

FINAL REPORT FOR RMRC PROJECT 54

Project Title:	Synthesis of Use of Crumb Rubber in Hot Mix Asphalt					
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Project Objective:

GTR is a unique recycled material used in modifying asphalt binders. It is, however, not clear what the optimum method for incorporating GTR in asphalt is. Additional research can assist in developing the optimum methods of processing and blending GTR, reacting time between GTR and asphalt binders while blending, and the designing of GTR modified binders to yield similar, if not superior, performance to more conventional modified binders such as binder modified with SBS (Styrene Butadiene Styrene). There are also gaps in the knowledge regarding the merits of the solubility requirement in various binder specifications used currently, and there are concerns about using the PG grading system to compare GTR modified binders with polymer modified binders. This project is focused on adding knowledge to advance the understanding and appropriate use of GTR in asphalt modification.





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INTRODUCTION

Ground tire rubber (GTR) is a unique recycled material. Additional research to assist in developing optimum methods of processing and blending GTR with asphalts is required to yield the full potential of this material and may influence the more widespread use of GTR. If it can be shown that GTR-modified asphalt concrete has the potential to improve road performance, highway agencies may be less skeptical about incorporating the material into projects of various sizes, possibly even offering increased incentives for use of this waste product in an effective recycling process.

Various studies have been conducted to evaluate consequences of long term blending at high temperatures and dissolving the GTR completely in the asphalt binder. GTR has been shown in these studies to be the most effective in modifying asphalt binders when it is not completely dissolved, but swelled and adequately reacted with the binder (the fundamental concept of Asphalt-Rubber). There is also a contradiction between the attempt to advance the practice of implementing performance related concepts, such as PG grading, and the insertion of a solubility limit that does not appear to be justified for product performance. There is no published research to date that proves a lower solubility will lead to inferior performance. On the contrary, Asphalt-Rubber, has been produced and applied with 20% minimum TR content and has been very successful in providing extended service life of hot applied chip seals and hot mix asphalt, such as Asphalt-Rubber Composite Layering Systems (ARCLS).

This study started with a significant literature review and analysis of trends reported in the literature regarding GTR reaction time and attempts to dissolve it in asphalt. Also, the current practice of PG testing and grading developed by some agencies. The results of the literature review were summarized in a white paper format, and presented to the Binder Expert Task Group in Fall of 2010 to solicit feedback on the subject. The white paper is included in Appendix A of this report.

In addition to the literature review, an extensive experimental plan was conducted to compare different GTR modified binders (MB) were produced using recycled tire rubber by altering the percent modifier used, the reaction time allowed during the blending process, and the temperatures at which this blending was performed. This study investigates the variability in properties as the time exposed to elevated temperatures progresses as the potential for material degradation during this period may increase. Multiple percentages of GTR were used to modify the same base binder and each of these percentages was prepared at a range of reaction times. Each binder was characterized using current Superpave procedures, although it is argued that several testing procedures may not be appropriate for GTR-modified binders. Data collected in this study generally shows that standard Superpave procedures can be

used for GTR modified binders, resulting in effective binder characterization.

In addition to testing binders modified with GTR, binders and mixtures produced with other types of polymer modifiers were produced and tested. Mixtures were produced using one aggregate source and gradation mixed with four binders including the neat/unmodified binder, the neat binder modified with an elastomeric polymer, a plastomeric polymer, and with GTR. All three modifications were done to modify the base binder, which is a PG 64-22, to an equivalent PG grade of PG76-22. Repeated creep testing was performed on the mixes to determine if the elastic or plastic effects of corresponding binders was observed, and GTR mix response was compared to the response of mixes containing the polymer-modified asphalts to determine the elastic versus plastic effects of the GTR. In addition to mechanical testing, two-dimensional (2D) image analysis was performed on the mixtures to allow for quantification of the aggregate structure to determine if the decreased solubility of GTR influences the aggregate structure within the mix. This characterization was then compared to the mechanical performance to see if internal structure may serve as an indicator of a mixture's resistance to permanent deformation.

MATERIALS

Binders

Initial factors for consideration thought to contribute to the development of optimized methods and practice included comparison of GTR produced by various methods of grinding (ambient versus cryogenic) and comparison of maximum rubber particle size used in asphalt concrete modification (i.e., passing No. 30 mesh versus passing No. 80 mesh). These two factors were soon determined to be a nonissue after a detailed literature review. It was found that ambient grinding of tire rubber is far more common than cryogenic application of freezing and pulverizing. This distinction between methods and the degree of use is partly due to the associated costs of each method, leading to wider-spread selection of the more economical method. In ambient grinding of materials, a varying particle size distribution can be achieved and the maximum particle size can be selected. Although the maximum particle size is a controlled variable, to obtain a smaller maximum size a once ground tire rubber can be passed through the grinding process subsequent times. This clearly results in increased costs associated with a smaller particle size, as reducing the size results in a minimum of twice the time invested. For this reason, GTR of particle size passing No. 30 mesh was used in this study, as it can be obtained from a single grinding process and is thought to have a lower solubility than a smaller particle size. If this material can be tested using standard practice for modified binders, while resulting in meaningful data, smaller particle sizes should also produce acceptable results. All GTR-modification was completed using the wet process, which was a modification of the asphalt binder, and then adding the modified binder to aggregate for production of mixtures (as opposed to using the dry process, which would consist of adding the GTR

particles to the dry aggregate allowing it to act as a surrogate aggregate and is then blended with the asphalt to produce a mix). The wet process was selected as this will simulate a similar procedure to other binder modification, resulting in a modified product that can be characterized in the binder phase prior to incorporation in a mix.

Materials used for the binder phase of this study began with a single base binder (PG 64-16) and then expanded to a second base binder with the same high temperature grade but broader overall range of applicable temperatures (PG 64-22). These are to be used as unmodified binders and as well as modified with minus No. 30 GTR, elastomeric polymer, or plastomeric polymer. These modified binders were produced in the laboratory and also compared to a commercially available Tire Modified Asphalt Concrete (TMAC) terminal blend material, MAC-10TR, which is produced by incorporating 10% GTR in a pressurized vessel allowing for elevated temperatures and pressures. This results in complete solubility of the GTR. The polymer modifiers were used at percentages found to result in a single and double grade bump of the high temperature grade (to a PG 70-16 and PG 76-16, respectively). GTR was initially incorporated at 10 and 20% to cover the range currently used by the limited suppliers and contractors accepting of GTR products and later prepared at a percentage that would result in a similar grade to the alternate modifiers. Each percentage of GTR was prepared at 45, 65, and 85 minutes of blending/reaction time and later with the additional time of 360 minutes. The true-grade of each combination of percentage and reaction time was determined to see how the high temperature grading is effected by each factor. Reaction times for GTR-modification were measured following a 15 minute period dedicated for incremental introduction of the rubber particles into the binder. The terminal blend material was reheated and blended for 10 minutes to ensure homogeneity prior to pouring.

Mixtures

In addition to testing of the modified asphalt binders, gradations were determined for aggregates to be used for production of asphalt concrete mixtures. Two gradations were used, fine and coarse, which were determined by matching the broadest range allowed by WisDOT specifications. The fine and coarse grain-size distributions can be seen in Figure 54-1 accompanied by WisDOT control points. Aggregates were washed prior to batching for precise control over the volume of mineral filler in each mixture, and it can be seen in Figure 54-1 that mineral filler (minus 0.075mm) was adjusted to match between the two gradations, eliminating any significant influence of varying mastic properties.



Figure 54-1: Fine & coarse grain-size distributions used in production of asphalt mixtures (Coenen, 2011)

TESTING PROCEDURES

Binders

The un-modified, modified, and terminal blend binders were tested in the un-aged condition for rotational viscosity in the high temperature range $(135 - 175 \,^{\circ} \text{C})$ using the rotational viscometer (RV). RV testing was conducted at both 135°C (Superpave Volumetric Mix Design, AASHTO M 323-07) as well as 175°C as indicated by ASTM D 6114-97 (Standard Specification for Asphalt-Rubber Binder). Material was brought to the desired test temperature within 30 minutes and the spindle (SC4-27) was inserted and rotated for an additional ten minutes to ensure equilibration of the material. Data was then collected for approximately three minutes at six second intervals.

The initial viscosity testing of GTR-modified asphalt binders were conducted in the same manner as testing of the neat binder. However for a more complete understanding of GTR-modified asphalt binder behavior as a function of high temperatuer storage, a time sweep covering over five hours was used for data collection beyond the 30 minute ramp to test temperature and 10 minute equilibration time in an attempt to more accurately identify when a constant viscosity is observed, as well as possible cause for the initially observed reduction.

Also the binders were tested in the RTFO-aged condition for viscoelastic behavior defined by complex shear modulus (G^*) and phase angle (δ) at medium to high pavement temperatures using the dynamic shear rheometer (DSR). The results are used to determine the PG grade of each binder. Also, data for binder testing was collected from Repeated Creep and Recovery (RCR) and Multiple Stress

Creep and Recovery (MSCR) tests on the various binders. The RCR test was developed during the NCHRP 9-10 project. Bahia et al. (2001) recommended the repeated creep recovery test (RCR) using the dynamic shear rheometer (DSR) to evaluate the resistance of asphalt binders to permanent deformation. The repeated creep tests were performed at a constant stress rate, 100, 3200, and 10000 Pa, which is applied for 1-s followed by a 9-s rest period during which zero stress is applied, thus completing one cycle. A total of 1000 creep and recovery cycles were performed on RTFO aged binders. One thousand cycles was selected to capture full behavior of modified binder over a wide range of time. Accumulated strain is reported over the test period for each binder. The MSCR test was developed to reduce the number of samples at each stress level and it is the following development of RCR test and in its final shape, according to AASHTO TP70 standard "Standard Method of Test for Multiple Stress Creep Recovery (MSCR) Test of Asphalt Binder Using a Dynamic Shear Rheometer (DSR)" or American Society for Testing and Materials standard (ASTM) 7405, the test consists of 10 cycles of 0.1 kPa stress creep and recovery, followed immediately by another 10 cycles of 3.2 kPa stress creep and recovery. All binder testings' were conducted at temperatures of 46, 58, and 70°C.

Mixtures

Mechanical testing of mixtures was conducted in accordance with standard protocol for repeated creep testing, commonly known as flow number. The flow number is defined as the number of cycles required for the sample to begin exhibiting tertiary creep, or flow, which is more clearly defined as the number of cycles which corresponds to the minimum rate of change in permanent axial strain of the specimen under a repeated load test. When plotting the total accumulated strain versus number of cycles, a distinct primary (I), secondary (II), and tertiary (III) zone is identified on the creep curve as seen in Figure 54-2. The transition from secondary to tertiary creep corresponds to the minima of the rate of change of axial strain as shown in Figure 54-3.



Figure 54-2: Typical creep curve for an HMA mixture (Coenen, 2011)



Figure 54-3: Plot of rate of change of permanent axial strain versus cycles for a typical HMA mixture (Coenen, 2011)

The flow number test consists of repeatedly loading a sample under stress control, with a 0.1 second pulse of a prescribed stress, followed by a 0.9 second rest period. This cyclic, compressive loading is intended to simulate repeated passing of traffic/vehicles over the roadway and, when testing for resistance to permanent deformation, is continued until failure occurs. Failure is defined by clearly exceeding the transition from secondary to tertiary creep and when the sample has lost the capacity to withstand the prescribed stress. The test is performed on a 100 mm diameter specimen of 150 mm in height, produced by cutting and coring a standard gyratory specimen. Specimens for this study were

compacted to a consistent air void content or density by targeting 6.5 + 0.5 percent air voids. Asphalt mixture testing was conducted at 46 °C (i.e., the 7-day average high pavement temperature in the region where testing was conducted). The 7-day high temperature is chosen since this research is focused on resistance to permanent deformation, which is a consequence of high temperatures and heavy loads. Heavy loads can be seen as arbitrarily defined. For this reason mixture testing was conducted at two stress levels, 50 and 150 psi (345 and 1034 kPa) intended to span the average stress level found in field measurements.

Image Analysis

This part of the study utilized state of the art software (iPas, Image Processing & Analysis System) to determine the internal structure of HMA. In this study, the total number of occurrences of aggregate-to-aggregate "contact" within the matrix is more appropriately defined as the aggregate proximity index (API). Since the precise point of contact within a 3-dimensional matrix is rarely captured in a 2-dimensional slice of the specimen, a specified separation distance must be defined. The 2-D image will not show the perimeter of aggregates to be within contact unless the specimen was sliced immediately adjacent to the 3-dimensional contact point. For this reason, any distance larger than immediately adjacent is seen as a small distance between aggregate perimeters in a planar image. This distance is defined in this study as the surface distance threshold (SDT) value and it is a user defined value which is adjustable based on resolution of the planar image. By defining the SDT and minimum aggregate size to consider for calculation of total number of occurrences, the software searches the entire image for locations that meet criteria. In other words, any time that the perimeter of two aggregates larger than the selected minimum aggregate size are within the SDT from one another, an occurrence is tabulated. For images of both coarse and fine gradations used in this study, processing of the image was intended to remove aggregate that were fully consumed by the binder and may be viewed by the software as the mastic phase rather than aggregate. This distinction was made by determining the intensity spectrum for images of each gradation and defining the localized maxima as air voids, mastic, and aggregate (from low to high intensity, respectively), and then re-processing the image using a threshold just below the start of aggregate intensity in the spectrum (i.e. threshold value of 150). This results in a processed image with clearly defined aggregate. The viewing level can then be zoomed in iPas to determine the approximate diameter of remaining aggregate in the image. This was done for both the coarse and fine gradations, resulting in aggregate equal to or greater than a diameter of 0.6 mm remaining in the processed image. In order to account for an equivalent percent retained for each gradation, a minimum aggregate size of 0.6 mm was selected for images of fine gradation and 2.36 mm for coarse

gradation. This was combined with an SDT of 0.15 mm (determined from median filter selection and image resolution) for analysis of API.

RESULTS

Binders

Results for the neat binder testing show consistent values of viscosity as a function of testing time at constant temperature. Also the viscosity decreased as temperature increased (from 0.4 Pa*s at 135°C to just over 0.07 Pa*s at 175°C), as seen in Figure 54-4.



Figure 54-4: Viscosity of Valero 64-16 asphalt binder (neat)

The results of 10% GTR blended for 45 minutes are shown in Figure 54-5. It can be seen from in this figure that a constant viscosity was not observed after the 30 minute ramp to test temperature followed by ten minute equilibration period. The first three minute data collection period did not extend far enough to capture the time at which a constant viscosity is achieved. Therefore, data was collected for a second three minute period following an additional 20 minutes of equilibration (resulting in a combined 30 minute equilibration period) beyond the results of Figure 54-5. The additional data can be seen in Figure 54-6. Figure 54-5 clearly shows the viscosity steadily reducing from approximately 1.85 to nearly 1.75 Pa*s while Figure 54-6 shows a constant viscosity of approximately 1.7 Pa*s.



Figure 54-5: Viscosity of GTR-modified asphalt binder with 10% GTR and blending time of 45 minutes (10 minute equilibration)



Figure 54-6: Viscosity of GTR-modified asphalt binder with 10% GTR and blending time of 45 minutes (30 minute equilibration)

The results of the five hour time sweep for viscosity of 10% GTR at temperatures of 135 and 175°C can be seen in Figures 54-7 and 54-8, respectively.



Figure 54-7: Change in viscosity of GTR-modified asphalt binder as a function of time at 135 after modification with 10% GTR using ablending time of 65 minutes at 135°C.



Figure 54-8: Change in viscosity of GTR-modified asphalt binder with 10% GTR as a function of time and afterblending for 65 minutes at 175°C

Figures 54-7 and 54-8 clearly indicate a difference in behavior at different temperatures for the binder that was modified with GTR in the laboratory. These results are also very different than the results of viscosity testing of terminal blend asphalt binder with 10% GTR that are shown in Figure 54-9. The terminal blend viscosity measurements were recorded over a period of two hours in order to span the time period where laboratory blended material showed varying behavior at two temperatures. It is clear that the terminal blend asphalt has a very stable viscosity as a function of time at both temperatures.



Figure 54-9: Viscosity of terminal blend asphalt-rubber binder with 10% GTR (TMAC MAC-10TR)

Analysis of binder results

The results collected during the initial phase of this project indicate that reaction time has major effects on viscosity of GTR-modified binders. It is found that there is a possibility for segregation of rubber particles during the early times but, after a certain period of time, a rapid increase in viscosity takes place, which is indicative of reaction/swelling of the rubber. Figure 54-7 depicts this behavior as it shows an initial decrease followed by a short period of stable viscosity readings similar to that observed between Figures 54-5 and 54-6. However, at times longer than 30 minutes, an increase in viscosity is observed. This trend changes with temperature, and, as seen in Figure 54-8, when the testing temperature is increased from 135 to 175°C, the decrease and stability were no longer seen, though the same perpetual increase in viscosity was identified at both temperatures. It is clear from Figures 54-7 and 54-8 that the time required for this swelling is temperature dependent. To support this hypothesis of swelling of the rubber particles, the terminal blend material was tested at 135 and 175°C as terminal blend material is said to be fully reacted prior to shipment. Figure 54-9 confirms that stable/constant viscosity measurements are obtained from terminal blend material immediately following a 30 minute ramp to test temperature and ten minute equilibration period. The viscosity of the terminal blend material remained relatively constant over the duration of the two hour data collection as well.

These observations of changing properties with extended periods of time at elevated temperatures match that of the previous studies reviewed. Such studies (Zanzotto and Kennephol 1996, Bullin et al. 1996, and Glover et al. 2000) indicate that this change in properties could actually lead to elimination of the benefits of GTR modification. The studies suggest that the change in properties is not only due to the

swelling of the rubber particles, but that there is degradation of the rubber itself which is leading to a diminishing positive influence of adding the GTR to the asphalt binder. Such studies will be summarized in greater detail in a White Paper that is in preparation regarding "The Effects of Digesting Crumb Rubber in Modified Binders" intended to supplement this report.

The increase in viscosity readings, as observed in Figure 54-8, show that though the increase continued for times approaching more than 300 minutes, a reduced rate of change was observed after approximately one hour. The decrease in rate suggested a constant reading could be reached. For this reason, an additional blend beyond the original blend times of 45, 65, and 85 minutes was produced using a blending time of 360 minutes to see if blending/reaction time for the rubber influences this rate of change in viscosity readings. The results of the extended blending time are shown in Figure 54-10, which indicates that the Rotational Viscometer at 175°C mimicked that of the shorter blending times, as seen in Figure 54-8. Further consideration suggests that if the increased reaction time does not stabilize the viscosity measurements, perhaps the cooling of each sample after pouring in the test cylinder for RV causes the variability. Then the behavior seen in Figures 54-8 and 54-10 may be influenced by the reheating cycle during the 30 minute equilibration period to bring the material to 175°C. To test this hypothesis, a new blend of 10% GTR was produced using a blending time of 45 minutes. Replicate RV samples were taken immediately following blending time and the remainder of the material was kept at the elevated temperature in an oven. Additional samples were pulled from the material at 50, 100, 200 and 300 minutes after brief agitation, and the samples were then tested for immediate viscosity readings during three minute data acquisition period following ten minutes of rotating the RV spindle as prescribed by ASTM. The first three readings from immediately following blending, 50-minute pull and 100-minute pull can be seen in Table 54-1.



Figure 54-10: Viscosity of GTR-modified asphalt binder with 10% GTR and blending time of 360 minutes at 175°C

Table 54-1: Viscosity of GTR-modified asphalt binder with 10% GTR and	
blending time of 45 minutes at 175°C, samples pulled after storage at elevated temperatur	re

"Pull"	Time	Viscosity
(after blend	ing)	(Pa*s)
0		0.399
50		0.488
100		0.487

Table 54-1 shows that visosity measurements begin to show stability prior to 300 minutes of testing with the RV when material is stored at an elevated temperature and instantaneous viscosity measurements are taken, but the roughly 20% increase in viscosity readings from immediately following blending time to 50 minutes later still suggest an influence beyond blending or reaction time provided by elevated temperatures.

The temperatures used for PG-grading ranged from 64 to 88°C and the MSCR test was conducted at 58°C. It can be seen in Table 54-2 as well as Figure 54-11 that 10% GTR provided an average increase of three grades (+18°C change) from the original binder (referred to as three *grade bumps*) whereas the terminal blend material only provided two grade bumps for the same amount of GTR. The change indicates an increase of approximately two degrees improvement in grade per 1% GTR for laboratory blended binders. Additional calculations show that this is equivalent to approximately 3% GTR per desired grade bump (based on PG grading intervals of six degrees). This allows for a more direct comparison with the polymer modified binders presented in Figure 54-11 and Table 54-2. Results show that 2% SBS results in a single grade bump and addition of a cross-linking agent at manufacturer's specifications increases the pass/fail temperature by a couple degrees but still does not cross the threshold of the next PG. Thus, true-grade testing indicates that 2% SBS (polymer) provides a one-grade bump and the addition of a cross-linking agent at additional cost does not change the PG grade of the material. Similarly, 10% GTR provides roughly a three-grade bump which indicates that for the binder used in this study, a 3-4% GTR can give a one grade bump. In other words, GTR content required for one grade bump is twice that required for the SBS polymer, though the virgin polymer has a significant cost associated with it whereas the GTR is a secondary use of a disposed material.

 Table 54-2: True-grade (Performance Grade, PG) of materials as defined by pass/fail temperature resulting in a G*/sinδ measurement of 1.00 kPa

Binder- React	Run 1	Run 2	Average	Std. Dev	COV	PG Grade	Bump
Valero Neat	67.4	67.2	67.3	0.141	0.2%	64	0
10GTR_45min	85.1	83.9	84.5	0.849	1.0%	82	3
10GTR_65min	84.7	83.9	84.3	0.566	0.7%	82	3
10GTR_85min	83.4	84.0	83.7	0.424	0.5%	82	3
10GTR_360min	82.3	81.4	81.9	0.636	0.8%	76	2
PG -10TR	78.7	78.8	78.8	0.071	0.1%	76	2
Valero + 2% SBS	72.9	73.0	72.9	0.071	0.1%	70	1
2% SBS + XL	75.4	75.4	75.4	0.000	0.0%	70	1



Figure 54-11: Pass/fail temperatures of laboratory blended materials

Material distinction can also be made using the MSCR test results as seen in Figures 54-12 and 54-13. Figure 54-12 shows the comparison of 10% GTR laboratory blends versus the terminal blend material with the same percentage of rubber by mass as well as the neat binder. It is clearly seen in the figure that at each stress level, the neat binder exhibits the least recoverable strain per cycle, and although the terminal blend resists a small portion of the strain seen by the neat binder at given stress levels (indicated by a vertical shift of the curve), the terminal blend also shows similar response in terms of permanent versus recoverable strain per cycle of loading (indicated by parallel curves). When looking at the 10% GTR blends produced using 45, 65, and 85 minutes for blending times, all show some improvement by lowering the strain exhibited at each stress level, although 65 minutes results appear to be far superior to 45 and 85 minutes. The shortest blending time of 45 minutes shows slight decrease in strain and a slight increase in recoverable strain per cycle, but a significant difference is seen when blending time is increased to 65 minutes, clearly seen in Figure 54-12. The strain is decreased by nearly an order of magnitude, and a majority of total strain per cycle can be seen as recoverable rather than permanent. The trend of increase does not continue for the 85 minute blending time, as the results for the 85 minute blending time appears to revert the material response back near what was seen for the 45 minute blend, which does not differ a great deal from the neat binder and terminal blend.



Figure 54-12: MSCR (time vs accumulated strain) at 58°C for neat binder, terminal blend and 10% GTR laboratory blends



Figure 54-13: MSCR (time vs. accumulated strain) at 58°C for neat binder, terminal blend and 20% GTR laboratory blends

Figure 54-13 includes the results for the 20% GTR blends. When 20% rubber was used, blending times did not show a significant difference in response relative to blending time but did show a significant improvement in recoverability as compared to the neat binder and the terminal blend. In fact, the grouping of the three blending times for 20% GTR shows nearly the same response shown by the 65 minute blend time when 10% GTR was used.

A base binder with similar PG grade was used to justify the effect of base binder on MSCR

results. Flint Hills binder (PG 64-22) was used as the base binder, and several modified versions of this base binder were also tested. Figure 54-14 shows the summary of MSCR results at 58 °C.



Figure 54-14: MSCR (time vs. accumulated strain) at 58°C for neat binder, plastomeric, elastomeric and GTR modified binders (Golalipour, 2011).

Figure 54-14 clearly shows that the behavior of the new base binder is very similar to the previously tested base binder. Also, it can be seen from the above figure that the behavior of different modified binders are very similar. The accumulated strain for all three modified binders is approximately equal, so it can be concluded that GTR modification can have the same behavior as other modifiers. MSCR testing for these binders was also conducted at 46 °C. Because the binder data would be correlated to mixture data, results at this temperature give a better indication of their performance. Figure 54-15 shows the MSCR results at 46 °C.



Figure 54-15: MSCR (time vs. accumulated strain) at 46°C for neat binder, plastomeric, elastomeric and GTR modified binders (Golalipour, 2011).

Also, the RCR test was conducted to see more complete behavior of binder. RCR tests were done at three stress levels to observe the effect of stress levels on binder behavior more clearly. Because this data should be correlated to mixture results, it was necessary to have wide range of binder behavior at different stress levels. RCR tests were conducted at the same temperature as mixture testing. The following figures show the RCR results at different stress levels.





Figure 54-16: RCR (time vs. accumulated strain) at 46°C for neat binder, plastomeric, elastomeric and GTR modified binders at three stress levels

RCR results show that GTR is a good candidate for modifying binder especially at high stress levels. It can be seen from Figure 54-16 that GTR modified binder has a very stable trend at different stress levels. Changing stress levels does not have a large influence on behavior of GTR modified binder.

Mixtures

Flow number (FN) testing was conducted for both fine and coarse gradations of limestone using each of the four binders at stress levels of 344 and 1034 kPa. Mixture testing was conducted at a temperature of 46° C. A summary of the test results for limestone mixtures is shown in Table 54-3.

	Stress (kPa)	
Sample	344	1034
Neat-Coarse	450	100
Neat-Fine	730	150
GTR-Coarse	690	160
GTR-Fine	1575	240
Plastomer-Coarse	3000	210
Plastomer-Fine	50000+	400
Elastomer-Coarse	1050	260
Elastomer-Fine	3150	430

Table 54-3: Summary of FN results for limestone mixtures compacted to $6.5\pm0.5\%$ air voids.

Results show consistently that the fine gradation had a greater FN value than the coarse gradation for each of the binder types and at both stress levels. Also, the FN values show consistent reduction when the stress level was increased. The results also show clearly that the mixtures with plastomer modification exhibited higher FN values than those with elastomeric modification at the lower stress level. In fact, the fine gradation with plastomeric binder did not exhibit tertiary flow within a 50000-cycle loading duration. However, this ranking changed at the increased stress level and the mixtures with elastomer and plastomer modifiers show very similar FN values that are still better than the neat asphalt and the GTR modified asphalt. These results suggest that there may be a shift in the component that governs mixture performance when changing from the low to high stress.

An example of the results obtained from FN testing at 344 kPa is shown in Figure 54-17. As shown in the plots, the plastomer with coarse gradation is performing similarly to the elastomer with fine gradation. The plastomer with fine gradation, however, lasted more than 50000 cycles without showing tertiary flow and is thus not shown in the plot.



Figure 54-17: Graph. FN results for limestone mixtures at 344 kPa.

It can be seen from Figure 54-17 and Table 54-3 that GTR-modification improves the performance beyond that of the unmodified binder but falls short of either virgin polymer modification. This holds true regardless of stress level or gradation. Because of the decreased solubility of the GTR particles, the GTR-modified binder may be considered a unique modification when compared to the virgin polymers. Consequently, it is necessary to investigate internal structure of the mixes to determine if this uniqueness is altering the structure in such a way that may be limiting the GTR mixes from performing to the level of the elastomer and plastomer polymers.

Image Analysis

To improve the accuracy of 2-dimensional images used to represent a 3-dimensional specimen, a total of six images for each specimen were analyzed. These six images come from opposing faces of three physical cuts that divide the specimen into four equi-volume slices, V1=V2=V3=V4 (based on the dimensions of the cut and cored samples, though for ease of cutting the imaging specimens were not cut and cored). This technique and locations of slices as described here can be seen in Figure 54-18.



Figure 54-18: Location of slices for planar imaging of specimens

The images were analyzed to calculate the Aggregates Proximity Index (API) per 100 square centimeters (API/100cm²), which was used to represent the internal aggregate structure of each mix type. This information is tabulated below.

Mix Type	API/100cm ²
LSC_CBE	103
LSC_GTR	80
LSC_Neat	64
LSC_SBS	114
LSF_CBE	298
LSF_GTR	291
LSF_Neat	280
LSF_SBS	301

 Table 54-4: Average API/100cm² for limestone mixes

It can be seen in Table 54-4 that the binder modification type has a significant effect on the API values, and that API values of modified binders are higher than the neat binder in both gradations. Also, fine gradation (LSF) mixtures show consistently greater API values than the coarse gradation (LSC)

mixtures, even when minimum aggregate size was selected to account for equivalent percent of aggregate retained. Furthermore, the ranking of the API within each gradation is similar to that obtained from mechanical testing of the mixtures. This finding led to investigating the relationship between internal aggregate structure and performance. This is important to determine if perhaps the structure may act as an indicator of the performance, and also to determine if the GTR structure is an outlier when compared to the unmodified and virgin polymer-modified response and structure. Correlations of FN with API are shown in Figure 54-19.



Figure 54-19: API versus flow number at 1034 kPa for (a) coarse and (b) fine gradation mixes

It can be seen from Figure 54-19 that there is a high correlation between the internal aggregate structure and the mechanical performance (resistance to permanent deformation) conducted at the higher stress level of 1034 kPa. The correlations of relationship diminish when comparing API to mechanical performance at the lower stress level, suggesting the increased influence of binder properties at the lower stress applied to mixtures.

Figure 54-20 compares the accumulated strain of the mixtures at the last recorded cycle for the lowest FN for that gradation. This cycle count was selected to capture the mixture behavior as late in the mechanical test as possible, without changing the reference cycle between binder types. For this reason, cycle 800 for coarse gradation and cycle 2000 for the fine gradation was chosen. Accumulated strain was chosen to eliminate any loss of information caused by parallel curves in mixture the mixture data. Since mixture testing was conducted for several thousand cycles in some cases, the total accumulated strain at any given point may be different for various binder types, while the plot of accumulated strain versus cycles may show the curves to be parallel to one another indicating similar recoverable and non-recoverable components of the response within each cycle. The non-recoverable creep compliance (Jnr) was appropriate for the binder when using data collected from the MSCR test described earlier because the MSCR test is conducted for 10 cycles at each stress level and clearly distinguished between the binder Jnr and the mixture accumulated strain suggests that binder properties have a significant influence on the mixture behavior at the 344 kPa testing of the mixtures.



Figure 54-20: Binder Jnr versus mixture accumulated strain at 344 kPa for (a) coarse and (b) fine gradation mixes

This data, in combination with the API acting as an indicator of mixture performance at the 1034 kPa stress level, suggests that this study may have captured the transition of critical component dictating mixture performance as the stress level applied to a pavement is increased from 344 to 1034 kPa. It appears that binder properties may play a more significant role when the pavement is loaded at a lower stress level but that the load carrying component may shift in favor of the aggregate as the stress

experienced by the pavement increases. This is clearly only the beginning of investigating the stress level at which this transition may occur and if further improvements to 2-dimensional imaging may allow for better characterization, specifically clearer distinction between various mix designs (as it can be seen that API values fall in a very narrow range, specifically for the fine gradation mixes).

SUMMARY OF FINDINGS

The data presented in this report clearly indicates that GTR-modification of asphalt binder has the potential to improve the rutting resistance performance of the binder as well as mixtures incorporating the GTR-modified asphalt binder. However it is clear that the GTR modification used in this study could not produce superior or even equivalent performance to that of the virgin polymer modifiers in terms of rutting resistance.

The study results indicate that properties of GTR modified binders vary depending on amount of GTR used and reaction period. Thus the comparison to the use of virgin polymers in modification is very complex and requires further qualification. There is potential that GTR amount and method of reaction could lead to results that are equivalent or superior to virgin polymers. Also, considering the cost and environmental benefits of using GTR could show further advantages to GTR use.

State of the art internal structure image characterization of HMA samples has shown that the reduced levels of solubility of GTR do not significantly influence the aggregate structure in a manner different to that seen in the unmodified or virgin polymer modifiers. This characterization appears to work equally well for the GTR material and has indicated that internal aggregate structure may in fact act as a performance indicator, at least at higher stress levels. This study has shown through testing of binders and mixtures at various stress levels that a transition in rutting resistance may take place between loadings of 344 to 1034 kPa. Data suggests that binder properties play a significant role in dictating pavement performance at lower stress levels but that this significance is shifted to aggregate structure as stress is increased. Information presented here also distinguished between coarse and fine gradations, and indicates that binder modification could have significant effect on aggregate structure, particularly for mixtures produced with coarse gradation. This finding indicates that binder modification may be extremely beneficial in performance of stone matrix asphalt (SMA), which utilizes coarse and gap graded aggregates.

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APPENDIX A: WHITE PAPER ON EFFECTS OF DIGESTING CRUMB RUBBER IN MODIFIED BINDERS



White Paper on The Effects of Digesting Crumb Rubber in Modified Binders (MB)

February 2011

Ву

The University of Wisconsin-Madison Asphalt Research Group

In Collaboration with the Recycled Materials Resource Center

Executive Summary

With the advent of new processes for incorporating varying amounts of scrap tire rubber (TR) into binders for hot mix asphalt and seal coats, there appears to be a lack of clarity about the best method of using TR. There is also a lack of clarity in the identification of the products produced using various methods. One of the basic core variables of incorporating TR into asphalt binder is the level of digestion of the crumb rubber into the binder. The range in methods is from the extreme of full digestion of the TR in binder to the dry mixing of TR with aggregates with minimal digestion. The two extremes have their advantages and disadvantages. The partial digestion method (the TR is partially digested in the binder to create a gel-like composition through a specific time temperature reaction), which is the more established process, is called Asphalt-Rubber (AR). Asphalt-Rubber is clearly defined in the ASTM and other widely used standards (See footnote ⁽¹⁾ below). In recent years claims regarding the value of higher level of digestion have resulted in new products. Also, the test of solubility, to ensure the higher level of digestion, has been instituted in specifications.

The purpose of this paper is to summarize the results of important past studies evidencing that the complete digestion of TR in an asphalt binder does not necessarily improve the binder characteristics. The data collected from these studies convincingly demonstrates that there is a high risk in too much digestion, as it can virtually eliminate the stiffening and elastic influence of the TR on binder properties.

It has also been claimed that the need for full digestion is to allow use of the current PG binder testing system for these binders. This study, however, shows that Asphalt-Rubber binders (with partial digestion) have been successfully tested utilizing existing equipment for binder performance grading (PG) with or without minor modification of test geometries.

The data presented in this study indicates that there is no performance related benefits that merit requiring solubility testing, which is a test method required only to distinguish asphalt products with a high level of digestion from the more conventional asphalt rubber products.

The study recommends the appropriate use of the PG grading system for products such as Asphalt-Rubber. Such testing will allow performance based comparison of these different products, leading to better practice and more choices for designers and policy makers responsible for long lasting pavements and effective recycling of TR.

⁽¹⁾ Asphalt-Rubber binder describes rubber particles of specific size and properties added to and partially digested in asphalt

cement over an extended time, meeting specific proportion and test properties including minimum viscosities. Asphalt-Rubber is defined by both the American Society of Testing Materials (ASTM) D8-02, definition ASTM specification 06114/D6114 M-09, the Standard Specifications for Public Works Construction, The "Greenbook" Sections 203-11, 203-12, 302-9, 302-10, and Caltrans Standard Specifications Section 37-030 dated June 5, 2009.

Background and Need for this Paper

The binder specification currently utilized by Caltrans for modified binders (MB) also known as Terminal Blend in Hot Applied Chip Seal applications is shown in Table 1. The table shows that the MB produced with polymers and MB produced with tire rubber (TR) meet the same testing criteria, with the exception of minimum solubility.

This report is written to discuss the merits of the solubility requirement and document examples of studies conducted to evaluate consequences of dissolving the TR virtually completely in the asphalt binder. TR is shown in a number of studies to be the most effective in modifying asphalt binders when it is not completely dissolved, but swelled and adequately reacted with the binder (the fundamental basic concept of Asphalt-Rubber) ⁽¹⁾. It is also recognized that solubility is not a performance related property and many two-phase binders can be very effective in providing enhanced performance.

There is also a contradiction between the attempt to advance the practice of implementing performance related concepts, such as PG grading, and the insertion of a solubility limit that does not appear to be justified for product performance. There is no published research to date that proves a lower solubility will lead to inferior performance. On the contrary, Asphalt-Rubber, has been produced and applied with 20% minimum TR content and has been very successful in providing extended service life of hot applied chip seals and hot mix asphalt, such as Asphalt-Rubber Composite Layering Systems (ARCLS)⁽²⁾.

The other significant issue contained in the current MB specification is the lack of verification concerning the TR content. This lack of a control test opens the opportunity for not using rubber at all, or adding just a small amount that will pass the solubility requirement. Most MB specifications state minimum TR content requirements ranging from 8-10 percent. In fact, the solubility requirement discourages using rubber.

⁽¹⁾ Asphalt-Rubber binder describes rubber particles of specific size and properties added to and partially digested in asphalt cement over an extended time, meeting specific proportion and test properties including minimum viscosities. Asphalt-Rubber is defined by both the American Society of Testing Materials (ASTM) D8-02, definition ASTM specification 06114/D6114 M-09, the Standard Specifications for Public Works Construction, The "Greenbook" Sections 203-11, 203-12, 302-9, 302-10, and Caltrans Standard Specifications Section 37-030 dated June 5, 2009.

⁽²⁾ Asphalt-Rubber Composite Layering Systems (ARCLS) refers to a multiple layering system utilized in overlay strategies typically consisting of cold planning, a conventional leveling course, an Asphalt-Rubber Aggregate Membrane Interlayer (ARAMI) followed by final cap of Asphalt-Rubber Hot Mix (ARHM). The system ranges in total thickness from 2 inches to 3 inches depending upon if a leveling course is incorporated into the layering design.

One of the unique advantages of Asphalt-Rubber is it allows recycling of a waste product, and effectively saving the environment from accumulation of this solid waste by improving paving asphalts. The lack of specific requirements for tire rubber amount used defeats this important recycling advantage, and in fact discourages producers from using more rubber. This could put the solid recycling programs in California at risk of diminishing.

In recent years proprietary technologies have been developed which claimed that TR can be included in asphalts at higher concentrations but with using heat and shear it can be devulcanized to perform similar to polymer modified binders. Research however has shown that this is a risky proposition since TR could lose its effectiveness if it is highly de-vulcanized. It is suspected that in these newer products, which can meet the PG 76-22 TR specification of Caltrans, either lesser rubber is being included, or polymers are added to substitute for the lost effects of the rubber due to de-vulcanization. In either case it seems to be counterproductive because either very little TR rubber is recycled, or significant energy is wasted in de-vulcanizing and using virgin, high cost materials such as virgin polymers (e.g. SBS).

This report is prepared to shed some light on the subject of tire de-vulcanization and the consequences of dissolving tire rubber on critical properties of binders. It is based on a critical review of literature in which the effect of de-vulcanization, also called de-polymerization, has been carefully studied using rheological measurements similar to those used in the Caltrans specifications, as well as others.

		Grade		
Property	AASHTO Test Method	PG	PG	
		76-22 PM	76-22 TR ^b	
Original Binder	÷	<u>.</u>	<u>.</u>	
Flash Point, Minimum °C	T 48	230	230	
Solubility, Minimum % ^c	T 44 ^c	98.5	97.5 ^d	
Viscosity at 135°C,	T 316			
Maximum, Pa [.] s		3.0	3.0	
Dynamic Shear,	T 315			
Test Temp. at 10 rad/s, °C		76	76	
Minimum G*/sin(delta), kPa		1.00	1.00	
RTFO Test,	T 240			
Mass Loss, Maximum, %		1.00	1.00	
RTFO Test Aged Binder	·			
Dynamic Shear,	T 315			
Test Temp. at 10 rad/s, °C		76	76	
Minimum G*/sin(delta), kPa		2.20	2.20	
Dynamic Shear,	T 315			
Test Temp. at 10 rad/s, °C		Note f	Note f	
Maximum (delta), %		80	80	
Elastic Recovery ^g ,	T 301			
Test Temp., °C		25	25	
Minimum recovery, %		65	65	
PAV ^h Aging,	R 28			
Temperature, °C		110	110	
RTFO Test and PAV Aged Binder	•			
Dynamic Shear,	T 315			
Test Temp. at 10 rad/s, °C		31	31	
Maximum G*sin(delta), kPa		5000	5000	
Creep Stiffness,	T 313			
Test Temperature, °C		-12	-12	
Maximum S-value, MPa		300	300	
Minimum M-value		0.300	0.300	

Table 1. Modified Binder Specification for Hot Applied Chip Seal Applications ^a

Notes:

a. Do not modify binder using acid modification.

b. Supplier is required to certify 10% minimum scrap tire rubber modifier in binder.

c. The Department allows ASTM D 5546 instead of AASHTO T 44

d. For hot applied chip seal applications the solubility for field blended material will be a minimum of 93% and a binder profile at the temperature utilized for field production is required for suppliers,

e.

f. Test temperature is the temperature at which G*/sin(delta) is 2.2 kPa. A graph of log G*/sin(delta) plotted against temperature may be used to determine the test temperature when G*/sin(delta) is 2.2 kPa. A graph of (delta) versus temperature may be used to determine delta at the temperature when G*/sin(delta) is 2.2 kPa. The Engineer also accepts direct measurement of (delta) at the temperature when G*/sin(delta) is 2.2 kPa.

g. Tests without a force ductility clamp may be performed.

h. "PAV" means Pressurized Aging Vessel.

Studies on De-polymerization of Ground Tire Rubber in Asphalt Binder

The existing technologies of using scrap tires in asphalt pavements are recognized to be of two types: the dry and the wet technologies. In both cases the rubber is intended to be used as elastomeric filler. In the dry process, the rubber is assumed to form part of the aggregate component. In the wet process, which is used in Asphalt-Rubber, however, some of the three dimensional network of the vulcanized rubber is expected to de-polymerize or dissolve in asphalt. Most of the rubber remains intact with a somewhat loosened rubber matrix, swollen by the asphalts' oils. It is also known that the crumb rubber/asphalt mixtures have to be prepared under controlled conditions such that the danger of complete de-polymerization will not lead to the CRM's total digestion into the binder. There is, therefore, an optimum blending time and process during which rubber is incorporated effectively in a binder. Producers of Asphalt-Rubber binders today are very skillful in optimizing the use of high rubber amounts and incorporating it in asphalt binders for effective modification of performance properties of asphalts.

Another process for recycling rubber in asphalts that has been studied is called rubber pyrolysis. This process is used in terminal blend binders. In this process, heat and shear are used for the decomposition of vulcanized rubber to carbon black, gas, and unsaturated oils. This decomposition destroys the elastomeric behavior that is critical for the modification of asphalt binders. This destruction of the rubber has been shown to deteriorate the performance properties of binders in a few important studies that covered a wide range of mixing conditions and types of binders (Zanzotto and Kennepohl 1996, Bullin et al. 1996, and Glove et al., 2000). In one of these studies, authors also reported on attempts to at least partially re-polymerize the oils resulting from tire rubber pyrolysis in laboratories in order to recover some of the elastic effects needed, but indicated that such attempts could not achieve any success (Zanzotto and Kennepohl 1996).

In the following sections some of the critical data from these reports are shown and analyzed. The full citations of these reports are as follows:

- 1. Zanzotto, L. and G. J. Kennepohl, "Development of Rubber/Asphalt Binders by Depolymerization/Devulcanization of Scrap Tires in Asphalt", Transportation Research Record 1530, TRB, pp 51-58, 1996.
- 2. Bullin, J., et al., " DEVELOPMENT OF ASPHALTS AND PAVEMENTS USING RECYCLED TIRE RUBBER, Phase I: Technical Feasibility, Progress Report, Texas Transportation Institute, 1996.

3. Glover, C et al., "A COMPREHENSIVE LABORATORY AND FIELD STUDY OF HIGH-CURE CRUMB-RUBBER MODIFIED ASPHALT MATERIALS, Texas Transportation Institute, The Texas A&M University System College Station, Texas 77843-3135, Project No. 0-1460, November 2000

There could be a number of other studies that have covered this subject; these three selected reports appear to give sufficient information about the consequences of over reacting the rubber in asphalts and explain some of the risks involved in attempting to dissolve virtually all of the tire rubber.

Important Data Collected and Analysis

Study by Zanzotto and Kennepohl

In this study three forms of tire rubber (60 mesh, 20 mesh, and buffed rubber) were subjected to different combinations of heat and high shear for various times to study the effects of depolymerization on rheological properties. The properties measured included creep and recovery, viscosity, and some conventional tests. In addition, the amount of un-reacted rubber and tendency to separation during the hot storage were also tested.

Figure 1 is an example of the results reported in which the trend of deteriorating high temperature properties is clearly shown in terms of softening point as a function of increasing temperature of reaction for standard time of 2 hours. It is important to note that the softening point of the base asphalt is 36 deg C. As shown in Figure 1, at 260 C (500 F) the effect of 25 % tire rubber of 60 mesh is almost completely eliminated. The effect of de-polymerization shown could have significant un-favorable effects on high temperature performance, in particular the possible loss of resistance to rutting.



mesh), reacted for 2 hr.

(After Tenzette and Kennenhol 06)

Figure 2 taken from the same study explains what is causing this reduction in softening point. As shown there is a similar trend of decreasing amount of un-reacted rubber, in other words, the increase in dissolved rubber due to de-polymerization causes the softening point reduction.



FIGURE • Amount of unreacted rubber in rubber/asphalt mixture (25 percent CRM 60 minus mesh) after 2 hr reaction (unsolubles after reaction at 280°C, 3 hr subtracted from each value).

(After Zanzotto and Kennephol. 96)

The study by Zanzotto and Kennepohl suggests that there is an opportunity to optimize Asphalt-Rubber binders. If none of the rubber is "cured" in the sense of swelling and partial depolymerization, then viscosity could be very high and thus workability is a challenge. In addition, insufficient curing (defined as a time/temperature reaction of the binder) of the rubber could cause phase separation. However, curing (prolonged high temperature reaction causing virtually the complete de-polymerization of the TR) too much could eliminate the benefits of rubber and causes loss of the properties as was shown in Figure 1. (Loss of the properties = reduction of the softening point).

Bullin et al study

The study by Bullin and co-workers followed a similar path to the first study in the sense of measuring amount of dissolved rubber as a function of time, temperature and heat. Figure 3 is an example of such results which shows that as time of curing is extended, more rubber is lost, particularly at higher shearing rate. At 375 F, which is relatively a low temperature for production of Asphalt-Rubber, 30 to 40 % of the rubber is lost in 2 days at low agitation of 500 rpm and in less than 4 hours if high agitation (1500 RPM) is used. One can speculate that at higher temperatures and high shear rates, typical of conditions used in production of Asphalt-Rubber today, a significant amount of rubber can be lost in a few hours.



Figure 3. Amount of tire rubber dissolved as function of curing time at 2 different shearing rates. (After Bullin et al, 1996)

Bullin and co-workers concluded that the extent to which tire rubber dissolves into the asphalt is very dependent upon the curing environment. Under the curing conditions studied, dissolving the rubber could improve the properties of a binder by improving stability and reducing viscosity during construction. However, more work should to be done to look at the effect on performance properties, since it is doubtful that complete destruction of the rubber molecules, and thus its elasticity, is desirable. That is, the optimization of binder properties is very dependent on the extent to which the rubber particle is de-vulcanized and/or de-polymerized.

Bullin's group also concluded that the chemical composition of the asphalt, the rubber mesh *size*, and the rubber content are important variables as they influence the interaction of asphalt with rubber and the de-polymerization of the latter.

Glover et al study

The work that was started by Bullin at Texas Transportation Institute was followed by a more comprehensive study by one of his Co-workers (Dr. Charles Glover). Glover's study looked at the changes in performance grading with curing. Figure 4 below taken from Glover et al reports shows the changes in the rutting parameter (G*/sin δ) with curing at 400 and 450 F, typical of terminal blending, where G* is the complex modulus and δ is the phase angle. It is clear that after 3.5 hours at 450 F the values of G*/sin δ reduced to almost the level of the base binder. These results confirm earlier studies and indicate that too much curing could eliminate the benefits of rubber modification.



Figure A G*/sin(delta) of Original Binders After First Curing at Different Curing Conditions.

(After Glover et al, 2000)

Glover's group also looked at low temperature properties. The low temperature changes (as measured by Bending Beam Rheometer, BBR, stiffness) are shown in Figure 5 taken from Glover's study. It can be seen that there are better values (lower stiffness) initially, but as curing temperature is increased, these benefits are partially lost.



(After Glover et al, 2000)

Glover's group also paid special attention to changes in elasticity. They measured the changes in elasticity using the phase angel since it is known that as elasticity improves a reduction in the value of the phase angle is observed. Figure 6 is taken from one the studies of this group and it shows a dramatic increase in phase angle, indicating loss of elasticity, as a function of curing time. The loss in elasticity (increase in phase angle) is seen for all three blends tested.



Figure 6. Loss of elasticity as measured by the increase in phase angle with curing time. (After Glover et al. 2000)

Glover concluded that shear mixing and higher temperature can break down the rubber particles and digests the long polymer chains and crosslink structures. He indicated that the viscosity of modified binder decreases with additional curing, but remains elevated above that of the base asphalt material. He speculated that although a particle phase continues to coexist with the binder at longer curing time, its size distribution rapidly declines as shortened polymer chains are integrated into the binder phase, which yields a lower viscosity at hot-mix installation. However, it certainly is possible to cure too far. Materials produced at the highest level of curing indicate severe polymer degradation. These binders produced with long curing times and/or higher shearing have wider PG span, but do not always yield improvement on a PG specification basis.

Summary of Main findings from the Literature and Concluding Remarks

Multiple studies have been conducted to evaluate the impact of digesting TR in asphalts. The studies lead to similar findings that could be summarized in the following points:

- 1. High shear mixing and high temperatures can break down the TR particles and result in a rubber-reinforced binder with reduced viscosities at the time of the hot mix asphalt installation. The mixing and heating can be controlled to optimize viscosity. Optimized viscosities have already been established for Asphalt-Rubber binders allowing for pumpability in a Hot Mix Asphalt (HMA) production facility. The PG requirement of 3.0 Pa-s is most widely recognized as a requirement for "pumping" in a production facility and it can be achieved easily using current conventional Asphalt-Rubber production methods.
- 2. The studies reviewed clearly show it is possible to cure rubber for too long a period at high temperatures. Materials produced at the highest level of curing indicate severe rubber degradation. This high level of curing can widen the PG span only incrementally and does not always yield improvements on specification basis required for a specific traffic condition.
- 3. The studies reviewed, and others cited in these studies, that have used the PG grading testing system for Asphalt-Rubber binders, seem to indicate no concerns regarding the results when the geometry and sample preparation is done carefully. The perception that Asphalt-Rubbers cannot be tested using the standard PG grading system is not confirmed in the studies reviewed.
- 4. The PG grading system includes testing with 4 different types of testing equipment, 5 geometries, at 4 temperatures. As shown in the schematic referenced as Attachment A, the equipment includes the rotational viscometer to be used at mixing and compaction temperature (135 C), DSR at high (~ 60 C) and intermediate (~ 20 C) temperatures, and the BBR and Direct Tension (DT) at low pavement temperatures (~-20 C). Asphalt-Rubber can, and has been, tested with all these types of equipment successfully. Table shown in Attachment A is from a study conducted as part of the Recycled Materials Resource Center (RMRC) and depicts the comparison of the PG high grade for rubber reacted for various times, with the effect of SBS and a high-digestion TR asphalt (PG-TR). As shown in the table the results confirm that partial digestion is the best approach to achieve high PG grade. The only exceptions that need modification for Asphalt-Rubber are testing at high pavement temperature using the parallel plate geometry with one millimeter gap and the aging in the RTFO when a high percent of rubber is used. For the parallel plate geometry at high

pavement temperature, a simple modification by increasing the gap to 2.0 mm, instead of the 1.0 mm used currently, can solve the problem, of size of rubber particles and allow grading of the Asphalt-Rubber with rubber particles as large as 0.5 mm. For the RTFO problems, which, is in fact a problem for not only Asphalt-Rubber, but also other highly elastic polymer modified binders, the TFOT aging procedure can be used. The TFOT is a test that is known to compare well to RTFO, is a standard test, and is still used in many parts of the world as an acceptable short term aging procedure. These 2 modifications could make it possible to PG grade most of the Asphalt-Rubber binders used today.

- 5. The studies reviewed have reported simple methods of separating un-dissolved rubber using common solvents and simple centrifuge. It is expected that one of these tests could be standardized and used as a requirement to ensure sufficient rubber has been added to make an Asphalt-Rubber binder.
- 6. The solubility requirement in the current Caltrans specification is not justified and studies reviewed do not include any indication that such a test is necessary to ensure performance. The current specifications could jeopardize the solid waste recycling program for used tires. It favors producing binders with reduced amount of TR, with highly digested TR, and/or more virgin polymers without a performance advantage. The experience and advancement in incorporating rubber in asphalt is very mature and cannot be used to justify this specification.

A	TTACE	IMENT	A – Ta	ble Tal	ken from	Study #54	of
Binder- React	Run 1	Run 2	Average	Std.	COV	PG Grade	Bump
Valero Neat	67.4	67.2	67.3	0.141	0.2%	64	0
10GTR_45min	85.1	83.9	84.5	0.849	1.0%	82	3
10GTR_65min	84.7	83.9	84.3	0.566	0.7%	82	3
10GTR_85min	83.4	84.0	83.7	0.424	0.5%	82	3
10GTR_360min	82.3	81.4	81.9	0.636	0.8%	76	2
PG -10TR	78.7	78.8	78.8	0.071	0.1%	76	2
Valero + 2%	72.9	73.0	72.9	0.071	0.1%	70	1
2% SBS + XL	75.4	75.4	75.4	0.000	0.0%	70	1

