Crumb Rubber
Asphalt Fatigue Study
Phase 1: Binder Testing

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Contract Report

Crumb Rubber Asphalt Fatigue Study
Phase 1: Binder Testing

EcoRecycle Victoria

AUSTROADS

Environment Protection Authority New South Wales

Project Officer
John Oliver

January 2001
CR IC6496

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Executive Summary

Introduction and Aims

The overall crumb rubber asphalt fatigue project can be split into two components:

1. characterisation of crumb rubbers and rubber bitumen binders and
2. measurement of the fatigue properties of crumb rubber asphalts.

This report presents results on the first stage of the work: the crumb rubber and rubber binder investigation.

Two types of crumb rubber binders were studied:

- commercially available preblends, and
- blends of crumb rubber and bitumen digested (manufactured) in the ARRB TR laboratory.

The main aims of the binder study were to obtain data on the properties of crumb rubber binders and to select appropriate binders for use in the asphalt study. The data developed would assist in the development of specifications for crumb rubber binders, and help rubber binder manufacturers to produce binders with specific properties.

Procedures

Crumb rubbers supplied by various manufacturers were sieved to determine their particle size distribution and their bulk density then measured. The bulk density of rubber particles had been shown in earlier work to be related to the elastic properties of digestions of the rubber particles in bitumen. On the basis of the results, one rubber (RE1) was selected to study the effect of a number of variables on the digestion process and another three rubbers (RG, RM and RL) to study the effect of rubber type.

In the digestion process, a sample of the selected rubber was stirred with bitumen at a controlled temperature and samples taken for testing at 0.5 h, 1 h, 2 h and 24 h. Digestion was carried out under a blanket of an inert gas to prevent oxidation of the binder.

Samples of the digestions prepared in the ARRB TR laboratory and from four preblended crumb rubber binders (PA, PB, PC1 and PC2) were tested using a dynamic shear rheometer and an Elastometer.

The Elastometer stretches a sample of material held at low temperature (where asphalt fatigue failure is most likely). From the force applied to stretch the material and the distance it stretches before rupture, a toughness parameter can be calculated.

The dynamic rheometer subjects a small sample of binder to sinusoidal oscillation and measures the resulting displacement. From this data, the modulus and phase angle of the binder is calculated for a range of rates of loading and test temperatures. A second test carried out on the instrument involves rotating the sample a fixed amount and then releasing it. The amount by which the sample returns to its original position is the per cent angular recovery and gives an indication of how elastic the material is.

Interpretation of Results

The modulus is an indication of the stiffness of the binder (high modulus means a stiff binder and one liable to crack easily during fatigue testing). The phase angle gives an indication of whether the material is behaving as an elastic material or as a viscous material or what the mixture of the two behaviours is. The modulus and phase angle basic data are plotted as a function of rate of loading (frequency). Such graphs can be difficult to interpret so, in the report, the plots are shown as a function of temperature at fixed frequencies.

A number of parameters derived from modulus and phase angle have been proposed as indicators of the fatigue behaviour of asphalt. There is international debate as to which are the most appropriate. One of these, G* sin delta (G* is the modulus, delta is the phase angle), has been used in the current study. While interpretation of the data is complex, in general good fatigue resistance is indicated by:

- a low value of G* sin delta
- a high value of angular recovery
- a high value of toughness.
- small change in modulus (and phase angle) with temperature (low temperature susceptibility).

Results

Time and Temperature of Digestion

The effect of time and temperature of digestion on elastic properties was determined using rubber RE1. The results indicated that, except for 24 h digestions, increased time and temperature of digestion should result in a more fatigue resistant binder. Digestion at high temperature for periods as long as 24 h may have caused degradation of the rubber.

Oxidation of Digestions

Oxygen was excluded during the digestion process by saturating the space above the binder with carbon dioxide, an inert gas. It is important, however, to know what effect oxidation has on crumb rubber binders. Such information is of value to crumb rubber binder manufacturers and also indicates the effect that high temperature storage has on crumb rubber binders.

To determine the effect of the presence of air on the digestion process, rubber RE1 was digested in 170 bitumen at 180°C with no carbon dioxide blanket, and the results compared with an equivalent digestion where carbon dioxide was used.

The results indicated that heating of crumb rubber digestions at high temperature without a carbon dioxide blanket caused oxidation of the binder. The effect was small for exposure periods of two hours or less but large for the 24 h exposure. As with pure bitumen, crumb rubber binder became harder and more elastic due to oxidative polymerisation reactions. This resulted in a binder with improved high temperature rutting resistance but much poorer low temperature fatigue resistance.

Effect of Rubber Type

Samples of four rubbers were each digested in a bitumen and the properties of the digestions measured.

The results indicated that the all the rubber modified binders tested should have improved fatigue resistance compared to the parent bitumen. Rubber RL recorded the best values of the performance indicators and rubber RG the worst.

Addition of Oil

Rubber RE1 WAS digested at 180°C in bitumen 170 for 0.5, 1, 2 and 24 h with and without the addition of extender oil.

Results from the different tests were contradictory. The G* sin delta parameter indicated much improved fatigue resistance due to oil addition whereas the toughness results suggested fatigue resistance would be much reduced.

Rubber Particle Size

A rubber crumb sample was sieved to produce two different sized fraction from the same rubber source. The two fractions were digested in a bitumen at 180°C for 0.5, 1, 2 and 24 h.

A somewhat confused picture emerged from the results but, particularly for longer digestion times, it appeared that smaller particle size may result in a more fatigue resistant material.

High Shear Mixing

A high shear mixer was used to determine whether shearing of the rubber particles during the digestion process produced a change in properties. A crumb rubber sample was digested normally in bitumen 170 at 180°C for 30 minutes then the blend was subjected to high shear stirring for periods of 5, 15 and 30 minutes.

All the performance indicators suggest that the high shearing mixing regime used had little or no effect on the important properties of the mix. Only one set of conditions (rotor speed, separation distance between rotor and fixed plate, etc.) was tried and it is possible that other conditions might produce a different result.

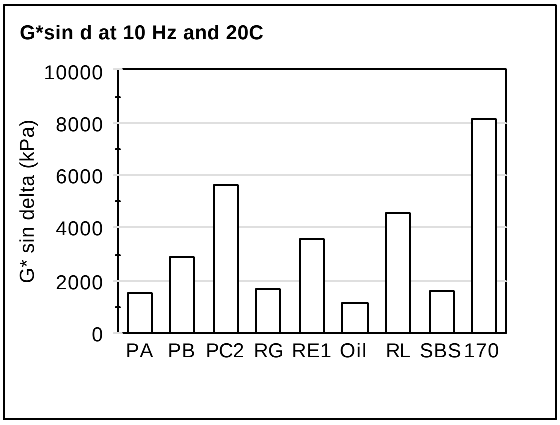
Crumb Rubber Preblends

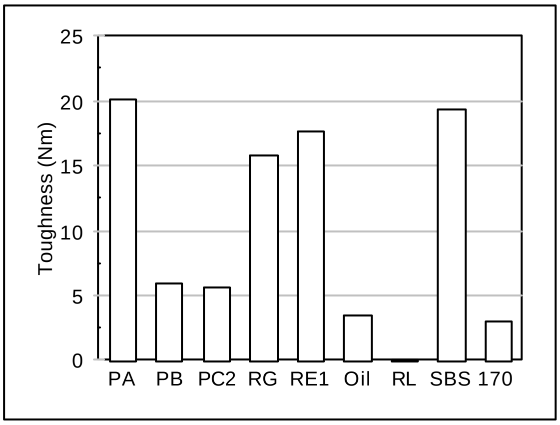
Samples of the four crumb rubber preblended binders provided by external suppliers were heated and cast into test specimens for dynamic shear rheometer and extensiometer testing.

The properties of binder PA were superior to the other three binders for each of the fatigue performance indicators: G*sin delta, angular recovery and toughness. The remaining binders showed inferior values of the indicator properties, with PC2 being the poorest performer.

Comparing the Performance of Different Binders

In the plots below, digestions of different crumb rubber in bitumen at 180°C for 2 hours are compared with commercially manufactured preblends, an SBS binder and a neat bitumen. The SBS binder was a premium grade polymer modified binder developed for asphalt use and provided excellent fatigue resistance in laboratory testing. Also included is a sample of RE1 which was digested with extender oil, labelled in the graph as “Oil”.





The key performance indicators suggest that binder PA should provide the best fatigue resistance (it had a low value of G* sin delta and a high value of toughness). Its tan delta value was low suggesting that fatigue resistance was not obtained at the expense of compromising rut resistance. The properties of PA appear to be similar to the SBS sample which was a premium grade polymer modified binder with excellent laboratory fatigue performance.

The 2 h digestion at 180°C of rubber RG in bitumen 170 also provided good fatigue resistance as indicated by G* sin delta, and the RE1 digestion gave good toughness results. It is possible that more severe digestion conditions might produce an even better results.

Selection of Binders for Use in the Asphalt Study

In the asphalt study, mixes will be manufactured with different crumb rubber binders and the fatigue resistance of compacted specimens measured using a laboratory fatigue test which has been under development in Australia and elsewhere for several years. Based on the results of the binder study, the following program is proposed.

1. The preblended rubber PA should be used to manufacture specimens for determination of the preferred composition (grading and binder content) of the crumb rubber asphalt mix.
2. A binder content of around 7% should be targeted. This needs to be a compromise between high fatigue performance (fatigue resistance is directly related to binder content) and cost, while still ensuring satisfactory high temperature rut resistance.
3. The ALF 320 bitumen should be used as the control.
4. Mixes with the selected grading and binder content should be manufactured with rubber binders of (expected) intermediate fatigue resistance. Suitable binders may include PB, RG and RE1.
5. One of the above binders should also be manufactured with an extender oil and included in the program.

Conclusions

1. All the fatigue performance indicators ranked binder PA as best of the digestions and preblended crumb rubber binders tested. The indications were that there would be no problems with the rut resistance of mixes incorporating this binder, making it the prime candidate for inclusion in asphalt fatigue trials.
2. Time and temperature of digestion are interdependent i.e. increase in digestion time or digestion temperature (up to a limit) can result in improved properties.
3. Digestion of crumb rubber in bitumen at 220°C for periods greater than 2 hours (possibly 4 hours) may provide optimum properties for most blends.
4. Exposure at high temperature can cause degradation of crumb rubber binder properties particularly where there is ready access of the binder to air.
5. A high bulk density rubber tested gave results indicative of poor fatigue and rut resistance.
6. There was an indication that smaller particle size rubbers gave more rut resistant digestions but the differences were not great nor the results conclusive.
7. High shear mixing, using one set of conditions, appeared to have no effect on the fatigue resistance of the digestion tested.
8. Addition of extender oil produced conflicting results, with one test indicating a marked improvement in fatigue resistance and another test a marked reduction in fatigue resistance.

1. Introduction

1.1 This Report

The work program to investigate the fatigue properties of crumb rubber asphalts can be split into two components:

1. characterisation of crumb rubbers and rubber bitumen binders, and
2. measurement of the fatigue properties of crumb rubber asphalts.

This report presents results on the first stage of the work: the crumb rubber and rubber binder investigation.

Two types of crumb rubber binders were studied:

- commercially available preblends, and
- blends of crumb rubber and bitumen digested (manufactured) in the ARRB TR laboratory.

1.2 Aims of the Binder Study

The aims of the binder study were to:

- obtain information on materials available,
- test binders and select those suitable for asphalt fatigue testing,
- correlate the binder results with asphalt fatigue performance (when this became available),
- obtain information to assist in the development of rubber binder specifications, and
- develop data to assist in the manufacture of crumb rubber binders with specified properties.

1.3 Layout of the Report

Testing of crumb rubber from various suppliers is described in Section 2 of the report and, based on the results, a number of rubbers are selected for the manufacture of crumb rubber/bitumen digestions. Also included in Section 2 are details of the preblended crumb rubber binders provided by commercial producers.

Section 3 describes the experimental procedures used to (i) manufacture the digestions and (ii) subsequently test the digestions and the commercial crumb rubber preblends. There is currently debate on how binder test results can best be related to asphalt fatigue resistance and in Section 4 there is discussion on the interpretation of binder results.

A summary of the test results is presented in graphical form in Section 5. Among the variables studied were time and temperature of digestion, crumb rubber type, addition of oil, rubber particle size and high shear mixing. Also presented are the results of testing the preblends and, for comparison, neat bitumen and a high performance synthetic polymer modified binder known to provide good fatigue resistance.

The final section of the report presents an overall comparison of the materials and identifies those that may usefully be incorporated into the asphalt fatigue study which forms the second phase of the project

2. Materials

2.1 Crumb Rubber

2.1.1 Suppliers

Australian suppliers of crumb rubber and preblended rubber binders were contacted and asked to provide samples of their material for evaluation. To preserve anonymity, the crumb rubber suppliers were allocated a code as shown in Table I. The letter "R" indicates that the material is rubber crumb, the second letter is the supplier code and a third digit indicates different materials supplied from the same source.

Since it was not possible to obtain cryogenically manufactured rubber crumb, which in earlier work had been shown to have different properties from ambiently ground rubber, material from an earlier project was used (sample RL). For comparison purposes, a sample of the same age but with much lower bulk density was also included (sample RM).

Table I

Crumb Rubber

Code	Description
RE1	Ambient ground tyre crumb rubber < 440µm
RE2	Ambient ground tyre crumb rubber < 630µm
RE3	Ambient ground tyre crumb rubber < 710µm
RF1	Ambient ground rubber < 16 mesh
RF2	Ambient ground rubber < 30 mesh
RG	Ambient tyre retread sandings supplied in water soaked state
RH1	Retreader's tyre buffings 30/16 mesh
RH2	Retreader's tyre buffings < 30 mesh
RH3	Retreader's tyre buffings < 16 mesh
RI1	Process unknown 30/16 mesh used in preblend PB
RI2	Process unknown < 30 mesh used in preblend PB
RJ	Source unknown used in preblend PA
RK1	Cracker milled rubber < 12mesh
RK2	Cracker milled rubber < 16mesh
RK3	Cracker milled rubber < 30 mesh
RK4	Cracker milled rubber 30/16mesh
RL	Cryogenically manufactured crumb rubber used in a previous project
RM	Synthetic tyre rubber sandings used in a previous project

2.1.2 Test Procedures

Representative samples of the crumb rubbers were sieved to obtain their particle size distribution, and single sized fractions were tested to determine bulk density (Oliver 1981). The bulk density test has been shown to be related to the morphology of rubber particles (Oliver 1982) in that smooth faced solid particles have low bulk density while porous particles with attached tendrils have high bulk density. High bulk density has been correlated with improved elastic properties in bitumen digestions of crumb rubber particles (Oliver 1982)

In the bulk density test, 7.5 g of sieved rubber particles were boiled in 100 mL of water to remove trapped air. A weak detergent solution was then added to ensure reproducible wetting of the particles. The volume of the rubber was measured after the sample had been allowed to settle in the water for 15 minutes and the bulk density calculated.

2.1.3 Results and Discussion

The particle size distribution of the rubber samples is shown in Table II. Not all samples were tested since some were judged to be too coarse or lenticular to be used in the manufacture of bitumen digestions. Sample RG contained entangled or fused particles which could not be separated for sieving.

Table II

Grading of Rubber Samples

Sieve Size (mm)	Percentage Passing															
	RI1	RI2	RJ	RE1	RE2	RE3	RF2	RH1	RH2	RH3	RK1	RK2	RK3	RK4	RL	RM
2.36	97.2	99.8	100	99.8	100	100	100	100	100	100	100	100	100	100	100	100
1.18	95.4	99.0	99.1	99.2	99.4	99.8	100	100	100	99.8	82.9	99.8	99.8	100	89.1	100
0.6	90.6	51.4	28.6	98.0	94.3	84.7	95.2	69.1	84.3	6.1	3.7	22.9	76.4	47.0	4.5	99.3
0.3	50.7	28.2	5.5	93.7	27.6	15.9	31.2	36.3	42.9	1.1	0.4	1.1	18.0	8.1	1.2	75.5
0.15	16.3	10.0	0.2	37.2	4.6	1.0	3.7	11.5	14.6	0.7	0.0	0.2	1.8	0.4	0.3	22.3
0.075	1.3	1.3	0.0	1.8	0.2	0.2	0.2	0.2	1.3	0.2	0.0	0.0	0.2	0.0	0	1.1

The bulk density results for selected samples are presented in Table III. Not all samples were tested since the procedure is not applicable to large sized particles. Examination of the Bulk Density results suggested that samples RE, RF, RH, RI, RJ, and RK were similar. It was decided, therefore, to manufacture digestions with only one material from this group, and RE1 was selected since it was the most commonly used crumb. Digestions were also made with samples which had the two lowest bulk densities (RG and RM) and with sample RL which had the highest bulk density.

Table III

Bulk Density

Material	Bulk Density kg/m^3
RE1	261
RE2	309
RE3	275
RF1	289
RG	143
RH2	274
RH3	273
RI1	259
RI2	252
RJ	270
RK3	244
RK4	248
RL	385
RM	132

2.2 Preblended Rubbers

Only two manufacturers of commercially available preblended crumb rubber binders could be located, and samples from these manufacturers are identified in Table IV as PA and PB. An experimental material prepared by two different processes was supplied (samples PC1 and PC2). Also tested and included in Table IV is the bitumen which was used to manufacture the laboratory digestions together with a synthetic polymer modified binder (PMB). The PMB contained a styrene butadiene styrene double ended block copolymer and had been tested in the Victorian Accelerated Loading Facility (ALF) Pilot Fatigue Trial. The performance of asphalt made with the SBS binder was judged to be good in laboratory fatigue testing, and binder test results for the material were included in this report for comparison purposes.

Table IV

Preblended Rubber Binders

Code	Description
PA	Commercial preblended crumb rubber binder
PB	Commercial preblended crumb rubber binder
PC1	Experimental preblended crumb rubber binder
PC2	Experimental preblended crumb rubber binder. Same supplier as above but extra treatment.
SBS	SBS binder used in ALF fatigue pilot trial
170	Class 170 bitumen used to prepare laboratory digestions. Sourced from Geelong refinery and manufactured using propane deasphalting process.

3. Experimental Procedures

3.1 Manufacture of Crumb Rubber Bitumen Digestions

3.1.1 Apparatus

Digestion of crumb rubber samples in bitumen was carried out in a stainless steel beaker (the reaction vessel) which fitted closely into a large aluminium jacket which acted as a heat sink. The aluminium jacket sat on top of a 400 watt, temperature controlled laboratory hotplate. In order to control digestion temperatures and to ensure a safe working environment, the aluminium jacket and hotplate were wrapped in several layers of fibreglass insulation and then encased in a 20 L steel drum. This arrangement permitted temperature control to be maintained to within ± 5ºC of the specified temperature.

A special lid on the reaction vessel allowed carbon dioxide gas to be pumped into the head space above the mixture. The inert gas reduced oxygen access and thus oxidation of the mixture. The carbon dioxide flow rate was maintained at 2 L/min from the commencement of the digestion process.

The crumb rubber digestion was continuously mixed by an overhead stirrer motor which drove a simple 8 prong stirring blade with a propeller at its base.

3.1.2 Digestion Procedure

15% crumb rubber by mass of the total mixture was added to bitumen 170. The rubber was slowly sprinkled onto the preheated and stirred bitumen over a period of 1 to 2 minutes. The addition of the rubber crumb cooled the heated bitumen so a 10 minute period was allowed for the mixture to return to the preset temperature.

Samples of approximately 10 to 12 mL volume were removed at intervals of ½ 1, 2 and 24 hours for subsequent testing. These samples were permitted to cool to ambient temperature before being stored in a refrigerator at approximately 5º C until tested.

3.2 Dynamic Shear Rheometer Testing

Binders were tested in a CarriMed dynamic stress rheometer using parallel plate geometry. Cylindrical specimens were prepared with a diameter of 20 mm and height of 2 mm and subjected to a series of sinusoidal oscillation frequency sweeps. The test specimen was surrounded by a temperature controlled water bath. The test sequence was commenced at 5º C and temperature was increased in 5ºC steps to a maximum of 60°C.

Following the sinusoidal oscillation testing, a creep test (angular recovery) was performed at 60° C. This latter test is the rotational analogue of a controlled stress Elastic Recovery measurement (current Elastometers operate in the controlled rate of strain mode). The samples were strained for 10 s and then allowed to recover. Plots of angular displacement against loading time were obtained. From the plots, the per cent angular recovery was calculated using eqn (1).

$$\text{Angular Recovery} = \frac{D_{10} - D_{100}}{D_{10}} \text{ x } 100\% \qquad (1)$$

where D_n is the angular displacement of the sample (rad) after n seconds.

The angular strain obtained before recovery depended on the stiffness of the binder. For each sample the stress which would result in 0.2 radians angular displacement at the circumference of the samples was calculated and applied (the displacement actually recorded varied slightly from the calculated 0.2 radians). An angular displacement of 0.2 radians is approximately equivalent to a strain of 1.0 applied in Elastic Recovery testing.

3.3 Extensiometer Testing

Binders were tested in the ARRB TR Extensiometer to evaluate the rheological behaviour at large strain levels. Specimens with a square cross-section of 9 x 9 mm and 25mm length were cast between end plates and subjected to an elongation of 250 mm (i.e.1,000% strain) at a rate of 0.7 mm/s, during which the force exerted on the specimen was continually measured. The test was carried out at 4°C, with the specimens fully immersed in a temperature controlled water bath.

A typical test result is shown in Fig.1.

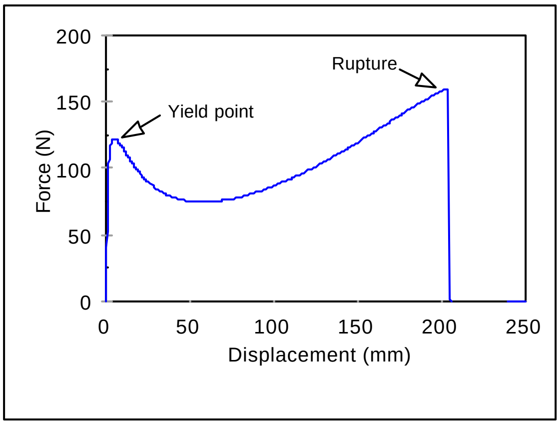


Figure 1. Typical extensiometer test result

A number of parameters were calculated from the test data, such as the first force peak and the displacement at which this occurs (yield point), the areas under the force/displacement curve to a number of displacements (yield point, 50, 100, 150, 200 and 250 mm), and the displacement at rupture if it occurs before 250 mm.

The area under the force/displacement curve to a specified displacement is called toughness, e.g. Toughness (250).

4. Interpretation of Results

4.1 Modulus and Phase Angle

The interpretation of data from dynamic shear rheometer measurements is currently the subject of international debate. Certain derived parameters have been proposed as indicators of fatigue and rutting resistance in asphalts but these have still to be validated. The situation is further complicated in that little or no performance correlation work has been carried out on crumb rubber binders and, since these contain particulate matter, they may perform differently to other polymer modified binders.

The primary data from oscillatory dynamic shear rheometer testing consists of a series of plots of shear modulus and phase angle against frequency at a range of temperatures. Normally a master curve is constructed from this data using a time temperature superposition procedure as described by Dickinson (1985). In this process the data sets for all test temperatures are combined to produce a single composite curve which is plotted in the form of phase angle against rate of loading at a single reference temperature (often 25°C). The relationships for other temperatures (within the range of the data) can be obtained by horizontally displacing the x axis (rate of loading) scale a calculated amount. An example of such a plot is shown in Fig. 2 and the boxed text explains the significance of certain features.

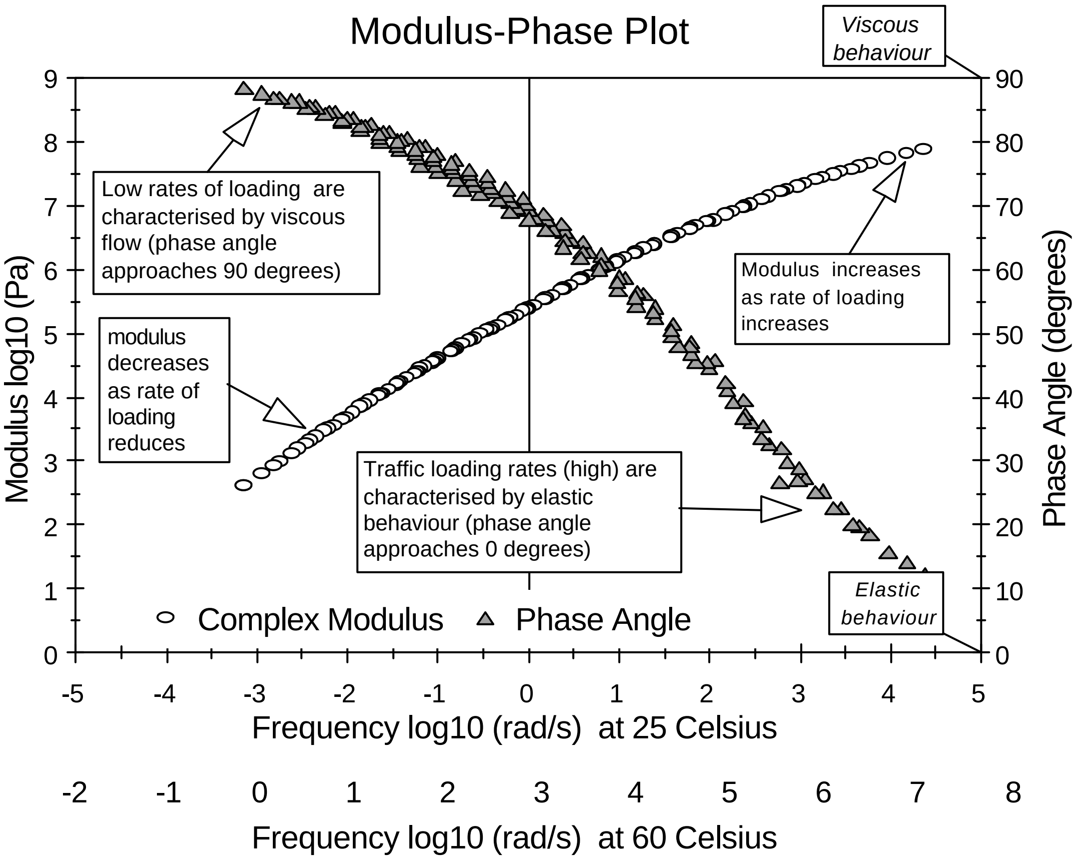


Figure 2. Example of modulus - phase plot for a Class 320 binder

Some workers find frequency plots difficult to work with so, in this report, a number of derived graphs are presented. The most important of these are plots of phase angle and modulus against temperature, and examples are shown Fig. 3.

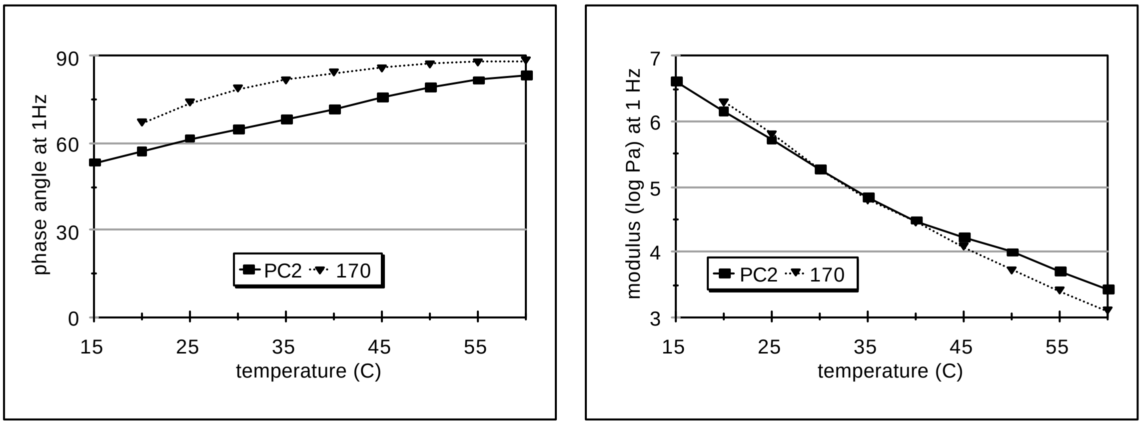


Figure 3. Example of modulus and phase angle vs temperature plots

As an indication of how such graphs can be interpreted, the following observations are made regarding Fig. 3. The phase angle plot indicates that the PC2 crumb rubber binder is more elastic than the base Class 170 bitumen at all temperatures (lower phase angles indicate more elastic and thus less viscous binder behaviour). The modulus plot shows that the crumb rubber modified binder has a slightly lower modulus than the base bitumen at low temperature suggesting that it may be more fatigue resistant. The modified binder also has a higher modulus at high temperature suggesting that it may be more rut resistant than the unmodified binder.

4.2 Angular Recovery

A high value of angular recovery indicates that a material is elastic at the test temperature (60°C) and able to almost fully recover when deformed. The test is carried out at higher strain levels than the dynamic shear testing and thus indicates higher strain binder behaviour.

A mechanism by which a binder might resist permanent deformation and cracking is to deform under loading but to fully recover when the load is removed.

4.3 Derived Parameters

G sin delta*

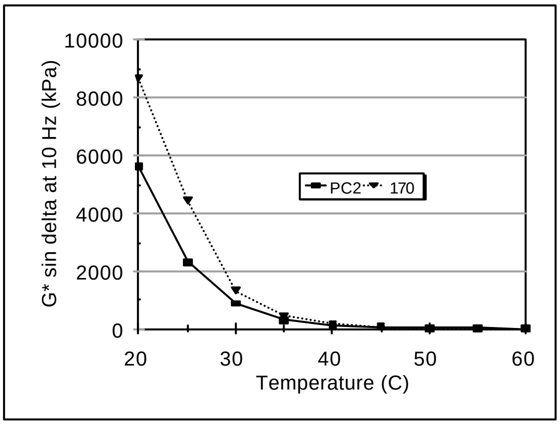


Figure 4. Example of G sin delta vs temperature plots*

Application of the dissipated energy approach to fatigue has identified the parameter G* sin delta as a likely indicator of fatigue resistance (modulus is referred to as G* and the phase angle as delta). This parameter has been adopted for use in the U.S. Superpave binder specification. Since laboratory fatigue testing of asphalt is commonly performed at 20°C and a loading rate of 10 Hz, G* sin delta was calculated under these conditions. An example of the plot produced is shown in Fig. 4. Lower values of the parameter would be expected to be associated with superior fatigue resistance. Thus PC2 would be expected to be slightly more fatigue resistant at lower temperatures than 170. The difference between the binders is small at high temperatures where fatigue failure is unlikely to occur.

Tan delta

Tan delta was found to correlate with asphalt deformation resistance for binders examined by Oliver et al. (1995). It is therefore considered a useful parameter to measure. Since the most severe rutting conditions on the road occur with slow moving vehicles, measurement of the parameter at a slow rate of loading is desirable. The slowest loading rate obtainable with the CarriMed was 0.1 Hz and this value was therefore used.

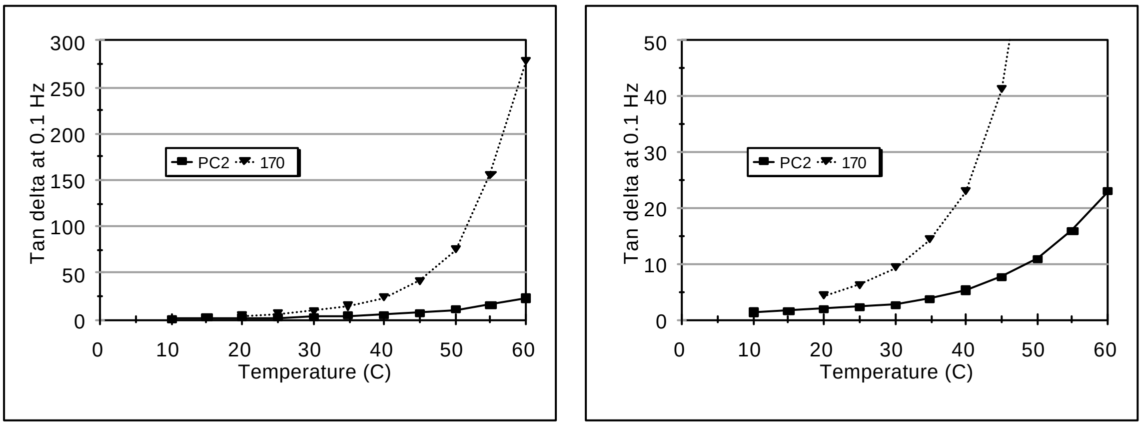


Figure 5 . Example of tan delta vs temperature plots

An example of plots of tan delta against temperature is shown in Fig. 5. The plots suggest that the PC2 binder will be more rut resistant than the unmodifed Class 170 bitumen (the lower tan delta, the greater the rut resistance). While it might appear that the difference between the two binders greatly increases at high temperature this is probably not the case and results from the tangent of the phase angle increasing rapidly as the phase angle approaches 90° (the phase angle for 170 was 89.25°, 89.64° and 89.80° at 50°C, 55°C and 60°C respectively). Because of the effect that small errors in tan (phase angle) have at phase angles close to 90°, it is advisable to restrict examination of such data to tan delta values below 50, as shown in the right hand plot.

4.4 Extensiometer Results

The extensiometer has only recently been developed and work still has to be done on the interpretation of results. However, it might be expected that the area under the force displacement curve (see Fig. 1), commonly referred to as "toughness (250)" will act as an indicator of the fatigue resistance of a binder. This area is the energy absorbed by the material to rupture. The higher the energy required for rupture, the greater is the likelihood that the material will be more resistant to rupture and hence more resistant to fatigue failure.

As the properties of an asphalt mix are greatly influenced by the binder properties, particularly for a low temperature failure mode such as fatigue, binders with high toughness can be expected to provide good fatigue performance asphalts.

4.5 Summary

Modulus (G*) and phase angle (delta) provide basic data on binder properties. Generally fatigue performance is improved by low values of G* at low temperatures (the binder is softer and less brittle). However, if G* was reduced at all temperatures, for example by using a softer base bitumen, then rutting would occur at high temperature. The aim, therefore, is to modify the base bitumen with rubber so that the change in modulus with temperature (the temperature susceptibility) is reduced.

Certain parameters, which can be calculated from the modulus and phase angle data, have been proposed as indicators of service (road) performance. G* sin delta (under low temperature and fast loading rate conditions) is expected to correlate with fatigue resistance while tan delta (under high temperature and slow loading rate conditions) may indicate rutting resistance.

Angular recovery is analogous to Elastic Recovery and this parameter is currently used to evaluate Australian polymer modified binders. A high value of Elastic Recovery indicates that the binder is elastic and able to almost fully recover after being loaded.

A number of parameters can be calculated from Extensiometer data. Toughness (250) is considered to be the one most likely to correlate with fatigue resistance.

In summary, good fatigue resistance may be indicated by

- a low value of G* sin delta
- a high value of angular recovery
- a high value of toughness.

Both rutting and fatigue performance may be improved by

- small change in modulus (and phase angle) with temperature (low temperature susceptibility).

Good rutting resistance may be indicated by

- a low value of tan delta
- a high value of angular recovery.

5. Results

5.1 Effect of Time and Temperature of Digestion

5.1.1 Introduction

The time and temperature of digestion of crumb rubber with bitumen are known to have an important effect on the resulting crumb rubber binder (Oliver 1982). The major effect is probably absorption by the rubber particles of aromatic oils in the bitumen. This causes softening and swelling of the rubber particles so that a comparatively large proportion of the binder consists of soft rubber. At the same time, the bitumen phase hardens because of loss of oils. These are all physical changes.

Chemical reactions also occur, with the rubber depolymerising at high temperatures. In the case of natural rubber (new truck tyre treads) this results in fairly rapid loss of elastic properties and a reduction in the “viscosity” of the binder (Oliver 1982). Compared to natural rubber, synthetic rubber (as found in car tyre treads), is more resistant to the effect of temperature and must be raised to a higher temperature or held at high temperature for longer periods before depolymerisation has an important effect on binder properties.

In order to study the magnitude of the effects, a commonly used crumb rubber (RE1), typical of Australian production, was digested in a Class 170 bitumen over a range of digestion times and temperatures. A rubber concentration of 15% by mass of the total binder was used in all cases.

5.1.2 Test Results

Modulus

The test program generated a large volume of data. In order to assist in the observation of important trends, the amount of data presented has been restricted. In the case of modulus, phase angle and derived parameters, only values at 20°C and 60°C and 1 Hz frequency are shown in the following graphs. Results at 20°C are related to the fatigue properties of a binder while results at 60°C should indicate its resistance to permanent deformation (rutting).

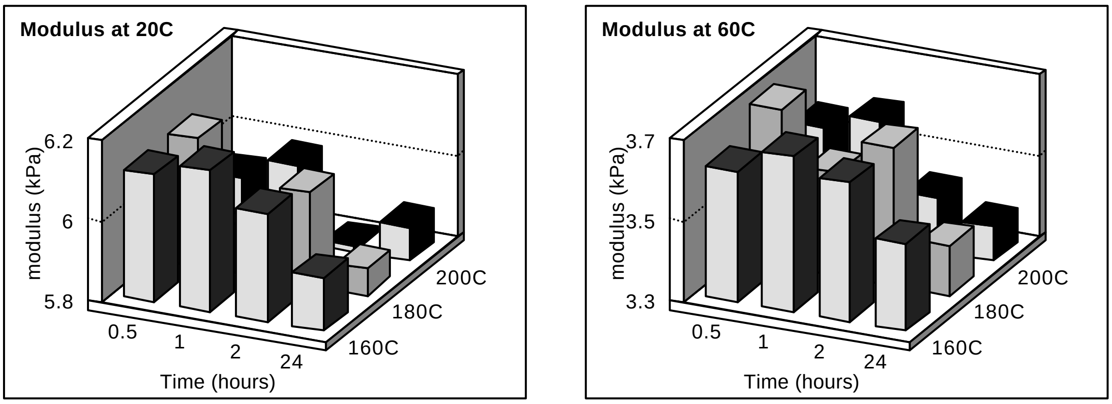


Figure 6. Effect of digestion time and temperature on modulus (RE1 digested in 170)

It should be noted that the scale of the y-axis (modulus) in Fig. 6 has been expanded to permit trends to be more easily observed. Small differences between results should, therefore, be treated with caution since these may be of the same order as unavoidable experimental error.

Increase in time and temperature of digestion generally resulted in a reduction in modulus. This became more pronounced at the extremes (24 h digestions at all temperatures and 2 h digestions at 200°C).

Phase Angle (Delta)

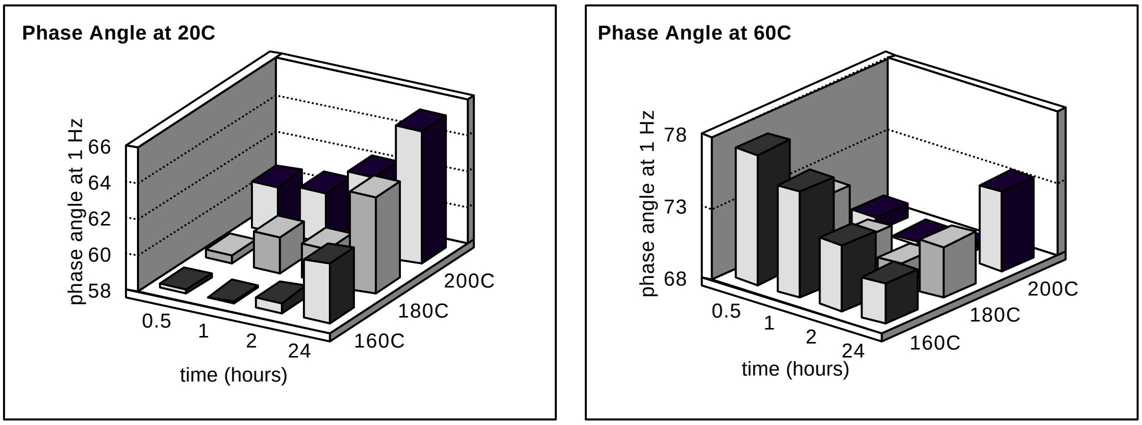


Figure 7. Effect of digestion time and temperature on phase angle (RE1 digested in 170)

The results for phase angle at 20°C appear to be the reverse of those at 60°C. At 20°C, increase in digestion time or temperature caused an increase in phase angle and thus resulted in a less elastic material. At 60°C, however, for digestions of less than 24 h, phase angle decreased with time or temperature of digestion resulting in a more elastic material.

This can be expressed more simply by saying that the temperature susceptibility of the binder was generally improved by increased time and temperature of digestion - that is to say the change in phase angle with measurement temperature was reduced as digestion conditions became more severe.

Derived Parameters

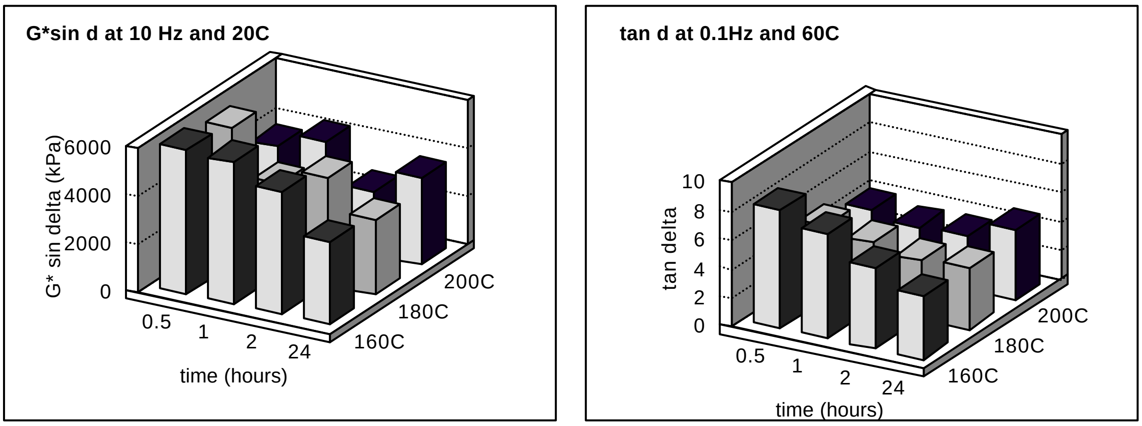


Figure 8. Effect of digestion time and temperature on G sin delta and tan delta (RE1 digested in 170)*

As indicated in Section 4.3, low values of G* sin delta may indicate improved fatigue properties. A reduction in G* sin delta was obtained by increased time or temperature of digestion. The same reasoning applies to tan delta which has been correlated with rutting resistance. Increased time and temperature of digestion should produce a binder with improved rut resistance.

Angular Recovery

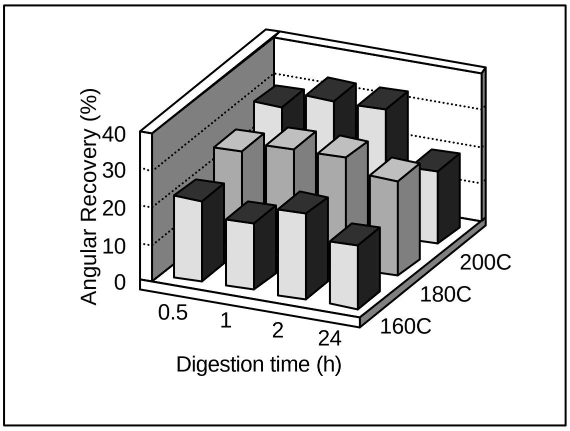


Figure 9. Effect of digestion time and temperature on angular recovery (RE1 digested in 170)

Similar trends to the above were obtained in the case of angular recovery. Increasing the time and temperature of digestion resulted in a more elastic material except in the case of the 24 h digestion data. It is likely that degradation of the rubber occurred in this case.

Extensiometer Testing

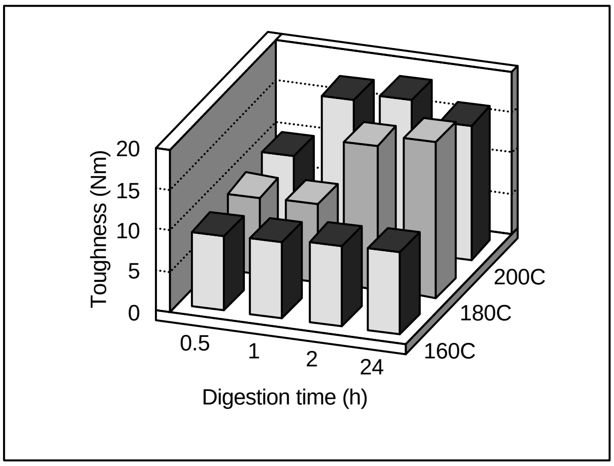


Figure 10. Effect of digestion time and temperature on toughness (RE1 digested in 170)

Toughness (250) is probably the best of the currently calculated extensiometer parameters for indicating improved fatigue resistance, and this has been plotted in Fig. 10. With the exception of the 24 h digestion results, increase in time and temperature of digestion gave improved toughness which would be expected to result in improved fatigue resistance.

5.1.3 Summary

All the parameters measured were generally in agreement in indicating that, except for 24 h digestions, increased time and temperature of digestion should result in a more fatigue resistant binder. Digestion at high temperature for periods as long as 24 h may have caused degradation of the rubber.

5.2 Oxidation of Digestions

5.2.1 Procedure

As indicated in Section 3.1, the crumb rubber/bitumen digestion procedure used in the ARRB TR study includes a measure to reduce oxidation of the binder during the digestion process. Oxygen is excluded by saturating the space above the binder with carbon dioxide, an inert gas. It is important, however, to know what effect oxidation has on crumb rubber binders. Such information is of value to crumb rubber binder manufacturers and also indicates the effect that high temperature storage has on crumb rubber binders.

To determine the effect of the presence of air on the digestion process, rubber RE1 was digested in bitumen 170 at 180°C with no carbon dioxide blanket, and the results compared with an equivalent digestion where carbon dioxide was used.

5.2.2 Results

Modulus and Phase Angle

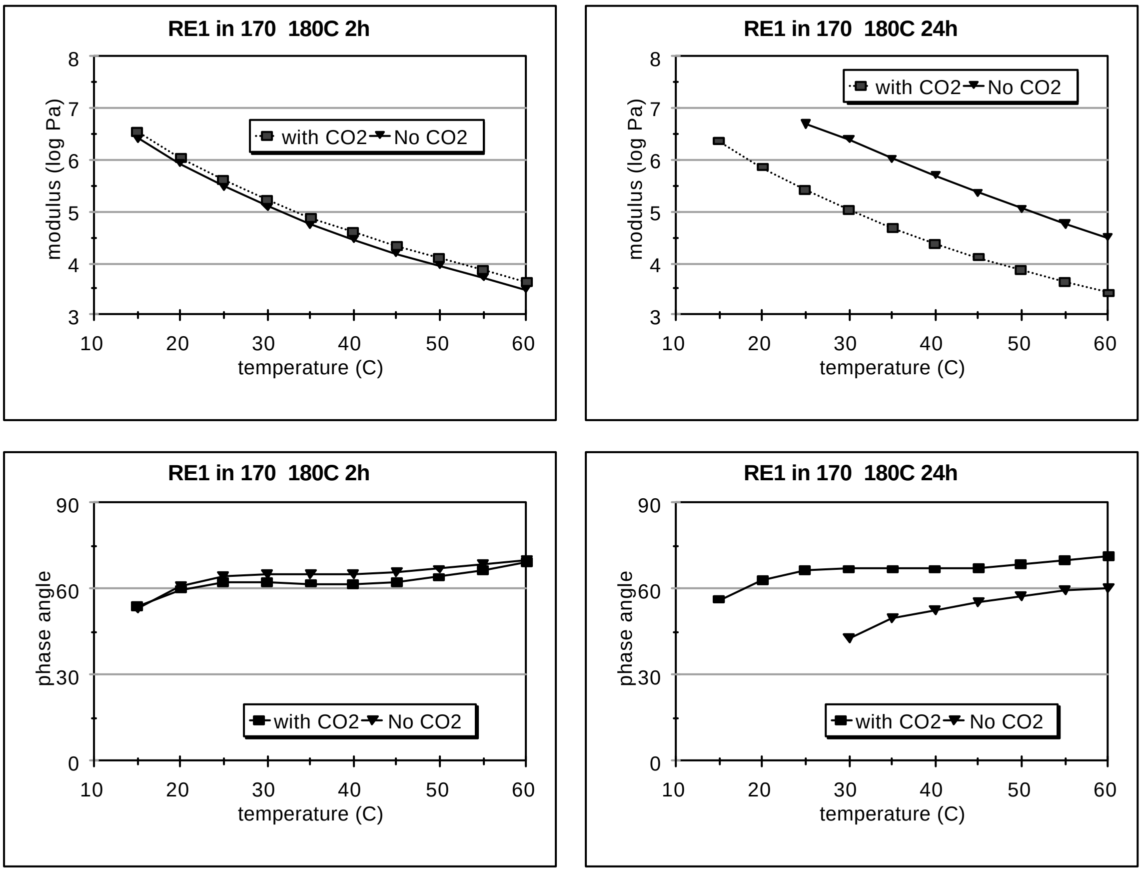


Figure 11. Effect of carbon dioxide on modulus and phase angle for 2 and 24 h digestions

The modulus and phase angle results indicate that exposure to air for up to 2 h at 180°C had only a small effect on properties: the modulus increasing slightly and the phase angle generally decreasing slightly. The 24 h results, however, showed very large changes in properties. The modulus very substantially increased and there was a corresponding decrease in phase angle. Such behaviour could be explained by oxidation of the bitumen which would increase the modulus of the material and make it correspondingly more elastic. It is possible that some rubber cross linking also occurred.

Derived Parameters

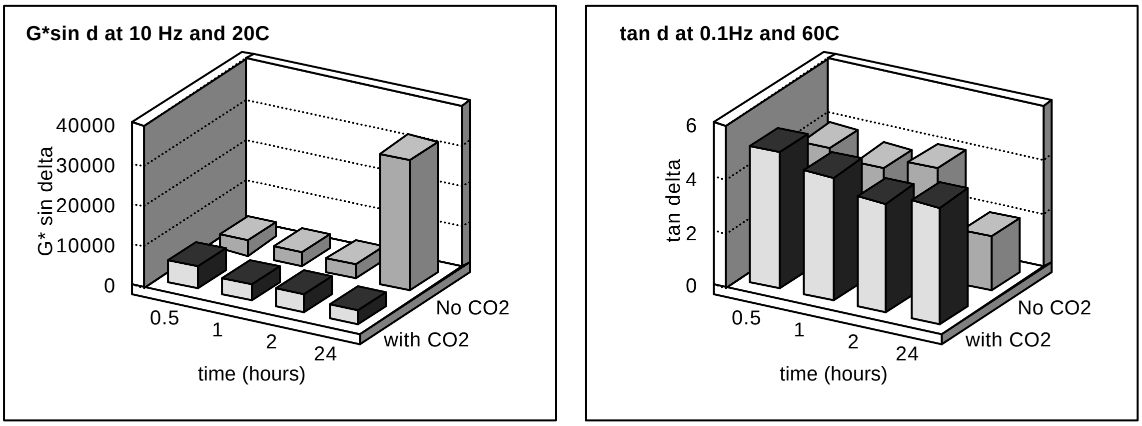


Figure 12. Effect of carbon dioxide during digestion on G sin delta and tan delta*

As might be expected from the modulus and phase angle results, the 24 h result for G^* sin delta shows a very large increase indicating a much less fatigue resistant material. On the other hand, the tan delta value decreased, suggesting a more rut resistant material at high temperature (60°C).

Angular Recovery and Toughness

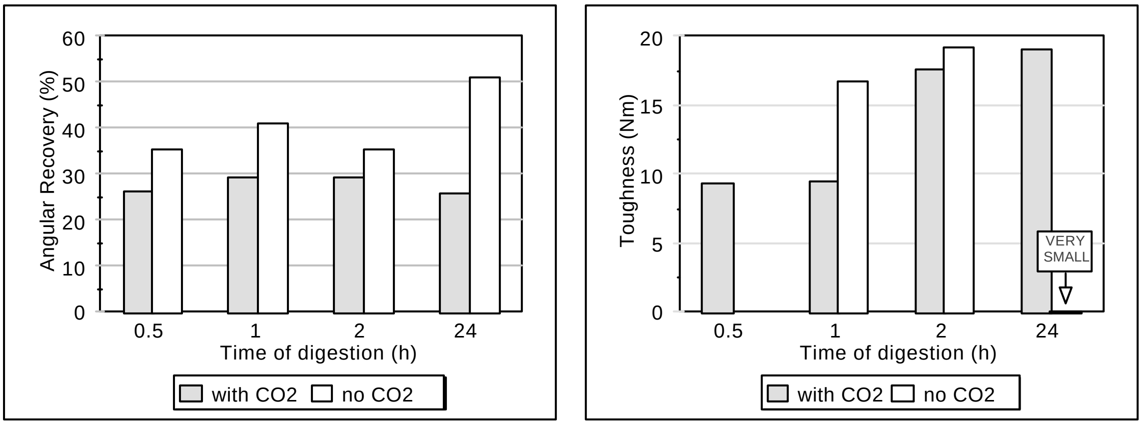


Figure 13. Effect of carbon dioxide angular recovery and toughness
(PC2 digested in 170 at 180°C)

Angular recovery increased with time of digestion, possibly as a result of oxidative polymerisation of bitumen components and some rubber cross linking. However prolonged oxidation resulted in very low toughness, with the sample failing at very low displacement.

5.2.3 Summary

Heating of crumb rubber digestions at high temperature without a carbon dioxide blanket caused oxidation of the binder. The effect was small for exposure periods of two hours or less but large for the 24 h exposure. As with pure bitumen, crumb rubber binder became harder and more elastic due to oxidative polymerisation reactions. This resulted in a binder with expected improved high temperature rutting resistance but much poorer low temperature fatigue resistance.

5.3 Effect of Rubber Type

5.3.1 Procedure

As indicated in Section 2.1.3, four crumb rubbers were selected to cover the range of low to high bulk density. These rubbers were RE1, RG, RM and RL. Samples of each rubber were digested, at a concentration of 15% by mass of total binder, in bitumen 170 at a temperature of 180°C for periods of 0.5, 1, 2 and 24 h.

5.3.2 Results

Modulus and Phase Angle

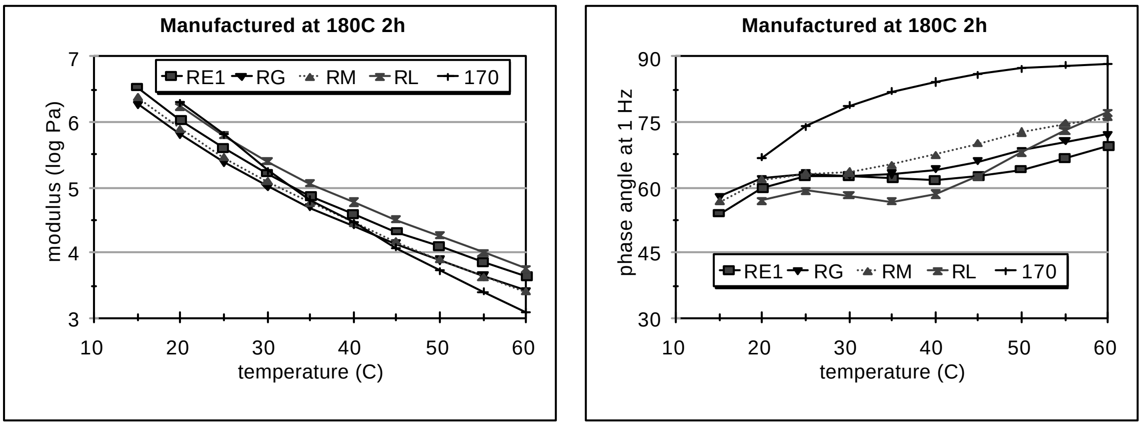


Figure 14 Digestions of four rubber crumbs in bitumen 170 for 2 h at 180°C

Comparison of the modulus results for the rubber digestions and the base bitumen (170) shows that the modulus temperature susceptibility of all the rubber digestions was superior to neat bitumen (the modulus of the digestions changed less with temperature than bitumen). It is desirable in the road situation to have a binder which undergoes as small a change as possible within the service temperature range (0°C to 60°C).

Examination of the phase angle plot indicates that rubber RE1 experienced the greatest change in phase angle with temperature, and rubber RL the least. All the rubber digestions were more elastic than the base bitumen from which they were manufactured.

Derived Parameters

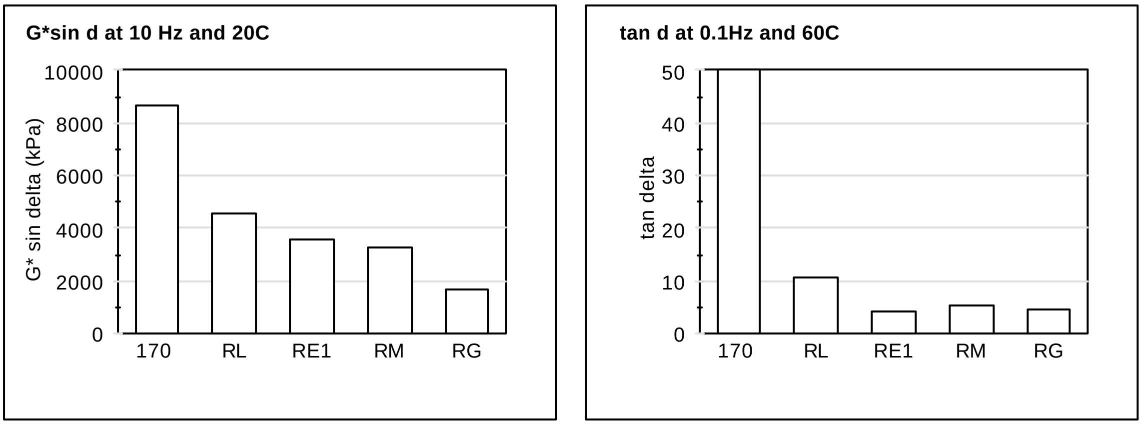


Figure 15 G sin delta and tan delta for 2h digestions of rubber in bitumen 170 at 180°C*

The results indicate that the all the rubber modified binders should have improved fatigue and rutting resistance compared to the parent bitumen. Rubber RL had the highest (least desirable) values of G* sin delta and tan delta of the four rubbers. RG had the lowest value of G* sin delta indicating that it may have the best fatigue resistance of the four.

Angular Recovery and Toughness

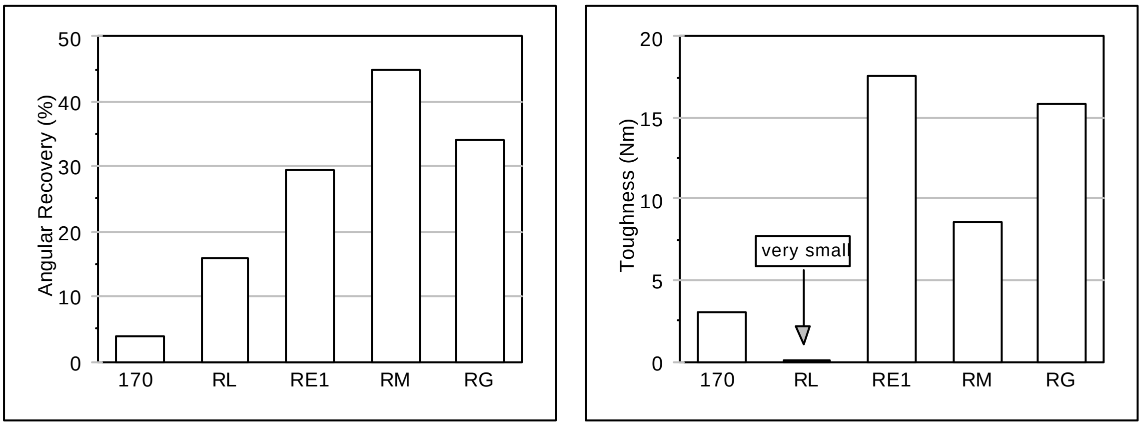


Figure 16 Angular recovery and toughness for 2h digestions of rubber in bitumen 170 at 180°C

RL again had the poorest properties of the four crumb rubber binders. While RM gave the highest angular recovery, RE1 had the highest toughness result.

5.3.3 Summary

The temperature susceptibility of bitumen was improved through digestion with rubber. Of the rubber crumbs tested, RG had the lowest G* sin delta (a low value is expected to give good fatigue resistance) and quite high values of angular recovery and toughness (again expected to indicate give good fatigue resistance). L1 had the poorest values in all cases.

5.4 Addition of Oil

5.4.1 Procedure

Rubber RE1 was digested at 180°C in bitumen 170 for 0.5, 1, 2 and 24 h with and without the addition of extender oil. The extender oil consisted of two components and was added before digestion of the rubber commenced. The component oils were Mobilsol 40, which was added at a concentration of 5% by mass of binder, and a recovered sump oil (from diesel fuelled vehicles) which was added at a concentration of 2.3% by mass of the binder.

5.4.2 Results

Modulus and Phase Angle

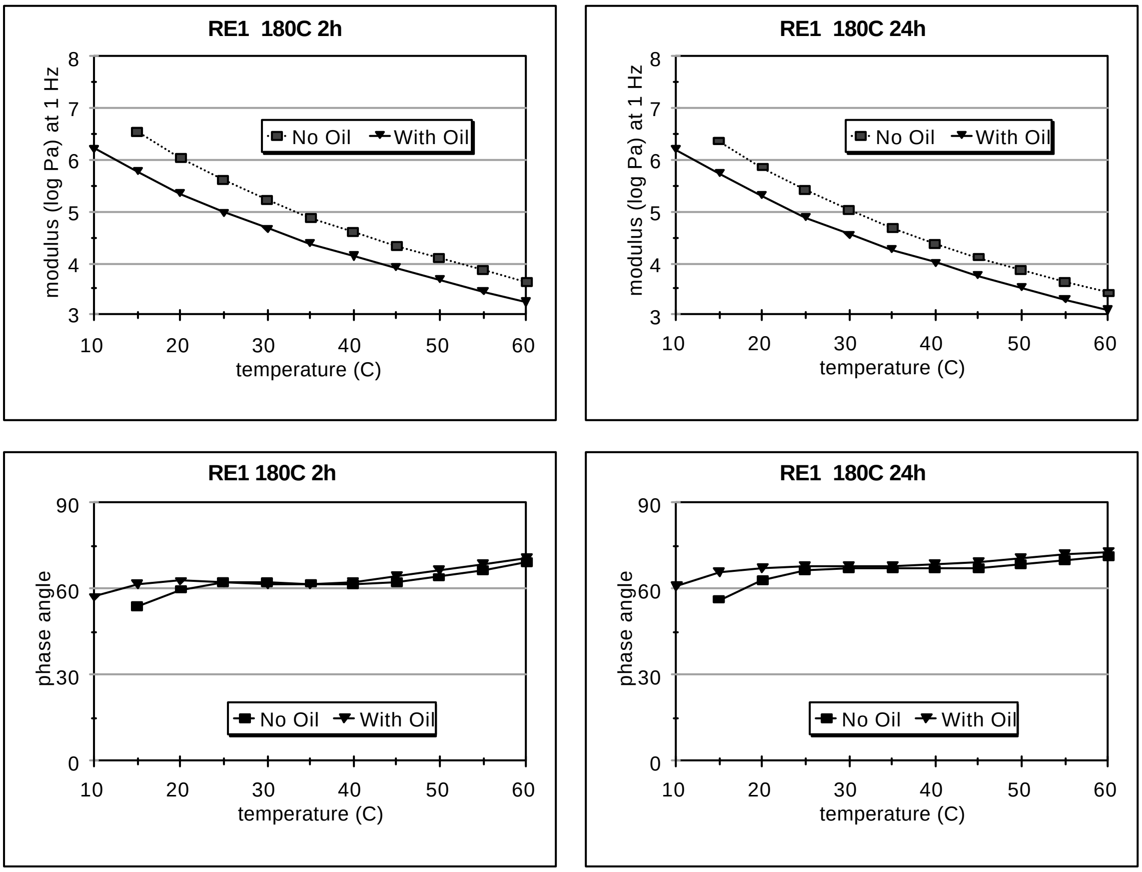


Figure 17. Effect of oil on modulus and phase angle for 2 and 24 h digestions

The effect of oil was to reduce the modulus of the binder across the entire temperature range. Phase angle appears to have been to have been affected only at low temperatures, where the binder with oil was less elastic than the binder without oil.

Derived Parameters

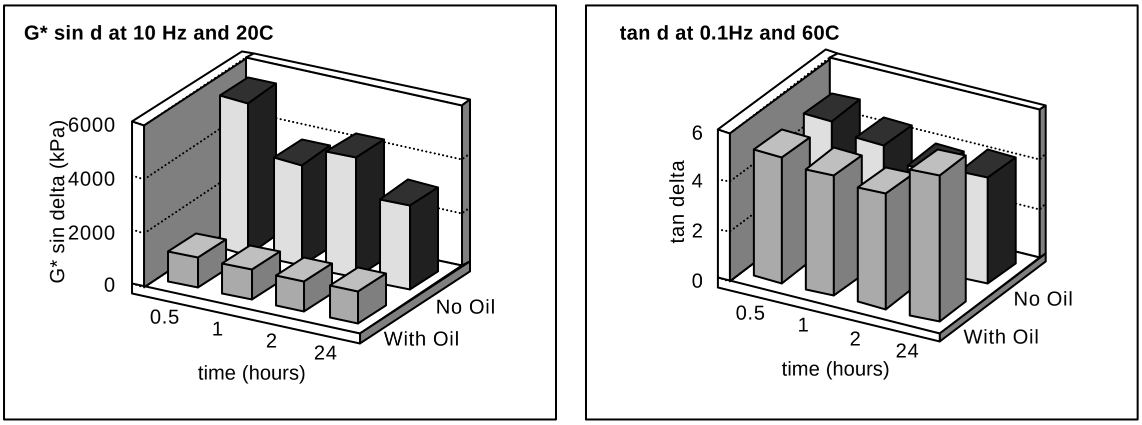


Figure 18. G sin delta and tan delta for digestions of rubber RE1 in bitumen 170 with and without oil at 180°C*

The G* sin delta plot indicates that the fatigue properties should be improved by the addition of oil. Surprisingly, the tan delta results suggested that rut resistance would not be much reduced by oil addition.

Angular Recovery and Toughness

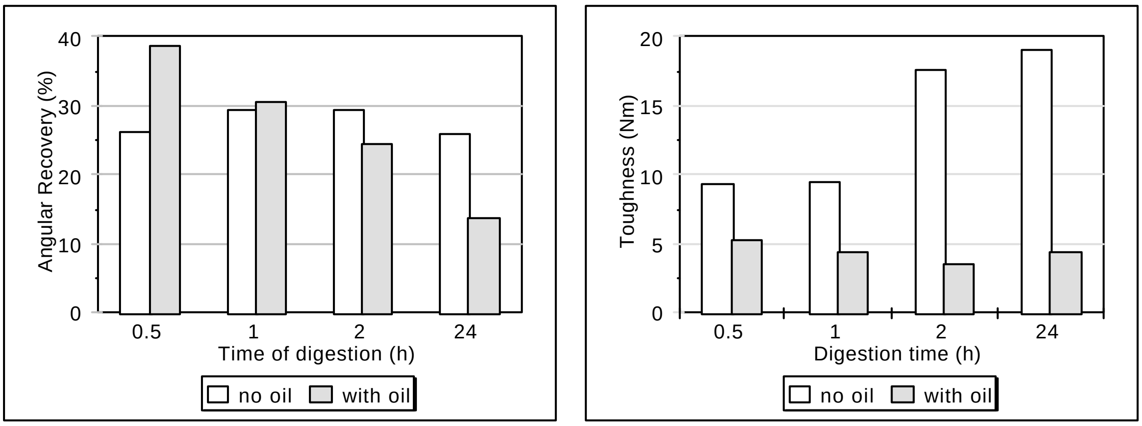


Figure 19 The effect of oil on angular recovery and toughness for 180°C digestions

After the first hour of digestion, angular recovery was reduced by the addition of oil. Toughness was much lower for all digestion periods indicating greatly reduced fatigue resistance, in contrast to the G* sin delta results which suggested the opposite.

5.4.3 Summary

The addition of oil may not only soften the bitumen phase in a crumb rubber binder but may swell and soften the rubber particles. The test results present a confusing picture in that G* sin delta was greatly reduced by the addition of oil, suggesting that fatigue resistance will be much improved. Toughness, on the other hand was much reduced by oil addition suggesting that fatigue resistance will be poorer. It may be useful to resolve the effect of oil addition by fatigue testing asphalt specimens.

5.5 Rubber Particle Size

5.5.1 Procedure

Rubber RE3 was sieved to produce a 600 to 300 µm fraction and a 300 to 150 µm fraction. A single source of material was used, rather than two materials of different sizes, in order to ensure that the two fractions differed only in size and not in rubber composition. The two fractions were digested in bitumen 170 at 180°C for 0.5, 1, 2 and 24 h.

5.5.2 Results

Modulus and Phase Angle

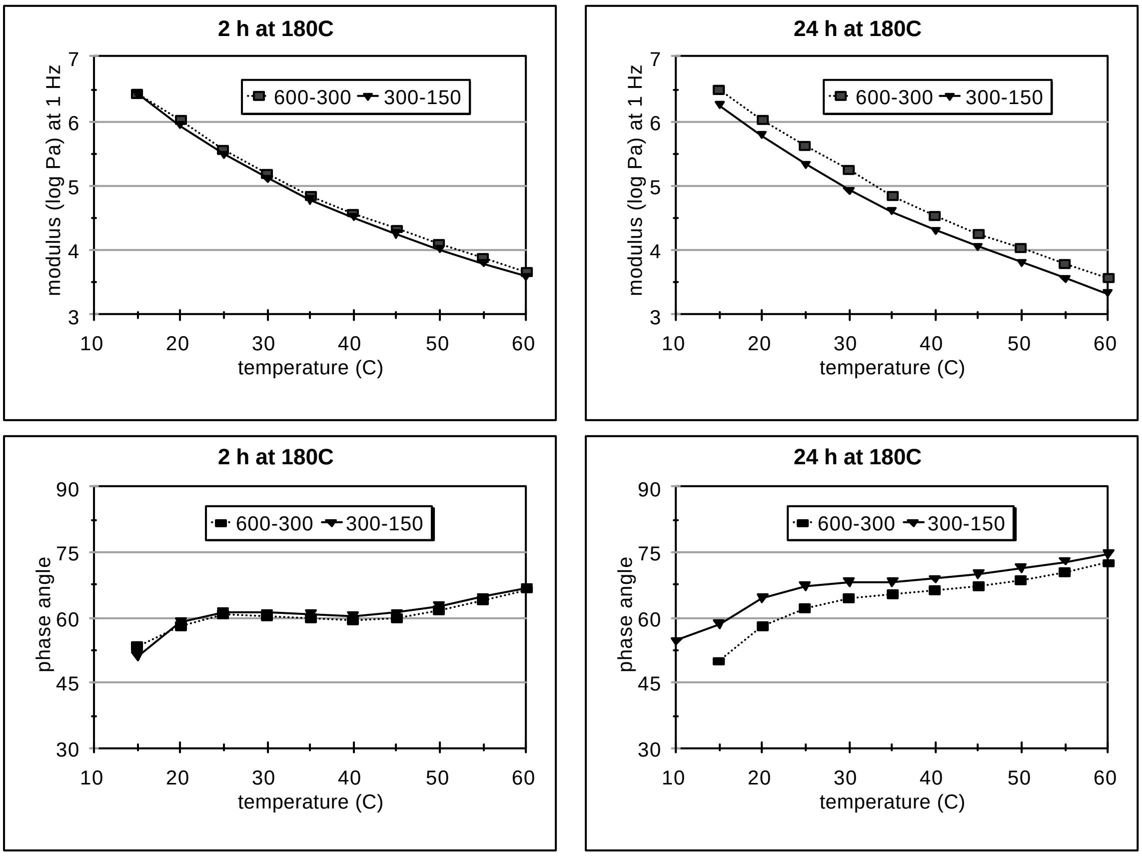


Figure 20 The effect of particle size on modulus and phase angle

The results indicate that there was little difference between the two sizes after two hours digestion. After 24 h, however, the finer material (300-150 µm) produced a softer binder which was less elastic than the 600-300 µm fraction.

Derived Parameters

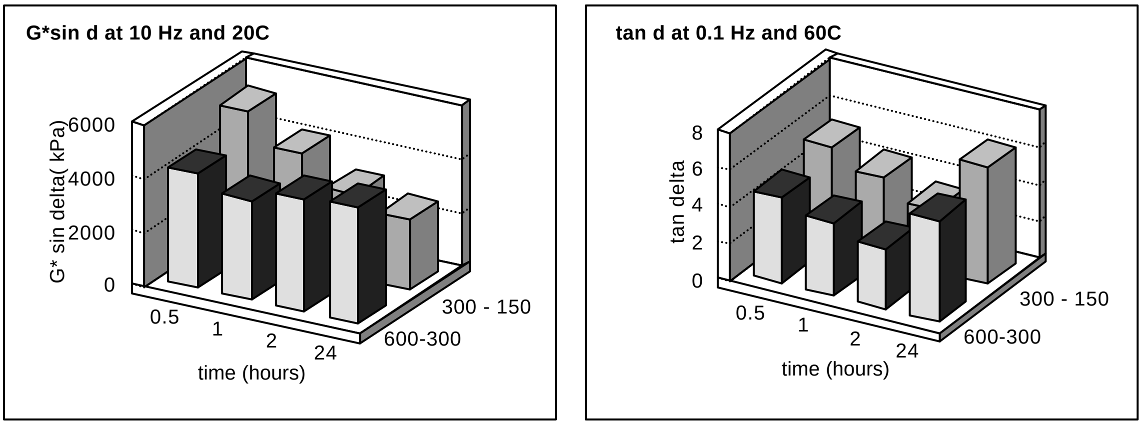


Figure 21 The effect of particle size on G sin delta and tan delta for digestions at 180°C*

Fig. 21 shows a rather confusing picture for the fatigue indicator. For digestion times of 1 h or less, G* sin delta was lower for the 600-300 µm fraction than for the 300-150 µm fraction. This trend was reversed for longer digestion periods.

In the case of tan delta, the 600-300 µm results are lower than the 300-150 µm results for all digestion periods

Angular Recovery and Toughness

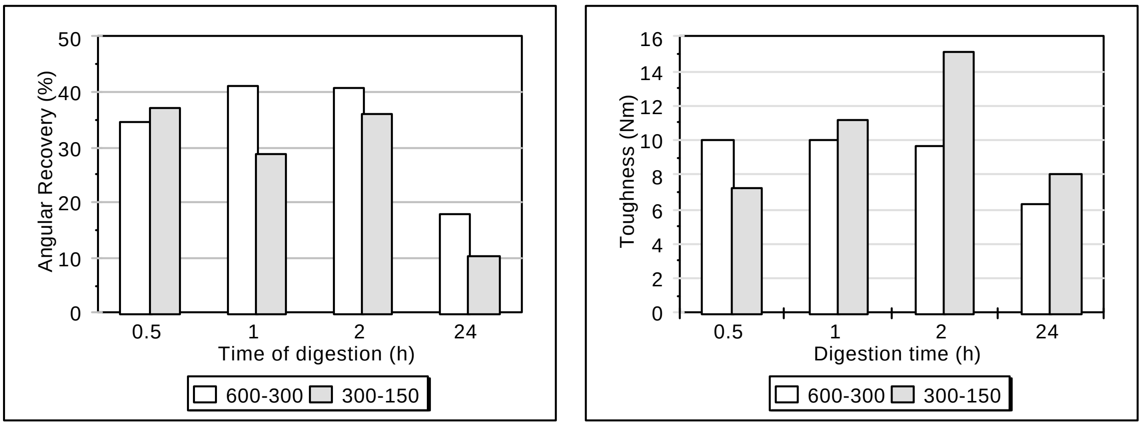


Figure 22 The effect of particle size on angular recovery and toughness for digestions at 180°C

From one hour onwards the finer material had lower elastic recovery than the coarse. This result is not in agreement with earlier findings (Oliver 1982). In the case of toughness, however, the finer material gave a superior result, except for the half hour digestion.

5.5.3 Summary

A somewhat confused picture emerged from the results as to the effect of rubber particle size on fatigue performance but, particularly for longer digestion times, it appears that smaller particle size may result in a more fatigue resistant material.

5.6 High Shear Mixing

5.6.1 Procedure

A Silverson high shear mixer was used to determine whether shearing of the rubber particles during the digestion process produced a change in properties. Rubber RE1 was digested normally in bitumen 170 at 180°C for 30 minutes then the blend was subjected to high shear stirring for periods of 5, 15 and 30 minutes.

5.6.2 Results

Modulus and Phase Angle

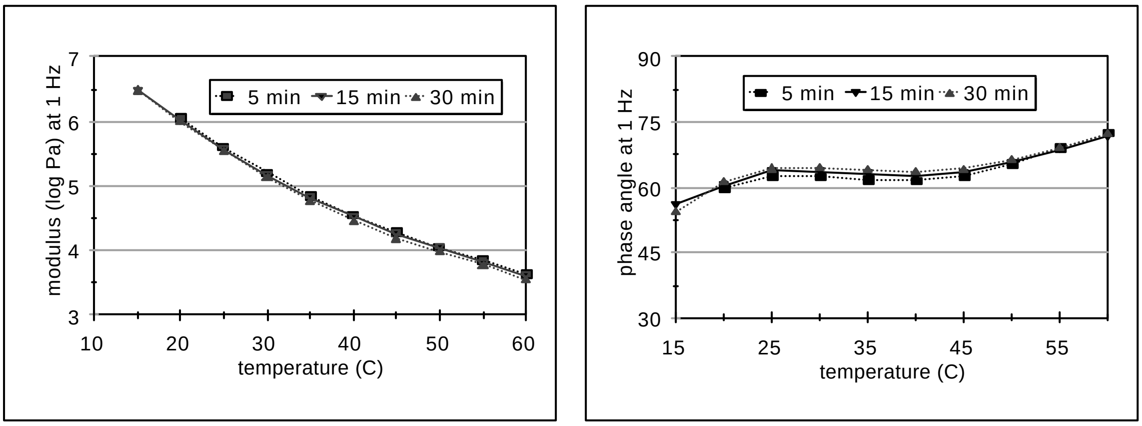


Figure 23 The effect of high shear mixing on RE1 in bitumen 170 at 180°C

The modulus and phase angle results in Fig. 23 suggest that high shear mixing had a negligible effect on the properties of the digestion.

Derived Parameters

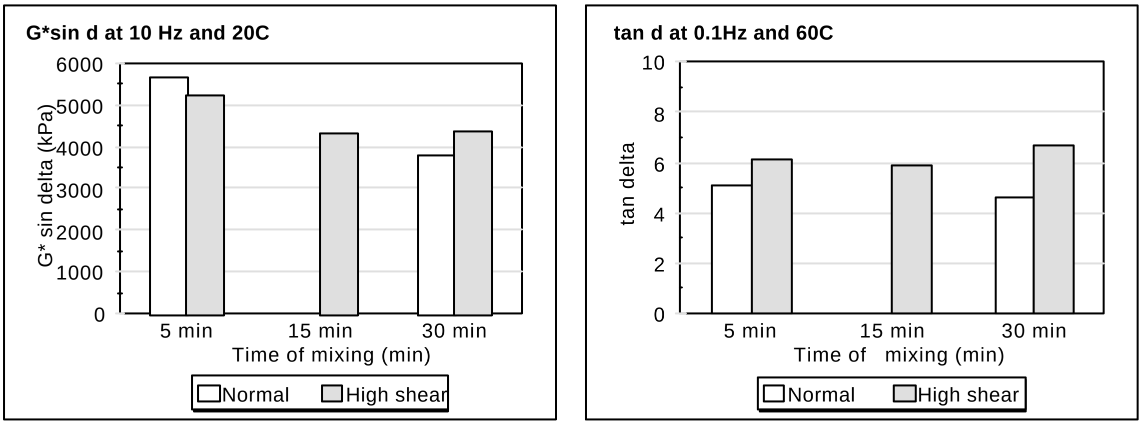


Figure 24 The effect of high shear mixing on G sin delta and tan delta for digestions at 180°C*

The parameters G* sin delta and tan delta also indicated that there was little difference between high shear and normally mixed digestions.

Angular Recovery and Toughness

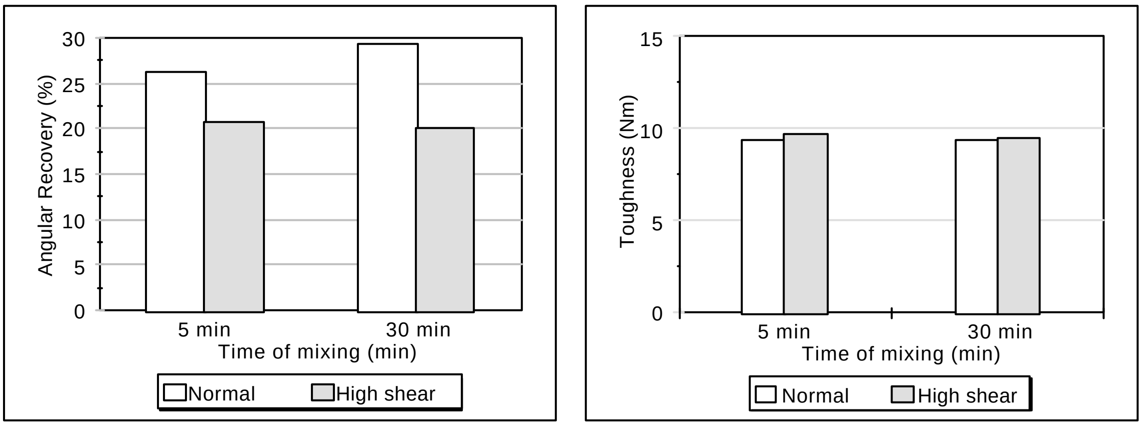


Figure 25 The effect of high shear mixing on angular recovery and toughness for digestions at 180°C

Angular recovery and toughness results showed either negligible difference between the control and high shear samples or, in the case of angular recovery, a reduction with high shear mixing.

5.6.3 Summary

All the performance indicators suggest that the high shearing mixing regime used had little or no effect on the important properties of the mix. Only one set of conditions (rotor speed, separation distance between rotor and fixed plate, etc.) was tried and it is possible that other conditions might produce a different result. The appearance of the high shear digestions was smoother than the control samples suggesting that a major effect of the particular high shear mixer used might have been to size reduce the rubber particles.

5.7 Crumb Rubber Preblends

5.7.1 Procedure

Samples of crumb rubber preblended binders provided by external suppliers were heated and cast into test specimens for dynamic shear rheometer and extensiometer testing.

5.7.2 Results

Modulus and Phase Angle

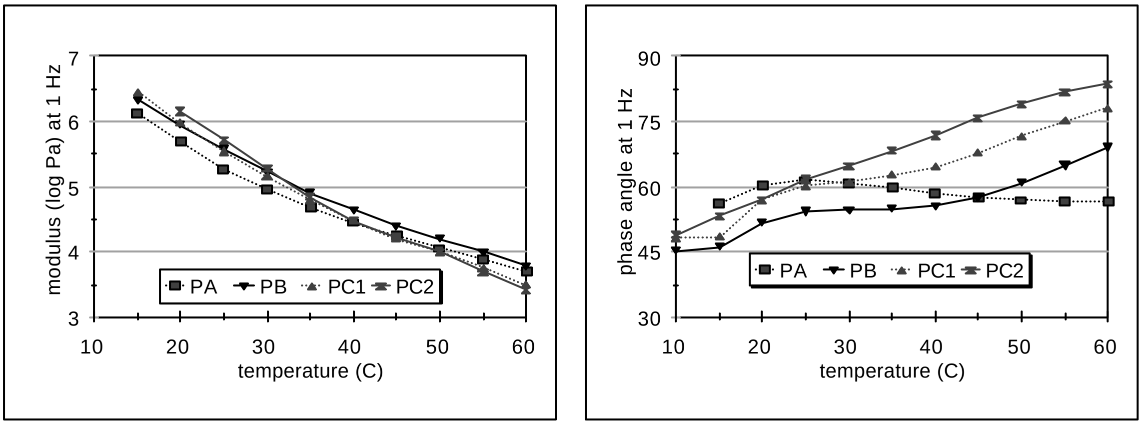


Figure 26 Modulus and phase angle vs temperature plots for four preblended binders

The properties of binder PA changed less with temperature than any of the other binders - a desirable property for good service performance. PC2 showed the greatest change with temperature.

Derived Parameters

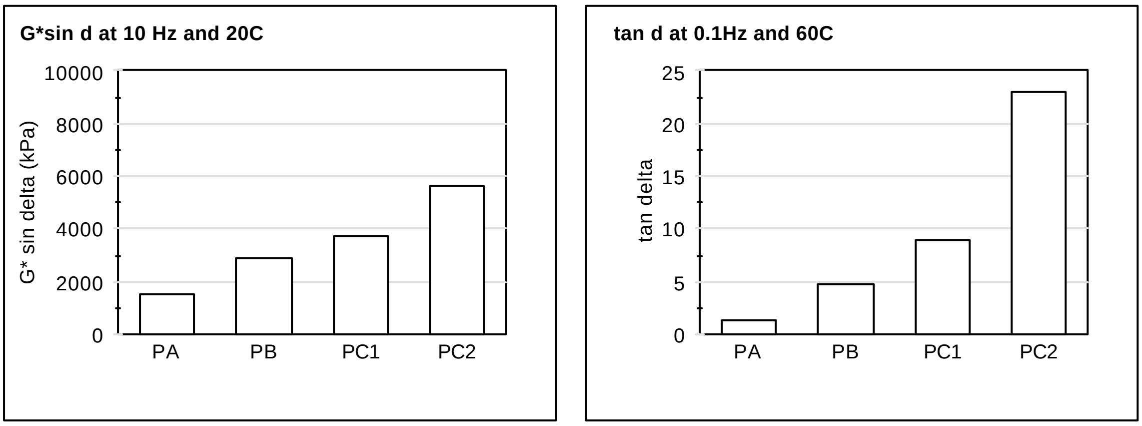


Figure 27 G sin delta and tan delta for four preblended binders*

Binder PA exhibited the lowest G* sin delta value - an indicator of good fatigue performance - while PC2 was the poorest performer. A similar ranking was obtained for tan delta.

Angular Recovery and Toughness

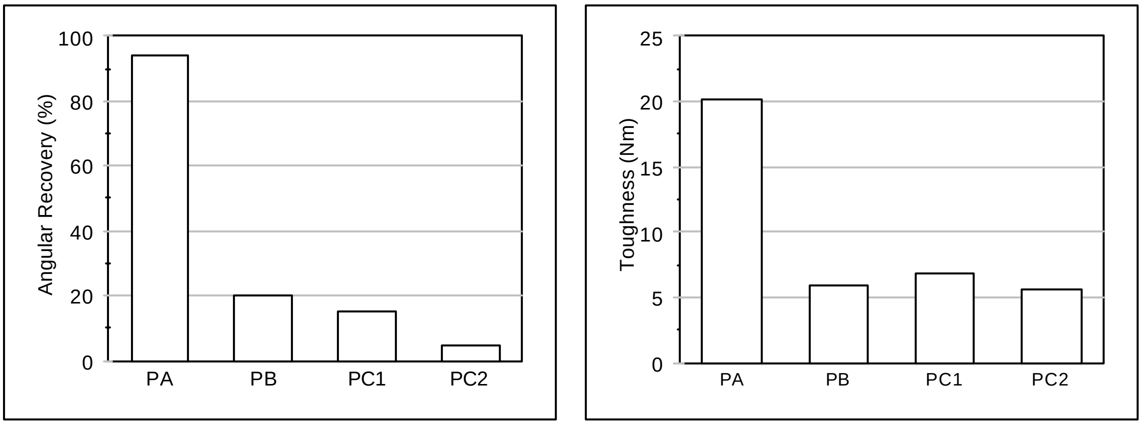


Figure 28 Angular recovery and toughness for four preblended binders

Again binder PA was superior to the other binders in terms of angular recovery and toughness. The remaining three binders generally showed similar levels of these properties.

5.3 Summary

The properties of binder PA were superior to the other three binders for each of the fatigue performance indicators: G*sin delta, angular recovery and toughness. The remaining binders showed inferior values of the indicator properties, with PC2 being the poorest performer.

6. Comparing the Performance of Different Binders

6.1 Introduction

Because of overlapping results it is difficult to compare all materials tested on one graph. The approach adopted below for the modulus and phase angle graphs is to compare the best and worst digestions, the best and worst preblends, a Class 170 bitumen and an SBS binder. For the other graphs, binder PB and digestion RE1 have also been included, as well as RE1 with extender oil labelled “Oil” on the graphs. The digestions shown were all 15% by mass of rubber digested for 2 h at 180°C.

6.2 Binder Data

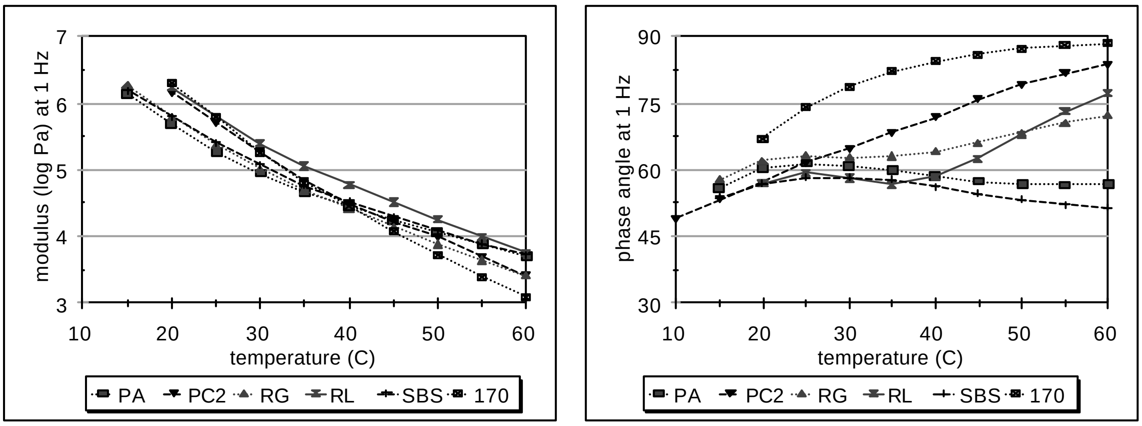


Figure 29 Modulus and phase angle comparison for six binders

The phase angle results, in particular, clearly indicate that there were major differences between the binders. All the rubber modified binders were more elastic at all temperatures than bitumen. PA and SBS have remarkably flat phase angle vs temperature curves suggesting that they will have both good fatigue resistance and good rut resistance. The SBS binder tested has been used in an ALF pilot trial, and performed very well in laboratory fatigue testing. It seems likely that binder PA will have similar properties.

Binder PC1 had the greatest phase angle vs temperature slope and is therefore unlikely to provide good performance in the field.

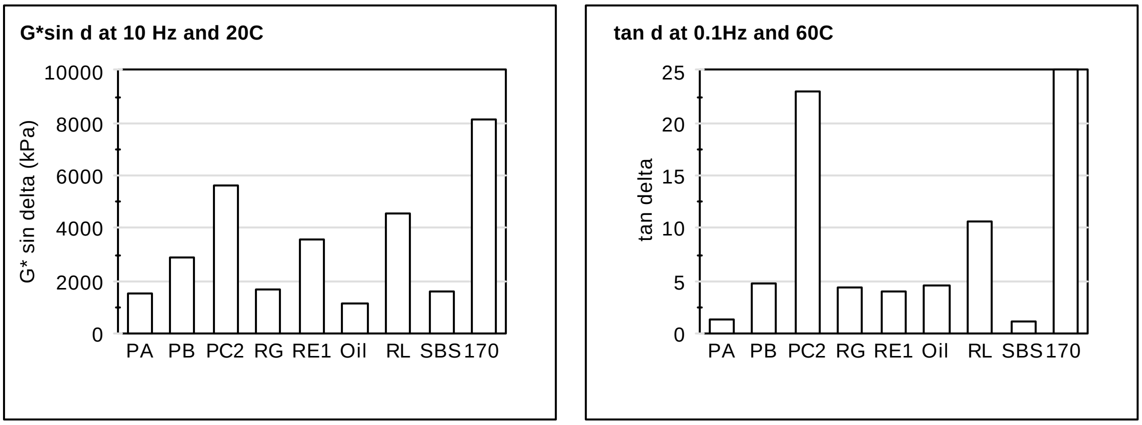


*Figure 30 G*sin delta and phase angle comparison for nine binders*

The G*sin delta plot confirms that binder PA can be expected to give similar fatigue performance to the SBS binder. The result for digest RG suggests that it should also provide good fatigue performance.

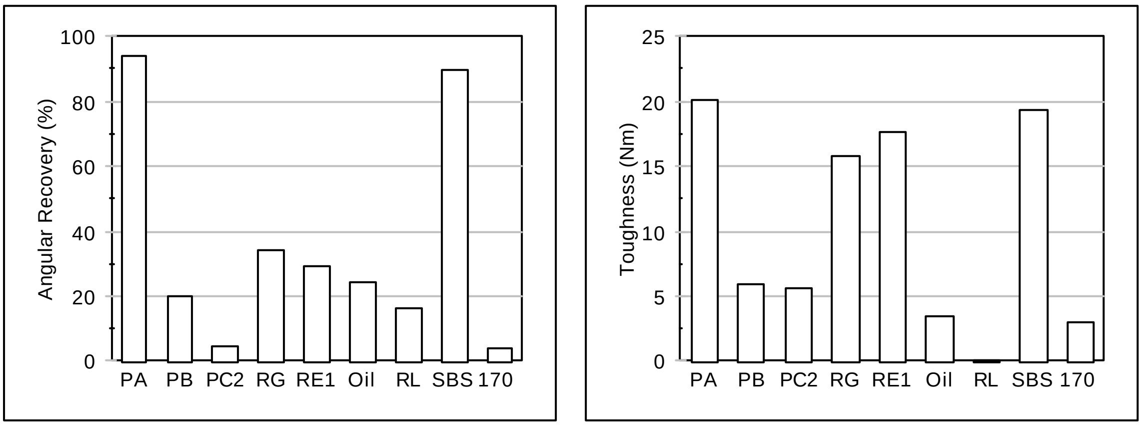


Figure 31 Angular recovery and toughness comparison for nine binders

Both the angular recovery and toughness results indicate that the fatigue performance of binder PA should be similar to that of SBS. Binder RG might be expected to provide a reasonably high level of fatigue resistance.

6.3 Summary

Nearly all the major performance indicators used suggest that binder PA will give good fatigue resistance and that its performance should be similar to that of the SBS binder tested. The SBS binder was a premium grade polymer modified binder developed for asphalt use and provided excellent fatigue resistance in laboratory testing.

The 2 h digestion at 180°C of rubber RG in bitumen 170 also provided good fatigue resistance as indicated by G* sin delta, and the RE1 digestion gave good toughness results. It is possible that more severe digestion conditions might produce even better results.

7. Conclusions

1. All the fatigue performance indicators ranked binder PA as best of the digestions and preblended crumb rubber binders tested. The indications were that there would be no problems with the rut resistance of mixes incorporating this binder, making it the prime candidate for inclusion in asphalt fatigue trials.
2. Time and temperature of digestion are interdependent i.e. increase in digestion time or digestion temperature (up to a limit) can result in improved properties.
3. Digestion of crumb rubber in bitumen at 220°C for periods greater than 2 hours (possibly 4 hours) may provide optimum properties for most blends.
4. Exposure at high temperature can cause degradation of crumb rubber binder properties particularly where there is ready access of the binder to air.
5. A high bulk density rubber tested gave results indicative of poor fatigue and rut resistance.
6. There was an indication that smaller particle size rubbers gave more rut resistant digestions but the differences were not great nor the results conclusive.

7. High shear mixing, using one set of conditions, appeared to have no effect on the fatigue resistance of the digestion tested.
8. Addition of extender oil produced conflicting results, with one test indicating a marked improvement in fatigue resistance and another test a marked reduction in fatigue resistance.

8. Selection of Binders for the Asphalt Study

8.1 Introduction

In the asphalt study, mixes will be manufactured with different crumb rubber binders and the fatigue resistance of compacted specimens measured using a laboratory fatigue test which has been under development in Australia (and elsewhere) for several years. In this section of the report, selection of binders for the asphalt study is discussed.

8.2 Discussion

The results clearly indicate that the preblended binder PA might be expected to have superior fatigue resistance to the other binders tested. It is proposed that this binder be used for the basic study to determine the binder content for the crumb rubber asphalt mix.

A bitumen should be included as a control and the choice is between a Class 170 and a Class 320. The softer Class 170 might be expected to provide better fatigue resistance but Class 320 has been used as a control in the ALF pilot fatigue trial.

A small number of other binders should be tested to determine the extent of the improvement which can be obtained with different binders and to obtain data so that a correlation between binder properties and asphalt fatigue resistance can be developed. These relationships will assist in the development of crumb rubber binder specifications and in the selection of the appropriate binder for a particular job.

Suitable candidates for binders with expected intermediate fatigue properties are digestions of RG and RE1 and preblended rubber PB.

Some tests suggested that the addition of extender oil improved fatigue performance while others did not. It may be useful to include an oil extended modification of one of the above binders. The fatigue results will not only indicate whether oil is beneficial but should assist in determining which of the performance indicators is more reliable.

8.3 Recommendations

1. The preblended rubber PA should be used to manufacture specimens for determination of the preferred composition (grading and binder content) of the crumb rubber asphalt mix.
2. A binder content of around 7% should be targeted. This needs to be a compromise between high fatigue performance (fatigue resistance is directly related to binder content) and cost, while still ensuring satisfactory high temperature rut resistance.
3. The ALF 320 bitumen should be used as the control.
4. Mixes with the selected grading and binder content should be manufactured with rubber binders of (expected) intermediate fatigue resistance. Suitable binders may include PB, RG and RE1.
5. One of the above binders should also be manufactured with an extender oil and included in the program.

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