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Highlights

- A Liquid Rubber (LR) is presented as solution to overcome common issues of asphalt rubber binders.
- LR-bitumen blends are manufactured at several temperatures and with various modifier contents.
- High temperature rheology, service temperature properties and storage stability are assessed.
- Analysis includes a comparison with a neat bitumen and a SBS modified bitumen.
- Results show that liquid rubbers could represent a breakthrough within the bitumen industry.

The final publication is available at

<https://doi.org/10.1016/j.conbuildmat.2018.03.226>

Citation:

D. Lo Presti, M.A. Izquierdo, A. Jiménez del Barco Carrión, Towards storage-stable high-content recycled tyre rubber modified bitumen, Construction and Building Materials, Volume 172, 2018, Pages 106-111, ISSN 0950-0618, <https://doi.org/10.1016/j.conbuildmat.2018.03.226>.

Keywords: Recycled tyre rubber; Liquid rubber; Bitumen modification; Storage stability; Asphalt rubber; No-agitation wet process

Towards storage-stable high-content recycled tyre rubber modified bitumen

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Abstract: The addition of crumb rubber particles as bitumen modifier can be currently considered as a well-established alternative to conventional polymers for bitumen modification. However, Recycle Tyre Rubber (RTR) modified binders still present drawbacks such as poor mix workability and hot storage stability. Within this study the authors try unlocking the full potential of devulcanised tyre rubber-heavy oils blend, named Liquid Rubber (LR), by exploring the possibility of tailoring recycled polymer modified bitumen with unconventional high-content of RTR and designed to overcome the above mentioned technological problems of RTR modified bitumen while keeping its advantages. Results show that LR-bitumen blends incorporating up to 30% RTR in weight of total binder clearly improves useful temperature interval of base bitumen by maintaining solubility values allowing them to be considered stable at hot-storage temperature. Furthermore, the LR modifier allows reducing usual manufacture temperatures up to 30°C by providing superior low and intermediate temperature rheology, however high service temperature properties are improved only at low strain.

Keywords: recycled tyre rubber, Liquid rubber, bitumen modification, storage stability, asphalt rubber, no-agitation wet process

26 **1. INTRODUCTION**

27 Bitumen modification has been carried out during decades in order to enhance performance
28 of conventional binder for asphalt mixtures. Traditionally, synthetic polymers of different
29 nature and functionality are used to enhance the resistance to the main distresses affecting
30 asphalt mixtures by increasing their useful temperature interval (UTI) [1]. However,
31 environmental concerns and economical savings opportunities have promoted the use of
32 recycled modifiers for bitumen. In particular, Recycled Tire Rubber (RTR) has been widely
33 studied due to the high saving of landfill space that its use implies and because of the good
34 performance it has shown as bitumen modifier [1]–[4].

35 When RTR is used as a modifying agent for bitumen, it is called crumb rubber modifier (CRM),
36 while the method of modifying bitumen with CRM before being incorporated into the mixture
37 is referred to as the Wet Process [5]. Bitumen – CRM interaction is material-specific and
38 depends on a number of basic factors, including processing variables (such as temperature,
39 time and applied shear stress), base binder properties (such as source and eventual use of oil
40 extenders) and CRM properties (such as source, processing methods, particle size and content
41 in binder) [6]–[9].

42 Depending on the adopted processing system and on the selected materials, the Wet Process
43 leads to two different technologies which main distinction is based on rotational viscosity of
44 the resulting rubberised binder at high temperature. On one hand, Wet process–high viscosity
45 is the traditional and more widely used technology obtained with mainly physical
46 modification process based on the swelling of 0–2mm CRM, reaction temperatures between
47 160°C and 260°C, reaction time varying from 45 minutes up to few hours and usually low
48 shear processing [9]. The final product allows obtaining a rubberised asphalt mix with benefits
49 that are basically linked to the binder’s increase in elasticity and viscosity at high

50 temperatures [10], [11]. Rubberised asphalt mixtures obtained with this technology showed
51 longer lasting performance compared to the conventional ones [12], [13]. However, these
52 materials require an accurate blend design and non-conventional continuous agitation during
53 hot storage to keep the RTR particles uniformly distributed. Moreover, they could present
54 several operative drawbacks such low workability, poor binder storage stability and the fumes
55 it emits during the paving process due to high operative temperatures (even 180°C).

56 The second technology is known as Wet process–No agitation and aims at producing CRM
57 bitumen not occurring in phase separation of the modifier from the binder during storage or
58 transportation. For this purpose, CRM has to be fully digested/dissolved into the bitumen
59 without leaving visibly discrete particles. This process prevents the constant agitation needed
60 in Wet Process-High viscosity and improves particles distribution and hot storage stability of
61 blends. The idea behind Wet-process-No agitation binders is therefore obtaining a technology
62 more similar to polymer modified bitumen rather than the above mentioned Wet Process-
63 High Viscosity. Some successful no-agitation wet process technology, such as terminal blends,
64 are already used however they still need to be manufactured at refineries and do not allow
65 achieving the superior performance provided by both the conventional wet process and
66 polymer modification [5].

67 The objective of this paper is to investigate the feasibility of unlocking the full potential of a
68 liquid polymer that has the potential to allow bitumen technologist to manufacturing in-
69 house storage-stable rubberised bitumen with very high-content of RTR. The above
70 mentioned liquid polymer was provided by innovator LLC and is named Liquid Rubber (LR). LR
71 is actually a family of semi-solid/fluid materials composed by a very high-percentage of RTR
72 plus other oils. In the recent past some LR was used in concrete [14] as well as bitumen
73 modifier by Fini et al. [15] who tested blends of bitumen and 15% LR showing that it can

74 enhance its low-temperature characteristics but adversely affects the base bitumen elasticity
75 and thus its resistance to rutting. This investigation goes a step further and investigates the
76 potential of maximising the re-use of RTR in bitumen modification by incorporating LR as
77 modifier in proportions from 5% to 60%. The experimental programme consisted in
78 monitoring high temperature rheology (Rotational viscosity) at different temperatures, as
79 well as assessing service temperature rheological and performance-related properties of the
80 blends. At last, solubility tests have been performed to estimate the behaviours of the blends
81 during hot storage. In order to have a direct feel of the level of modification obtained by using
82 several concentration of LR, the measured properties have been compared with those of the
83 base bitumen as well as with a Styrene-Butadiene-Styrene Modified Bitumen (SBS-MB)
84 currently used for asphalt mixtures.

85

86 **2. MATERIALS AND METHODS**

87

88 **2.1. Materials**

89 In this study the LR, an innovative modifier incorporating 50% RTR and 50% oils, is blended
90 with a 40/60 bitumen in different proportions from 5 to 60% of the final binder. Liquid Rubber
91 is a technology supplied by Innovators LLC in USA and obtained with a proprietary process
92 composed from a digestion tank, a main reactor and a cooling unit. In the first two tanks the
93 #8 (2.36 mm) fibre and glass free RTR is subjected to a process of devulcanisation and mixed
94 with heavy oils derived from petroleum and/or soy. The process is customisable, allows
95 having control of the off gases, and has a production rate of 12-25 gal/h of LR. The end product
96 is a sticky visibly homogeneous fluid that varying the composition and processing conditions

97 can have different viscosities (Figure 1). No other details of the production process have been
98 disclosed to the authors.



99
100 **Figure 1. Liquid Rubber (LR) aspect**

101 As mentioned earlier, two different binders were considered as references for the comparison
102 of LR and bitumen blends, namely: conventional 40/60 penetration grade bitumen (40/60
103 bitumen) and SBS-polymer modified binder (SBS-MB). Conventional properties of these
104 binders are shown in Table 1. The LR is a modifier not a bitumen and therefore it was decided
105 not to perform its conventional characterisation. Instead a complete rheological
106 characterisation of the high-temperature viscosity (Figure 2) and intermediate-temperature
107 rheology (Figure 5) was carried out. Figure 2 reports the high-temperature viscosity of the LR
108 and the reference material. A significant non-Newtonian behaviour of LR was detected during
109 the test within the temperature range 135°C – 200°C. For this reason, all measurements are
110 referred to a speed of 100 rpm in the rotational viscometer. As a result LR presents viscosity
111 values that are higher than the control binders at all considered temperatures.

112 **Table 1. Properties of conventional bitumen and SBS-M**

	40/60 bitumen	SBS-MB
Penetration at 25°C (dmm) – EN 1426	46	87
Softening Point (°C) – EN 1427	50.8	103

113

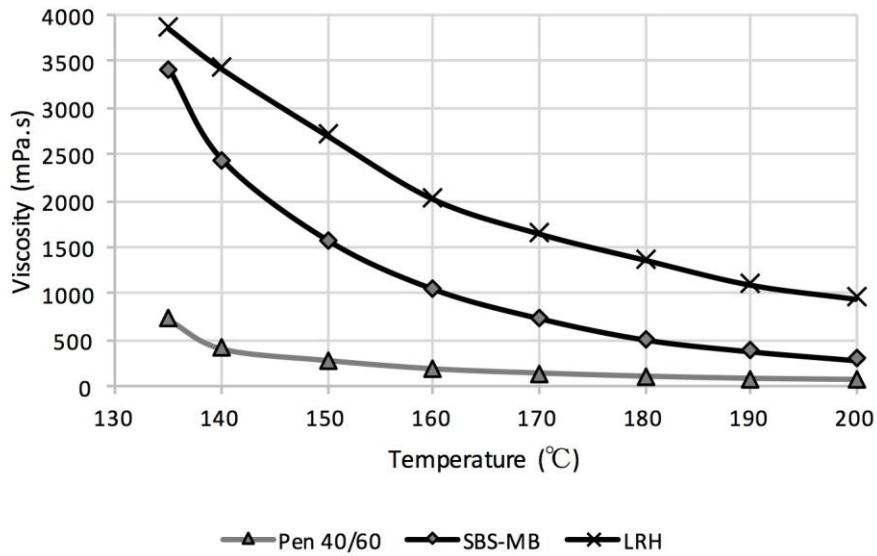


Figure 2. Rotational viscosity at 100 rpm of the reference materials vs. temperature

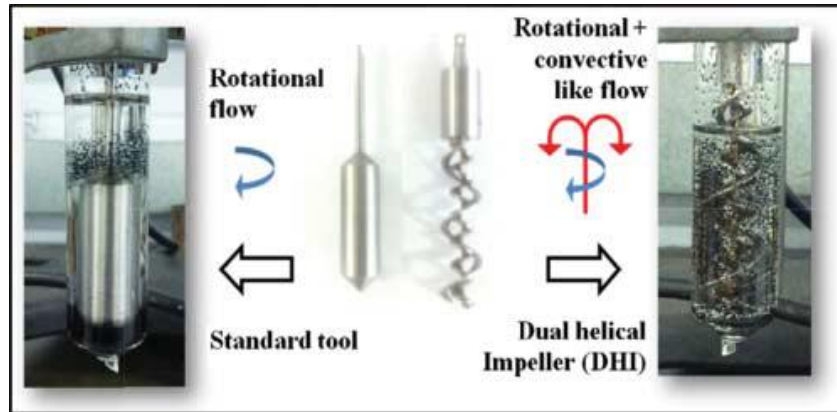
2.2. Methods

2.2.1. LR-bitumen blends' manufacture and high-temperature rheology

Rotational viscosity was the physical parameter controlled during the modification process. Therefore, as starting point, rotational viscosity tests build-up by means of low-shear blending protocol [17] were performed on the 40/60 bitumen and LR separately to assess their high-temperature rheology which in turns provide technical information for the LR-bitumen blend preparation.

Being the modifier in a liquid form, the blends were manufactured with very low-shear (200 rpm) by adapting a Brookfield Viscometer to operate as a mixer providing real-time viscosity measurements under a controlled environment. The use of a Dual helical impeller (Figure 3) allowed enhanced dispersion of the modifier during the mixing process [16]. When rubber and bitumen are being blended, the viscosity of the blend increases up to a maximum value (peak viscosity) and then suffers a plateau or a noticeable decrease with the reaction time. This stage is recognized as the peak of the modification process and was fixed at the point

130 where the viscosity did not increase more than 5% within 15 minutes [17]. The mixing was
 131 then continued for one hour more in order to ensure the stabilization of properties having a
 132 blending time of 75 minutes.



133

134

Figure 3. The Dual Helical impeller adapted by [16]

135

136

137

Table 2. Manufactured 40/60 bitumen – LR blends

BLEND NOMENCLATURE	LR content (%m/m)	Processing TEMPERATURE (°C)
5%LR	5	180
9%LR	9	180
29%LR	29	180
37.5%LR	37.5	180/150
50%LR	50	180/150
60%LR	60	180/150

138

139 LR percentages (in part of total mass) to blend with conventional bitumen were chosen
 140 varying from 5% up to 60%. Blends were produced at 180°C, which is a common manufacture
 141 temperature for rubberised binders. However, in order to have a clearer understanding of the
 142 modification process and to explore potentials of this modifier, the blends with higher LR
 143 percentages (over 29% LR) were also manufactured at 150°C. Figure 3. The Dual Helical impeller

144 adapted by [16]

145

146

147 Table 2 shows the manufactured blends of 40/60 bitumen and LR where LR proportions are
148 expressed in part of total mass.

149

150 **2.2.2. Estimating storage stability through solubility tests**

151

152 In order to assess potential storage stability issues of bitumen-LR blends, solubility tests were
153 carried out. Tests were performed with standard configuration (EN 12592:20114). Only for
154 the highest LR content (over 37.50% of the total mass) modified solubility tests were required
155 because tire crumbs were not dissolved completely and remained in particulate form during
156 interaction with bitumen. The modified test is widely used for CRM blends and consists on
157 using a wider sieve size (#200 - 75 μ m) [19]. This sieve prevents the equipment from getting
158 blocked due to bigger insoluble particles arising from the difficulties in the digestion/breaking
159 down of RTR particles during the manufacturing process of the blends.

160 **2.2.3. Service temperature advanced rheological investigation**

161 The core of the assessment was performed through rheological measurements carried out by

162 means of Dynamic Shear Rheometer (DSR) tests at asphalt mixtures service temperatures.

163 The controls and manufactured bitumen-LR blends were subjected to frequency/temperature

164 sweeps from 0.1 to 10 Hz and 0 to 80°C within their linear-viscoelastic range of strain. In these

165 tests, binders' rheological properties were measured in terms of the norm of the complex

166 (shear) modulus, $|G^*|$ (Pa), and phase angle (viscoelastic balance of rheological behaviour),

167 δ (°). Once measured, the data were used together with the time-temperature-superposition-

168 principle (TTSP) and shift factors to produce master curves at 30°C. At this point, only the

169 blends judged to be the most promising were selected to continue with low-temperature

170 properties characterisation and performance-related properties.

171

172 **2.2.4. Performance-related tests and low-temperature characterisation**

173 Multiple Stress Creep Recovery tests (MSCR) according to ASHTO T 350-14 and Time Sweep
174 according to J.P Planche et al. [18] tests were performed in order to evaluate respectively
175 rutting and fatigue resistance of binders. Both tests are performed in the DSR. MSCR test [20]
176 is used to assess binder ability to recover deformation, and consequently, their potential
177 resistance to rutting in the mixture. The test consists on the application of ten creep-recovery
178 cycles to a sample at different stress levels. Each cycle lasts 10 seconds, during which the
179 stress is applied for 1 second (creep) and then the sample is allowed to recover during 9
180 seconds (recovery). In this study, two levels of stress are considered: 0.1kPa and 3.2kPa. In
181 this sense, binders are evaluated within and without the linear-viscoelastic response range.
182 Tests were performed at 60°C by using the 25 mm parallel-plate geometry in the DSR.
183 On the other hand, Time Sweep is used to evaluate fatigue resistance [18]. In this test, binder
184 samples are subjected to a cyclic load in the DSR until failure. Failure criteria was selected as
185 the cycle in which the initial norm of the complex modulus drops 50% [19],[20], [21]. Tests
186 were carried out in stress-controlled mode at a frequency of 10Hz. Binders were tested at
187 20°C, which is an intermediate temperature at which fatigue phenomenon might take place,
188 and using the 8 mm parallel-plate geometry. Fraass Breaking point of the best LR-bitumen
189 blends was obtained according to the EN 12593:2015 in order to complete their
190 characterisation in the whole range of temperatures.

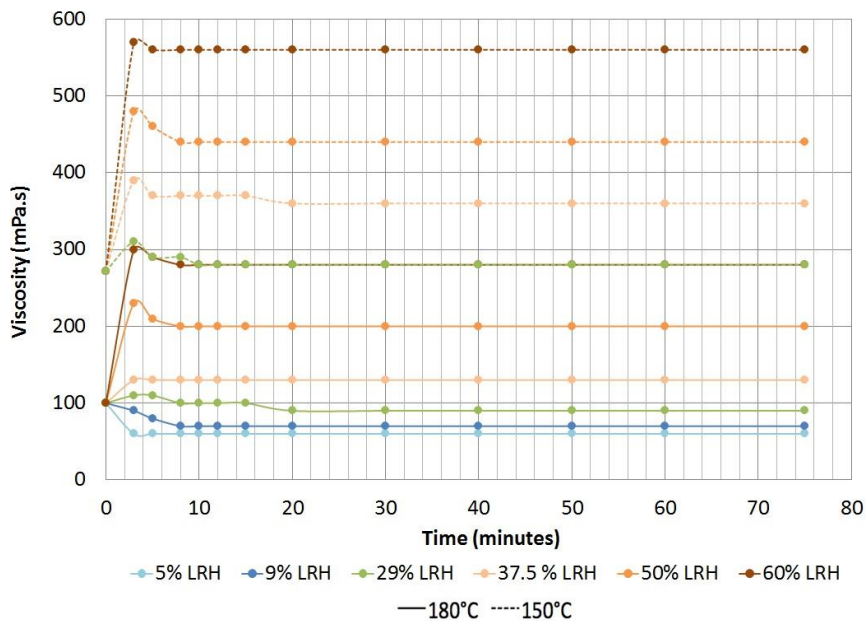
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192 **3. RESULTS AND DISCUSSION**

193 **3.1. LR-bitumen blends' manufacturing and high temperature rheology**

194 Figure 4 shows the high-temperature viscosity measurements of the LR-bitumen blends.

195 Viscosity values were taken during the 75 minute – manufacture of the blends at 180°C and
 196 150°C. It can be observed that LR provides an appreciable modification of bitumen properties
 197 (higher viscosity) only when at least 37.5% of mass of bitumen is replaced. For all cases, LR
 198 allowed achieving peak viscosity and properties stabilisation in less than 20 minutes. In
 199 addition, it can be said that LR is a very different modifier from the CRM. Results show that,
 200 regardless of the processing temperature, the reaction between LR and bitumen does not
 201 involve any swelling process and/or devulcanisation, depolymerisation and further digestion
 202 that are typical of bitumen modification with CRM [5]. Furthermore, LR-bitumen blends
 203 manufactured at 150°C do not show undergoing a different modification process than those
 204 manufactured at 180°C. This temperature is well below the typical processing temperature of
 205 CRM-bitumen blends.



206 **Figure 4. Rotational viscosity at 100 rpm of the reference materials vs. temperature; (b) Rotational viscosity of the**
 207 **blends vs. manufacture time**

208
 209 In Figure 4 it can also be observed that final viscosity of the blends is always below 1500 mPa.s
 210 (even with 60% LR and 150°C manufacture temperature), which is a typical value for
 211 conventional CRM modification [5]. These results show that LR-bitumen blends are far from

212 being classified as High Viscosity - Wet process and that despite of the high amount of
213 recycled tire rubber that they contain, they could provide good workability towards a
214 significant reduction in asphalt concrete manufacturing temperatures and relative energy
215 consumption.

216
217 **3.2. Hot storage stability through Solubility tests results**

218 Solubility tests were carried out for the blends manufactured at 180°C. Modified EN 12592
219 permitted to easily perform solubility tests of the blends with higher LR content. Results are
220 shown in Table 3 where it can be observed that LR-bitumen blends have solubility almost
221 comparable to the neat bitumen. This fact would be favourable for hot storage stability and
222 allow classifying these binders as No Agitation – Wet process. In order to check the effect of
223 processing temperature, the 29% LHR blend manufactured at 150°C was also subjected to
224 solubility test showing that solubility is not affected by processing temperature. These results
225 were considered satisfactory to reach a conclusion on storage stability, hence solubility tests
226 on the LR itself were not been carried out.

227
228

Table 3. Solubility test results

BLEND	% Soluble Material	% Insoluble Material
SBS-MB	99.15	0.85
Neat	99.93	0.07
5% LR	99.44	0.56
9% LR	99.05	0.95
29% LR	97.62	2.38
29% LR (150°C)	97.75	2.25
37.5% LR*	98.72	1.28
50% LR*	98.26	1.74
60% LR*	97.7	2.3

229 * Solubility tests performed with the modified configuration

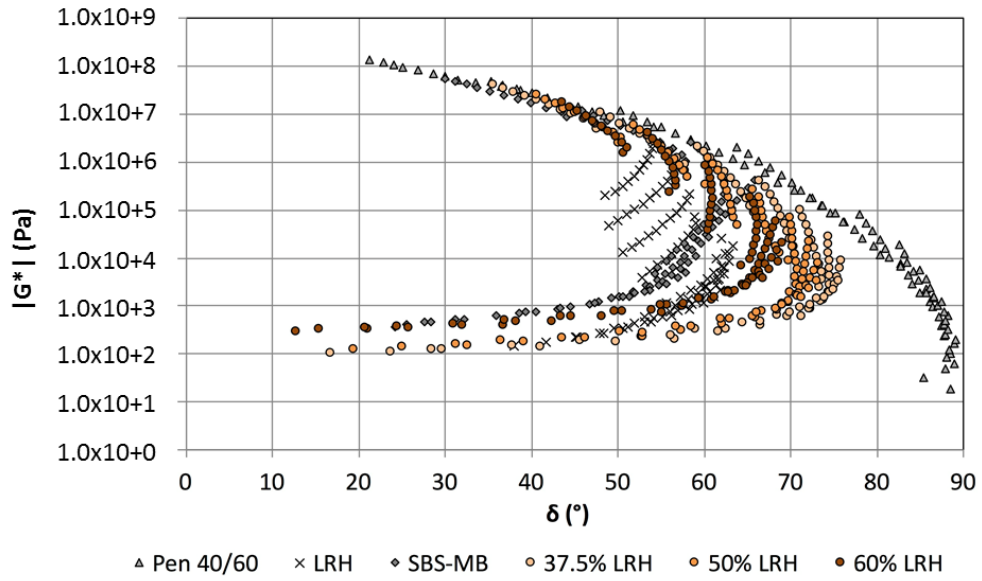
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234 **3.3. High and Intermediate service temperature rheology**

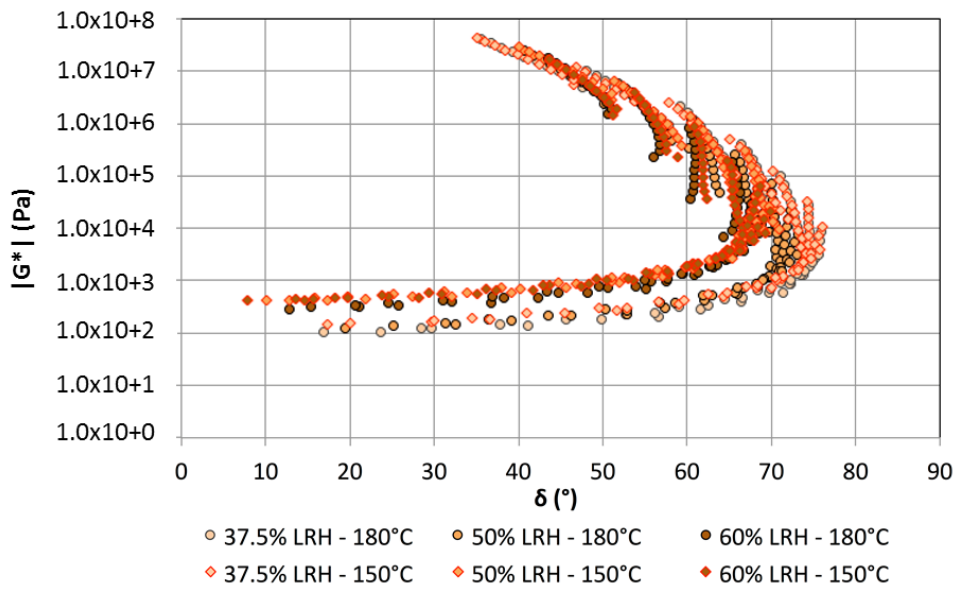
235 Advanced rheological characterisation was performed for the reference materials and LR-
236 bitumen blends. However, only those blends showing a significant modification of bitumen
237 properties are shown in Figure 5 in terms of Black Diagrams and master curves at 30°C.

238 LR shows to be a complex material that is non-Newtonian and non-thermo-rheologically
239 simple. Black Diagrams are the rheology fingerprint of a material and LR-bitumen blends have
240 comparable behaviour to the SBS-MB. In particular, the blend with 60% of LR shows having a
241 very similar rheology to SBS-MB with similar stiffness and elasticity over the whole range of
242 temperatures and frequencies. LR provides the blends with the benefit of a more viscous
243 behaviour at low temperatures (higher value of phase angle), but with a much lower stiffness,
244 especially at high temperatures (low frequencies). Higher content of LR leads to blends with
245 higher elasticity but lower stiffness over the whole range of temperature and frequencies.
246 This is in line with the composition of the modifier that provides an overall decrease in rigidity
247 at intermediate and low temperatures, due to the enhanced elastic behaviour conferred by
248 the rubber and decrease in stiffness due to the oils fraction.

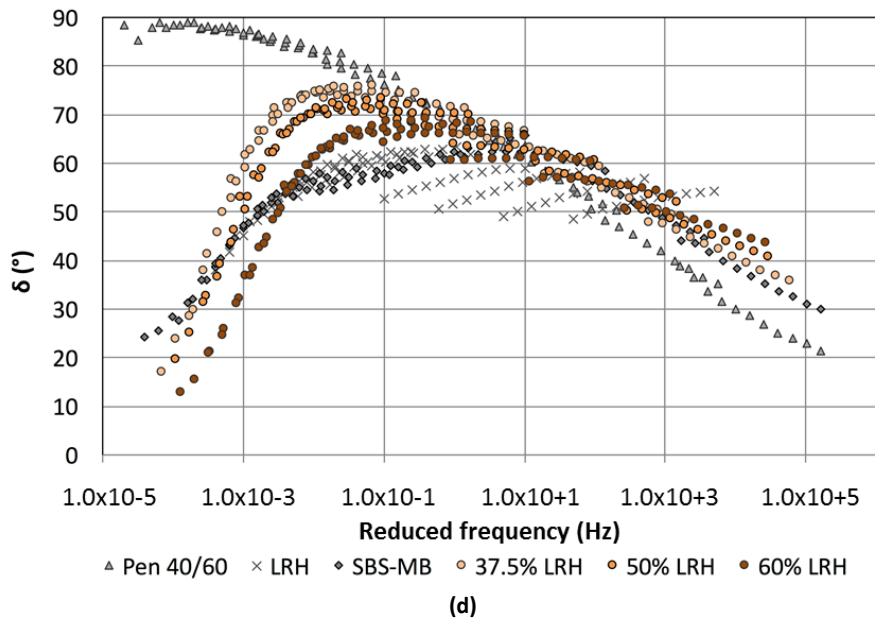
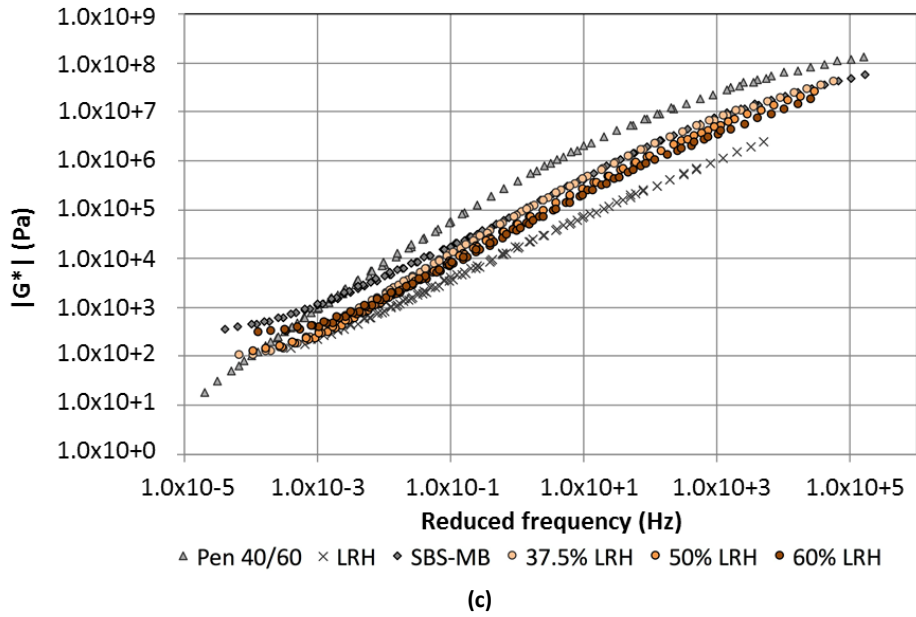
249 Figure 5 (b) shows that Black diagrams of the blends do not change whether they have been
250 manufactured at 150°C instead of at 180°C. In this sense, LR allows modifying bitumen with
251 high RTR content at a 30°C lower temperature than that used in classical processes with CRM.



(a)



(b)



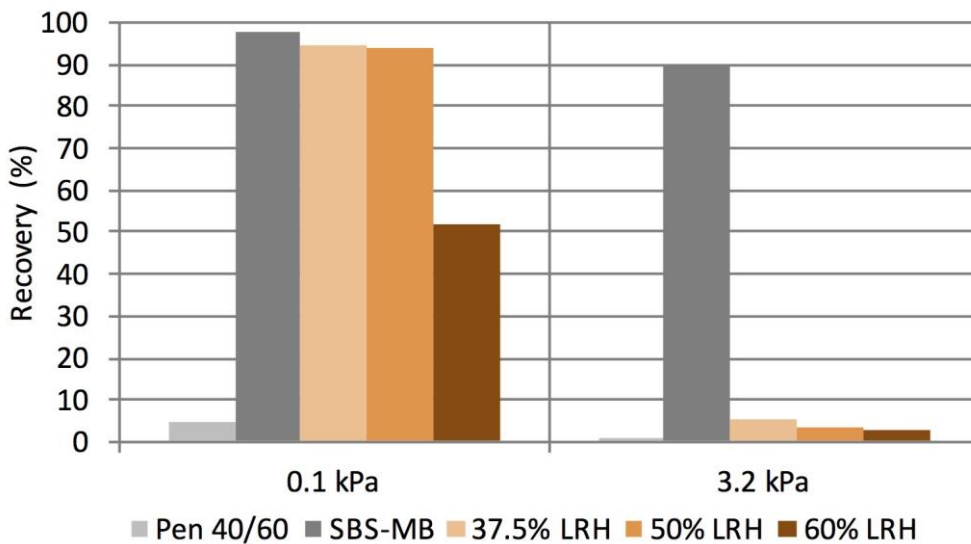
252 Figure 5. (a,b) Black diagrams; (c) Complex modulus master curves at 30°C; (d) Phase angle master curves at 30°C

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254

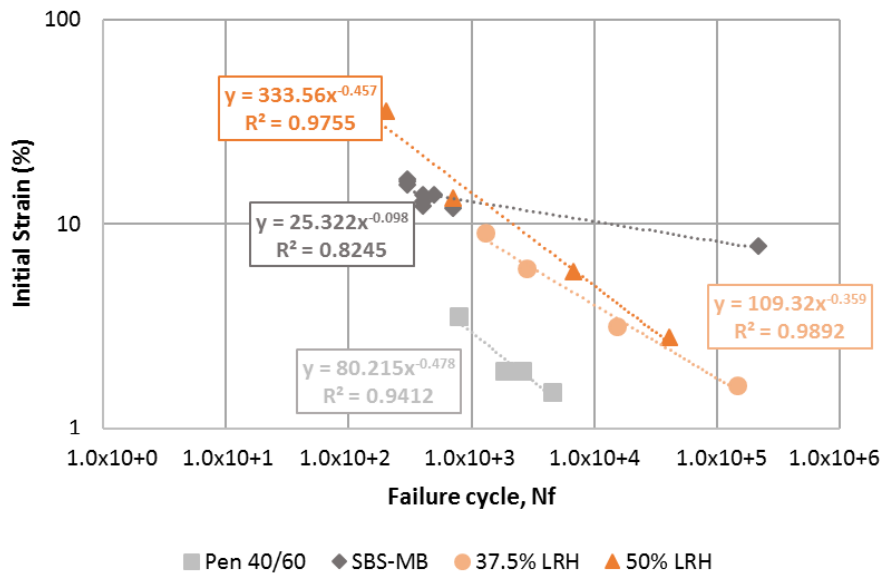
255 **3.4. Performance-related and low-temperature properties**

256 MSCR tests and Time Sweeps were carried out to better investigate the performance of the
 257 blends to resist different asphalt pavement distresses such as rutting and fatigue. Results are
 258 shown in Figure 6.

259 In Figure 6 (a), within the linear viscoelastic region (0.1 kPa), LR-bitumen blends perform well
260 as indicated by the high recovery that is higher than the 40/60 bitumen and comparable to
261 that of SBS-MB. This result appears to be in contrast to was found by previous researches [14]
262 and may be due to the nature of the LR or the different modification percentages. As the level
263 of stress increases, the modification is totally vanished and the material properties change.
264 This fact can be attributed to the absence of networking between the LR and the base
265 bitumen which in turns does not sustain higher strains and eventually breaks. The oil agent
266 contained in the LR might be the main factor that makes the modified material softer and
267 even weaker than neat bitumen. Higher content of LR leads to blends with lower recovery.
268 This shows an absence of a network between the modifier and the bitumen, which is
269 distinctive of commercially used SBS-MB. For this reason, 60% LR blend was discarded for
270 further tests.
271



(a)



(b)

Figure 6. (a) MSCR tests results at 60°C; (b) Time sweeps results at 20°C

272

273

274 Fatigue results are displayed in Figure 5 (b) and show that LR blends have similar or lower

275 slopes than the 40/60 bitumen, meaning that susceptibility to strain level is not significantly

276 different. However, LR shows having much higher more loading cycles until failure for the

277 same initial strain (more than an order of magnitude), which leads to assess that LR

278 modification greatly improves fatigue life of 40/60 bitumen. On the other hand, when

279 compared to SBS-MB, the fatigue resistance of LR blends is comparable to the SBS-MB only

280 at high strain levels (>10% which happens in pavements with heavy traffic), however, as the

281 initial strain decreases, SBS-MB exhibits much longer fatigue life due to the lower slope of its

282 fatigue law. Therefore, LR-bitumen blends overall significantly increase fatigue-related of

283 conventional bitumen, while it shows the lack of a polymer network when compared to SBS-

284 MB who shows superior fatigue-related properties in both strain susceptibility and fatigue

285 life.

286

287

288

289

Table 4. Fraass Breaking Point results

	Pen 40/60	SBS-MB	37.5% LR	50% LR
Fraass Breaking Point (°C)	-8	-19	-15	-25

290

291 At last, the binders were compared in terms of low-temperature properties. LR-blends low-
292 temperature rheology was deeply investigated in a previous investigation that clearly proved
293 how the oils present in the LR provides significant benefits to the LR-blends [14]. Hence, with
294 the aim of confirming this trend also by looking at conventional properties, in this
295 investigation the Fraass Breaking Point of the different binders was tested. Table 4 displays
296 that LR modification is able to decrease Fraass Breaking point of the bitumen more than 3
297 times in the case of the 50% LR blend. Furthermore, Fraass breaking points of LR-bitumen
298 blends are comparable or even better than that of SBS-MB. Although this test is not a strictly
299 performance-related test, Fraass breaking point is highly linked to low-temperature cracking
300 resistance of the material [22] and therefore it can be said that overall LR-bitumen blends
301 most-likely will improve low-temperature cracking behaviour of a conventional asphalt
302 pavement to a level comparable or even superior than SBS modified asphalt mixtures.

303

304 4. SUMMARY AND CONCLUSIONS

305 Recycling tyre rubber in asphalt pavement is a successful practice that is unfortunately not
306 use worldwide also due to important technological issues such as workability issues and the
307 lack of storage stability at high-temperatures. Liquid Rubber is a blend of heavy oils and
308 devulcanised recycled tyre rubber that allows an innovative delivery process for incorporating
309 tyre rubber in asphalt mixtures. This technology was explored in few previous investigations,
310 although none of the tried unlocking its full potential and shown that LR can allow
311 manufacturing superior bituminous binder incorporating unconventional high-content of

312 RTR. For this purpose, conventional bitumen has been modified with different percentages of
313 the modifier, corresponding to up to 30 wt.% of recycled Tyre Rubber. Blends' manufacturing
314 process was monitored in real-time through high temperatures rotational viscosity. In
315 addition, the effect of using different manufacturing temperatures was evaluated. LR-
316 bitumen blends were studied in terms of rheology, hot storage stability potential,
317 performance-related and low-temperature properties and were compared to the base
318 bitumen and a commercial SBS-MB. The following conclusions can be drawn:

- 319 - Overall, the selected LR is a non-Newtonian and non-thermorheologically simple
320 material that significantly decreases bitumen rigidity at low and intermediate
321 temperatures and, at the optimum content, can also provide a moderate increase in
322 resistance to plastic deformation of the base bitumen.
- 323 - LR-bitumen blends need much shorter processing time than standard asphalt rubber.
324 In fact, the LR do not show any sign of digestion/swelling during the manufacturing
325 and therefore seems not to require the high processing temperatures needed than for
326 the classical processes with CRM.
- 327 - Even with a recycled tyre rubber content of 30% in weight of total binder, these blends
328 have viscosities values well lower those of conventional asphalt rubber binders. This
329 of course can allow to significantly reducing asphalt manufacturing processing
330 temperatures as well as facilitating laying and compaction issues typical of rubberised
331 asphalts.
- 332 - LR-bitumen blends have very high solubility that indicates their potential to obtain
333 good storage stability. These facts allow classifying these blends as No Agitation – wet
334 process technology.

335 - LR-bitumen blends exhibits comparable rheology to commercial SBS-MBs. When
336 optimum dosage is achieved they show superior low-temperature properties, due to
337 the presence of heavy oils, longer fatigue-life than conventional bitumen although
338 they generally have not satisfactory stiffness and elastic recovery at high service
339 temperatures. These facts could be both attributed to the lack of structured network
340 which is instead a distinctive characteristic of polymer modified bitumen

341

342 Overall, results showed that liquid rubbers technologies could be already used in many
343 application within the bitumen industry, although further research should focus on
344 investigating the ageing potential of these blends. Currently, authors are also investigating
345 suitable solutions for improving LR-bitumen blends resistance to plastic deformations.

346

347 **ACKNOWLEDGEMENTS**

348 This work is part of a research project sponsored by a KTP Programme (Knowledge Transfer
349 Partnership No. KTP9265). The authors gratefully acknowledge its financial support.

350

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