecting three commonly available crack sealants (A, B, and C) and mixing of rice husk (at different percentages by mass) to 80/100 penetration grade bitumen. The program consists of adding four different percentages of rice husk (3%, 6%, 9% and 12%) by mass of bitumen in 80/100 penetration grade bitumen. The base bitumen was procured from Attock refinery, Rawalpindi, Pakistan. Conventional and rheological properties of base bitumen and the modified bitumen (sealant) were determined in the laboratory. Fig. 1 illustrate the testing program adopted for this research study.

The testing program involved two phases; mixing of rice husk was completed in the initial phase followed by comprehensive testing using conventional and rheological testing. Four different percentages of rice husk were selected and conventional testing was performed to confirm that the modified bituminous sealant should not be stiff enough that it losses viscosity and ductility level while applying in the field. A minimum ductility value of 30 cm was chosen to meet this criteria. Air dried rice husk material, grinded and passed through sieve no. 30 was utilized for modification of base bitumen.

Mixing of different percentages of rice husk for at least 30 minutes through a mechanical device at 160 ºC were made. The obtained samples after a uniform mixing were stored in a refrigerator till further testing. Both conventional and superpave testing were run on the samples. Mixture of base bitumen with 12% rice husk failed to achieve 30 cm ductility value. The other reason was the difficulty of handling this sample as the amount of rice husk was significant and the material crumbled while molding

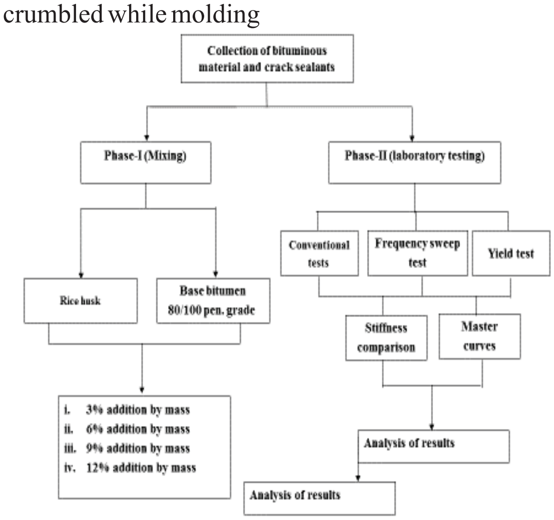

Fig. 1. Scope and methodology of the work

A. Conventional Testing

Several tests were conducted on bituminous crack sealants samples to ascertain the physical behavior. The results of ductility, penetration, softening point and flash and fire point test have been reported in Table I.

TABLE I
STANDARD SPECIFICATIONS OF ASPHALT

Test Description	Test Method (AASHTO)	Test results of base bitumen and modification with rice husk				
		80-100	3%	6%	9%	12%
Ductility @ 25°C (cm)	T 51	105	65	47	35	12
Softening point, (°C)	T 53	46	51	57	64	76
Penetration @ 25°C (1/10mm)	T 49	86	67	58	49	38
Flash Point, Cleveland Open Cup Apparatus (°C)	T 48	232	229	220	221	196

It may be noted from Table I that the ductility value of specimens decreases with an increase in percentage rice husk. Similar trends have been noted in penetration values. The softening point increases with an increase in the percentage. It may also be noted that at 12%, the ductility value of specimen were about 12cm and the flash point reduced to 196°C. It would be difficult to handle the mastic as well. Therefore based on the conventional testing, a percentage of 12% was dropped from the present scope of the study and three percentages were selected for further rheological testing. Similarly, tests were run on all the three sealants as reported in Table II.

TABLE II
CONVENTIONAL TEST PROPERTIES OF CRACK SEALANTS

Crack Sealant (ASTM Type)	Pen. at 25°C. 150g, 5sec, cone	Soft. Point	Flow at 60°C, 5Hr.s	Bond test at -20°F (50% extendibility) as per manufacturer			Resilience percent
	(dmm)	(°C)	(mm)	1st Cycle	2nd Cycle	3rd Cycle	
Sealant A	69	61	0.45	P	P	P	70
Sealant B	27	81	0.4	P	P	F	68
Sealant C	63	65	7.6	F	P	F	62

The available sealants were bituminous based and qualify for application in hot climatic area. Crack sealants were homogenized before preparation of test samples. Aging of sealants was conducted using rolling thin film oven at a constant temperature for at least 16 hours. Specimens in replicates were tested under temperature and frequency sweep and the results were reported in the proceeding sections. Fig. 2 shows prepared crack sealant using rice husk.

B. Dynamic Shear Rheometer

Various tests using a 25 mm diameter test specimen of bitumen were carried out on a DSR in accordance with AASHTO T-315 test protocol [xiv]. A strain level of 10% was chosen to run the frequency sweep and temperature sweep testing on rolling thin film oven aged specimens. Anton Paar DSR as shown in Fig. II works within a shear modulus range of 100Pa to 10MPa with its parallel plate geometry and capable of maintaining a test temperature from -30 °C to 120 °C.

Temperature sweep and frequency sweep tests were run at a temperature range of 28°C to 82°C (with an increment of 6 °C) and at a frequency range from 0.1 to 100 rad/sec using a dynamic shear rheometer. Data obtained from the test like storage modulus, loss modulus and deflection was utilized to calculate the complex shear modulus (G*), and phase angle (δ), respectively. Phase angle and complex viscosity (η) rutting (G*sin/δ) and fatigue factor (G*sin δ) were also computed to assess intermediate and high temperature behavior. Results were elaborated with the help of plot against different parameters. Comparisons of results were shown at intermediate (28ºC, 46ºC) and high temperatures (64ºC and 80ºC). The viscoelastic characteristics of the sealants and modifiers based on time-temperature superposition principle (TTSP) were interpreted in the form of shifted master curves at a reference temperature of 46ºC.

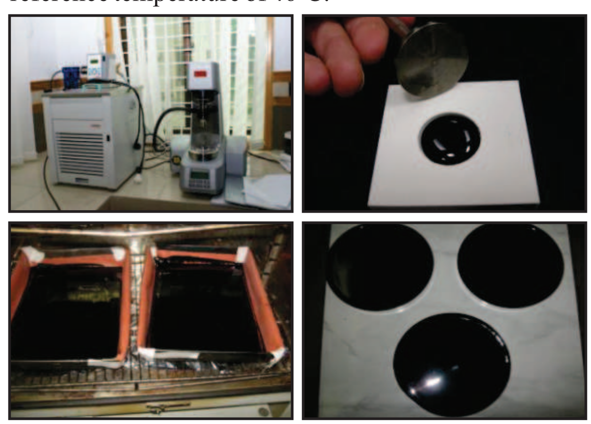

Fig. 2. Crack Sealant Prepared for Dynamic Shear Rheometer Testing

III. Results and Discussion

Data obtained from the frequency sweep and temperature sweep test were analyzed using a statistical tool to investigate the effect of addition of rice husk, temperature and frequency of loading on the complex shear modulus (stiffness) values of base bitumen and in general on the selected crack sealants.

A. Development of Master Curves

Bituminous crack sealants are the viscoelastic material that follow time and temperature superposition principle. This means that the effect of temperature can be ascertain by changing the loading frequency and vice versa. Sigmoidal fit parameters were utilized in the following relationship to ascertain the horizontal shift function, while shifting the different temperature stiffness data on a reference. Fitting of sigmoidal functions (δ, α, β, & γ) were obtained using the following relationship in a nonlinear least square regression iteration process [xv].

$$Log|E^*| = \delta + \frac{\alpha}{1 + e^{\beta + \gamma(\log Tr)}} \quad (1)$$

The sigmoidal functions depend on aggregate gradation, binder content and air voids. These parameters also represents the minimum value of E*, the maximum value of E* and the shape of the sigmoidal functions. A reference temperature of 46°C has been used for the development of master curves. Fig. 3 shows a comparison of developed master curves of different crack sealants and rice husk modified bitumen. It may be noted from Fig. 3 that all specimens are almost at the same stiffness range (complex shear modulus) except crack sealant B, which was relatively stiffer and showed higher softening point in Table II. The results of Table II are in line with the developed master curves in Fig. 3.

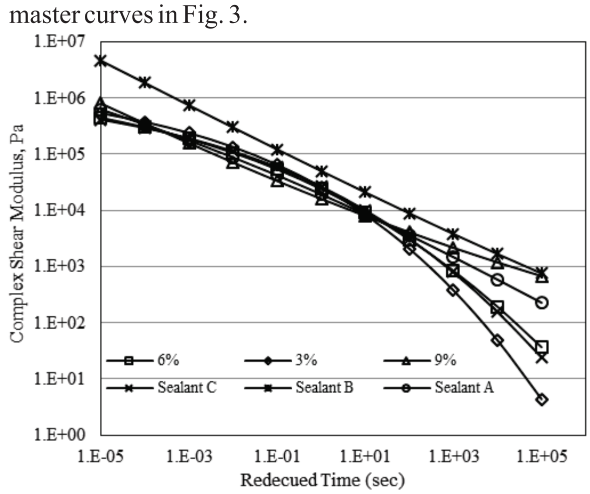

Fig. 3. Developed Master Curves of Different Crack Sealants and Bituminous Sealants

Master curves illustrates the time and temperature interdependency of material and temperature sensitivity in the form of a shift, which may be explained with the help of horizontal shift factors. It may be noted from Fig. 3 that all the sealants and modifiers showed almost similar range of stiffness at the highest frequency level, but the trends and lowest stiffness values changed with a decrease in frequency level. Moreover, the trend lines have shown different patterns. Fig. 4 and 5 showed a family of master curves with similar trends.

It may be noted from Fig. 4 that an addition of 9% rice husk yielded almost linear trend in the bituminous sealant, which is similar as sealant A and B. The stiffness value of bituminous sealant at lower frequency level was almost same as of sealant B. An addition of 9% rice husk in the bitumen therefore meet the desired criteria of producing a local modified sealant that can fulfill the stiffness requirement of available crack sealants. Similarly, Fig. 5 presents a comparison of master curves and their trends in case of 3% and 6% addition of rice husk and a soft sealant C. Almost similar range of stiffness may be noted at different frequency levels except at lower frequency range. Bitumen sealant with 6% rice husk showed better stiffness values.

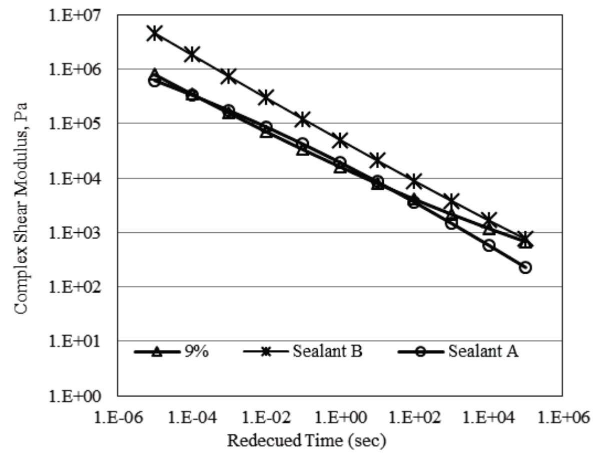

Fig. 4. Comparison of master curves of crack sealants with 9% modified bitumen

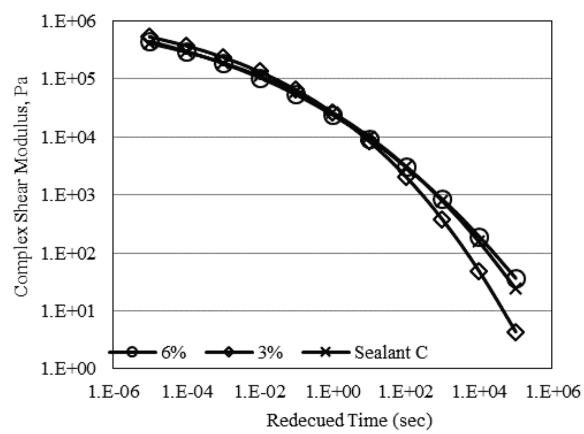

Fig. 5. Comparison of master curves of 3% and 6% modified bitumen with sealant

It may also be noted from Fig. 5 that master curves in case of softer crack sealants were in a curve like pattern. A comparison of shift factors were also made to see the effect of addition of rice husk to the temperature sensitivity of bituminous sealants. Shift factor (log a [T]) helps in moving the curves plotted at different temperatures to a reference temperature. Fig. 6 shows trends of shift factor corresponding to each addition of rice husk.

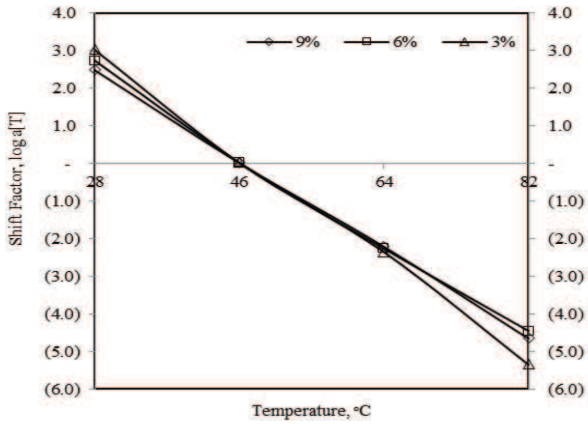

Fig. 6. A comparison of horizontal shift factors for bituminous

The basic purpose of comparing the horizontal shift factors was to ascertain the temperature sensitivity of bitumen with an increase in percentage rice husk. It may be noted from Fig. 6 that the addition of modifier (rice husk) decreases the sensitivity of sealants. As percentage rice husk increases, the complex shear modulus value also increases.

B. Yield Test

Yield test is relatively a simple and easy test to measure the grade of sealant. Yield test was performed at different temperatures using a DSR on 25mm diameter homogenized crack sealant specimens at a gap setting of 2mm. This test applies a monotonic constant shear-rate loading and measures resistance to yield-type failure. The test was performed in replicates at three different temperatures, which were at grade, above grade and below performance grade. The general pass/fail criteria of sealants was used as a minimum of 100% shear strain against a 150 Pa applied shear stress value. Same could be computed using the typical plot showing shear stress versus shear strain in Fig. 7.

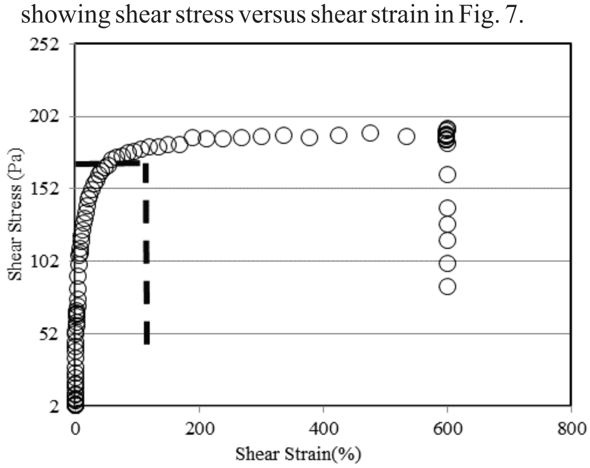

Fig. 7. Shear stress versus shear strain in a yield test

It may be noted from Fig. 7 that shear stress increases with an increase in shear strain to a maximum value and then stabilized at a certain shear strain value, which is a function of type of material. A drop of shear stress at which material failure to yield occurs, can be seen on the last limb of the curve. The results of yield tests at different percentages of strain for all crack sealants have been reported in Table III.

Table III
GRADING AT 150 PA SHEAR STRESS AND 100 % SHEAR STRAIN

Sealant type	Crack Sealants			Percentage rice husk in 80/100 Penetration grade		
	Sealant A	Sealant B	Sealant C	3%	6%	9%
Grade	76	82	70	64	70	76

It may be noted from Table III that with an increase in modifier (rice husk) percentage, the perfromance grade value increases. Yield test depicts an addition of percentage rice husk in the base 80/100 penetration grade bitumen.

C. Ranking of Crack Sealants

Based on the master curve date as developed through frequency sweep test the ranking of sealants was carried out to find the behavior of each material at different temperatures. The frequency and temperature sweep test and the yield test ranked different types of materials in similar order. A typical comparison of different sealants at 82ºC has been shown in Fig. 8.

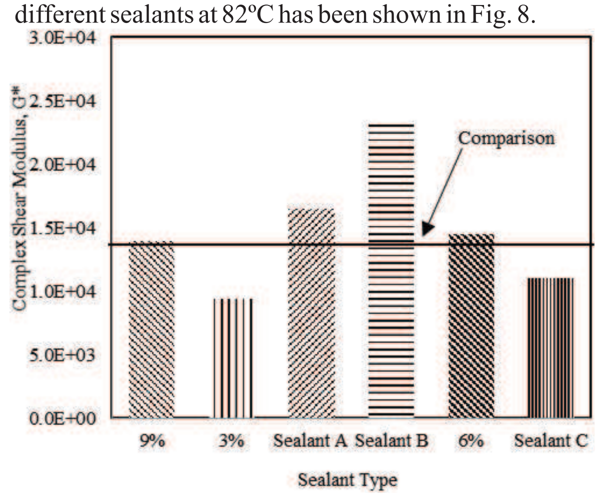

Fig. 8. Ranking of sealants based on complex shear modulus at 82ºC

It may be noted from Fig. 7 that the Sealant B showed the maximum value of G* at a stated temperature than sealant A and sealant C. The bitumen with 9% modifier produced higher value of G* and it decreased with a decrease in the percentage of modifier. At 3% modifier, the value of G* was minimum compared with different sealants. None of the sealants showed lower than 1 kPa of complex shear modulus at the stated temperature.

Present study highlights the effective utilization of local modifiers for sealing the pavement cracks. An addition of rice husk developed similar stiffness properties in bitumen as that of a crack sealants. The rheological results obtained from different tests showed similar grading of different materials being used in this study. It may be concluded from the present study that adding local modifiers such as rice husk provides a viable solution for pavement crack sealing, similar as using crack sealant products of different manufacturers.

IV. Acknowledgment

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V. Conclusion

This study presents characterizing the bituminous based sealants and a locally prepared bituminous based sealant by using rice husk. The criteria adopted to optimize percentage modifier was based on the linear visco-elastic range under a frequency and temperature sweep test. Yield test was also performed to confirm the results obtained from the other tests. The rheological testing phase was accomplished at intermediate and high temperatures, keeping in view the ambient temperature history. Following conclusions have been drawn from this study:

Developed master curves represent that the shear modulus has indirect relationship with the temperature as a decrease has been observed with an increase in temperature. Also, percentage increase in modifier increases the stiffness of sealants, which is in line with the results of previous studies. Sealant developed using rice husk showed a visco-elastic behavior. However, two different trends of master curves were obtained; curve and linear fits. Relatively stiffer sealants and bituminous sealant with 9% modifier showed linear fits, while the softer sealant showed a curve like trend. The percentage addition of modifier in the bitumen decreases the temperature sensitivity and increases the stiffness of the material. The conventional test results showed that both the penetration and ductility value of material had decreased with an increase in the percentage modifier. At the same time the softening point of sealant material had increased with an increase in percentage rice husk as modifier.

Based on the test data and plotted master curves at 46 ^{o}C, it was found that the stiffness in the form of complex shear modulus of bituminous sealant with 80/100+9% modifier yielded optimum value. With further increase in rice husk resulted in the brittleness of the material and causes a significant reduction in the flash point. The ranks obtained from the frequency and temperature sweep test and yield test were similar. Present study therefore recommends an optimum value of 9% rice husk to be used in the base 80/100 penetration grade bitumen for sealing the cracks in the pavements. With this percentage the modified bitumen was within the domain of sealants test results. The study thus recommends utilizing an effective and economical material instead of high cost crack sealants for hot climatic areas. This study reveals that both the ranking criteria of aggregate source yield similar results.

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