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# 2020-005, 2022-006, 2024-001: Digestion of Crumb Rubber in Bitumen – Summary Report


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July 2025

# Version Control

| Report version no. | Date | Released to client by | Nature of revision |
|---|---|---|---|
| 1 | 13/08/2025 | Jaimi Harrison | Final for publication |

# Executive Summary

This research project investigated the changes in conventional binder properties and binder chemical and physical properties that occur during crumb rubber digestion at different blending times and temperatures when varying the size and type of crumb rubber. Four different sources of crumb rubber were used (truck tyre, car tyre, conveyor belt and mining tyre) each at 2 different gradings (Size 16 and Size 30 as per Austroads ATS 3110:2023). Main Roads Western Australia (MRWA) Specification 511:2025 provides a single standard grading envelope. All crumb rubbers used were within the specified limits for MRWA Specification 511:2025 and Austroads ATS 3110:2023. Crumb rubber-modified binders (CRMBs) were produced at 2 different blending temperatures (165 and 190 °C) and 6 different blending times (1, 2, 4, 11, 24 and 36 hours). The constituent materials, CRMBs as well as the liquid phase and extracted crumb rubber particles following digestion were assessed.

Apart from minor exceptions, all CRMBs incorporating truck tyre-, car tyre-- and conveyor belt-derived crumb rubber were within the specified limits of MRWA Specification 511:2025. These findings provide confidence that CRMBs that derive from these 3 different rubber sources can be used interchangeably in MRWA practice. Mining tyre-derived CRMBs did not all meet specification limits. The characterisation and performance test results of these CRMBs were often markedly different to the other 3 rubber sources. It is, therefore, recommended that additional work be undertaken using a more mature production stream before mining tyre-derived crumb rubber is considered a direct alternative to the other sources for MRWA practice.

## Acknowledgements

The authors would like to acknowledge the technical support of the ARRB laboratory team, particularly Shannon Malone, Elizabeth Woodall and William Song, who prepared and tested the crumb rubber-modified binders for this project. In addition, they would like to acknowledge the technical and scientific contributions of Dr Alex Duan and Dr Yukie O'Bryan from the TrACEES platform at the University of Melbourne who performed a series of analytical experiments. Lastly, the authors would like to thank all suppliers who provided materials for this research.



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# 1 Introduction

## 1.1 Background

In August 2019, the Council of Australian Governments established a timetable to ban the export of waste plastic, paper, glass and tyres to build Australia's capacity for generating high value recycled commodities (Department of Climate Change Energy the Environment and Water 2021). In response, Western Australia's Waste Authority (2023) set targets to increase material recovery to 70% by 2025 and 75% by 2030 in their *Action Plan 2022–2023: Waste Avoidance and Resource Recovery Strategy 2030*.

Crumb rubber from end-of-life tyres has been used to modify bitumen for use in spray sealing applications in Western Australia (WA) for approximately 40 years. In recent years, crumb rubber-modified binders (CRMBs) have also been used in asphalt. Traditionally, CRMBs have been produced using truck tyre–derived crumb rubber (Airey et al. 2003).

It is expected that crumb rubber from other sources have similar composition and, so, warrant investigation. If crumb rubber derived from other sources can be used in CRMBs, it will reduce the amounts of these materials going to landfill. The research summarised in this report is, thus, critical for understanding a potential end market for the use of crumb rubber derived from car tyres, conveyor belts and mining tyres.

## 1.2 Project Scope and Objectives

This research project investigated the impacts of crumb rubber type and size as well as digestion temperature and time on the digestion of crumb rubber by bitumen and, subsequently, the resultant CRMB properties. Crumb rubber from truck tyres, car tyres, conveyor belts and mining tyres were used in this research. Ultimately, the findings of this project will allow a review of current Main Roads Western Australia (MRWA) specifications to make optimum use of crumb rubbers.

# 2 Literature Review

## 2.1 Crumb Rubber Composition and Manufacture

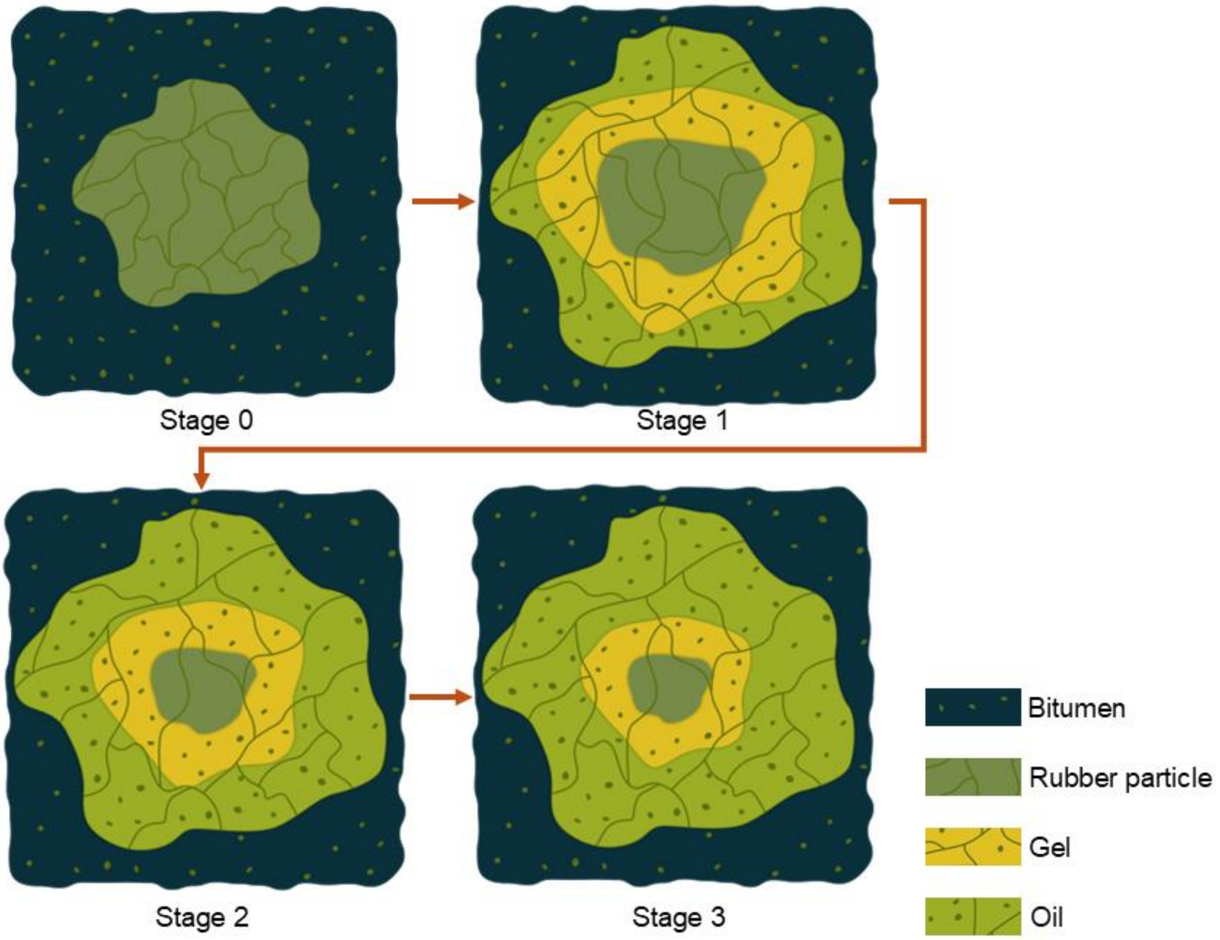
Tyre and conveyor belt rubber products are typically composed of natural and synthetic rubber, steel, fabric/textiles, and chemical additives. The ratio of these different components varies depending on the rubber application, as different ratios produce different characteristics (Jain 2016). The manufacturing process used to produce crumb rubber influences its morphology and, as a result, its physical properties (Khalili et al. 2019). Crumb rubber production typically involves shredding, followed by ambient or cryogenic grinding to produce crumbs with typical sizes ranging from 0.5 to 5 mm. Cryogenic grinding produces relatively smooth and spherical crumb rubber particles, which have lower surface area when compared to the more irregularly shaped products of ambient grinding (Lo Presti 2013). In Australia, crumb rubber is primarily produced by ambient grinding truck tyres (Harrison et al. 2019).

## 2.2 Crumb Rubber Digestion in Bitumen

Crumb rubber can be added to bituminous pavements either via the dry or the wet method. In the dry method, crumb rubber is blended with other aggregate components during the production of an asphalt mix. The wet method involves pre-blending the crumb rubber with bitumen to produce a CRMB. The CRMB is then used in road construction (Austroads 2014). Wet method blends typically include 15–20 wt.% crumb rubber when manufactured (Picado-Santos et al. 2020), whereas the dry method typically involves adding 1–3 wt.% crumb rubber to an asphalt mix. Crumb rubber has been added by the dry and wet processes to gap graded asphalt and open graded asphalt mixes (Austroads 2021). Australia predominantly adopts the wet method. The incorporation of crumb rubber in bitumen via the wet method has been linked to improved rutting resistance and resistance to thermal cracking (Bahia & Davies 1994).

When crumb rubber is added to hot bitumen via the wet method, the crumb rubber particles absorb the light fractions of the bitumen and swell up to 5 times their original size (Picado-Santos et al. 2020). This absorption has also been supported by the measured decrease of aromatics in the base bitumen following crumb rubber digestion. However, care needs to be taken when interpreting this decrease of aromatics as the same phenomenon may also be a manifestation of bitumen aging (Ould-Henia & Dumont 2008). The amount of swelling that occurs when crumb rubber is added to bitumen depends on the blending conditions and the presence of other added components (Picado-Santos et al. 2020). The process of crumb rubber swelling is schematically illustrated in Figure 2.1.

**Figure 2.1: Schematic illustration of crumb rubber swelling due to the absorption of the bitumen's light components**

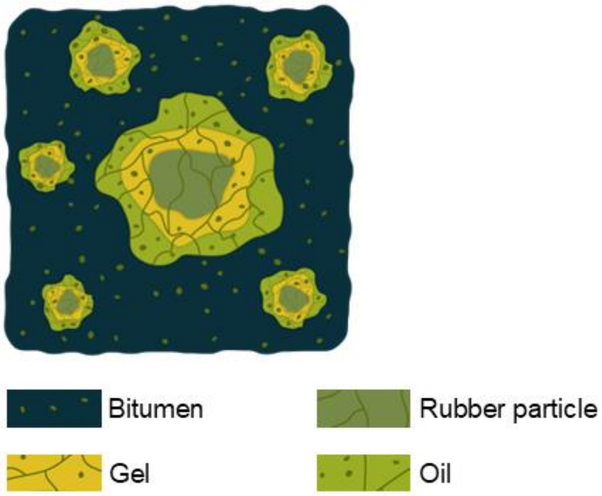



Source: Adapted and recreated from Southern African Bitumen Association (2019).

The addition of crumb rubber particles to bitumen causes changes to binder properties, which can lead to performance improvements to the asphalt manufactured using the CRMB. Asphalt containing CRMBs typically shows improved resistance to permanent deformation and fatigue at intermediate temperatures compared to asphalt produced using unmodified bitumen (Picado-Santos et al. 2020).

Upon further blending at elevated temperatures, the crumb rubber particles begin to dissolve into the bitumen and reduce in size. The dissolution of the crumb rubber causes a reduction in high temperature rheological properties (Billiter et al. 1997). This would be expected to reduce the resistance of the CRMB-containing asphalt to permanent deformation (Choi & Urquhart 2019). Crumb rubber degradation, which is defined by the scission of the rubber's chemical bonds, may also occur under severe interaction conditions, such as very high temperatures (Wang et al. 2021). The degradation and complete dissolution of crumb rubber particles is schematically illustrated in Figure 2.2.

**Figure 2.2: Schematic illustration of crumb rubber degradation due to prolonged exposure to bitumen at elevated temperature**




Source: Adapted and recreated from Wang et al. (2021).

The digestion behaviour of crumb rubber in bitumen depends on a variety of factors, including the rubber type, content, morphology and gradation, the base bitumen, blending temperature and digestion time. In WA, Stage 1 as per Figure 2.1 is targeted for the majority of crumb rubber particles in CRMBs for use in asphalt.

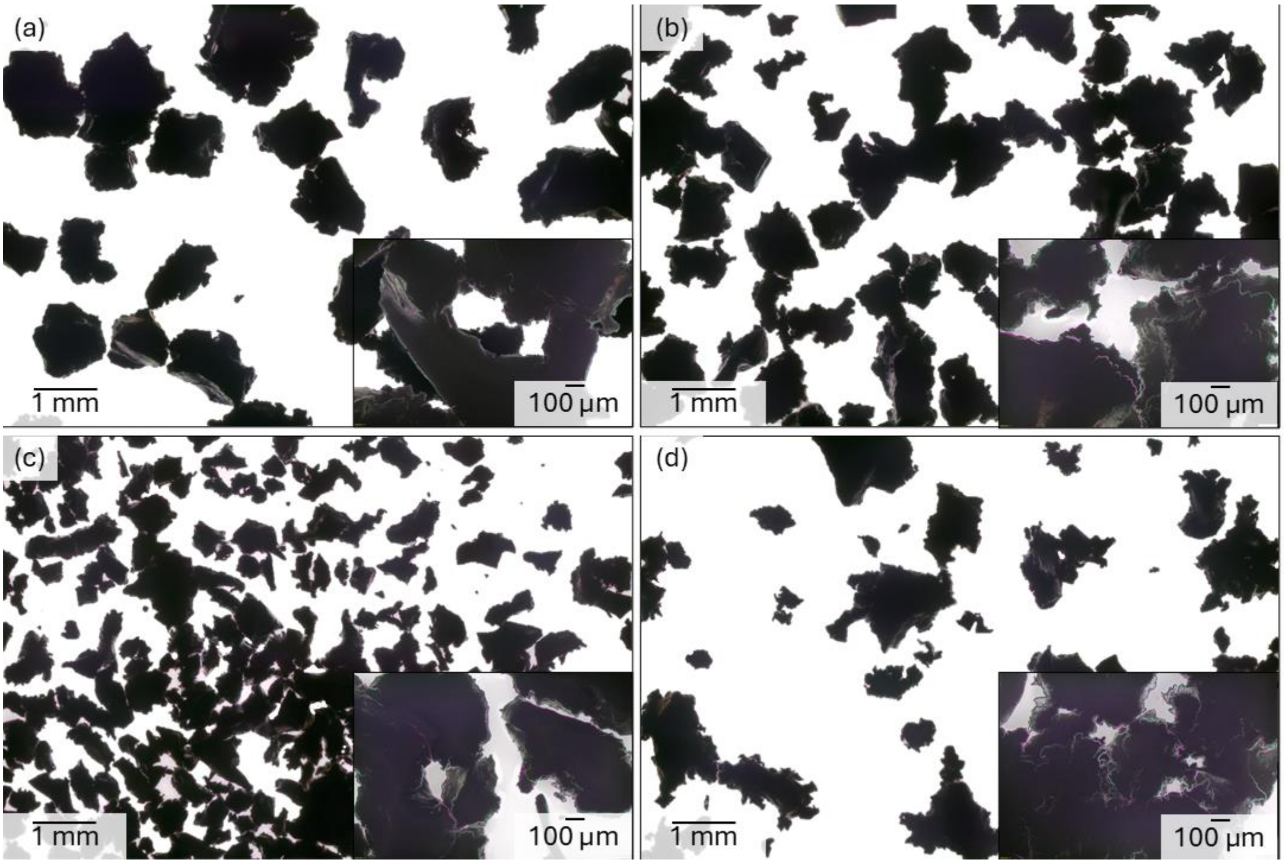
# 3 Materials and Experimental Methods

## 3.1 Materials

This project investigated the digestion potential of different types and sizes of crumb rubber in bitumen. A Class 170 (C170) bitumen, henceforth referred to as C170 and which is commonly used as a base for CRMBs in Australia, was sourced from WA.

Four different types of crumb rubber were supplied from 4 different end-of-life rubber sources, namely truck tyres (TR), car tyres (CT), conveyor belts (CB) and mining tyres (MT). Figure 3.1 shows optical microscope images of the different as-received crumb rubbers.

**Figure 3.1: Optical microscope images of the as-received crumb rubbers: (a) truck tyre–derived crumb rubber, (b) car tyre–derived crumb rubber, (c) conveyor belt–derived crumb rubber, and (d) mining tyre–derived crumb rubber**




All 4 were graded to meet Size 16 (S16) (coarse grading) and Size 30 (S30) (standard grading) requirements of MRWA Specification 511:2025 and Austroads ATS 3110:2023, as shown in Table 3.1.

Table 3.1: Size 30 and size 16 grading envelopes

| Particle size distribution | MRWA Specification 511:2025 (Size 30) | ATS 3110:2023 (Size 16) |
|---|---|---|
| Passing 2.36 mm | 100 | 100 |
| Passing 1.18 mm | 100 | 80 min. |
| Passing 0.6 mm | 60–100 | 10 max. |
| Passing 0.3 mm | 0–22 | – |
| Passing 0.15 mm | – | – |
| Passing 0.075 mm | 0–2 | – |

## 3.2 Sample Preparation

CRMBs of 3,100 g were prepared at the laboratory. The CRMBs comprised approximately 2,542 g of C170 accounting for 82 wt.% of the total blend mass and placed in a 4 L tin. The remaining 18 wt.% (558 g) of the blend comprised the crumb rubber. Blends using all 4 types of crumb rubber of both sizes were blended for 1, 2, 4, 11, 24 and 36 hours at 2 target temperatures of 165 and 190 °C. Sample naming conventions follow the parameters investigated as TypeSize_BlendingTemperature_BlendingTime. For example, a sample blended at 165 °C for 2 hours using size 30 truck tyres would be referred to as TR30_165C_2h.

The following procedure was followed for the first hour of blending:

- The 4 L tin (covered but not sealed) with the C170 was placed in a fan-forced oven at 150 °C until the bitumen was free flowing.
- The open 4 L tin was then placed in a heating block with a blanket of $CO_2$ at a flow rate of 5 L/min to prevent oxidation.
- The C170 was conditioned in the heating block until the C170 was at the target blending temperature (165 or 190 °C). A thermocouple was used to measure the temperature of the bitumen, which was controlled to the target temperature ± 10 °C throughout the blending process.
- Once the C170 was at temperature, the crumb rubber particles were gradually (approximately within a 5-minute period) added.
- A low shear mixer was used for blending at 1,300 rpm.

Following the first hour of blending, the CRMBs were transferred to a different oven for the remainder of the blending. This oven was also equipped with a low shear mixer and an inlet for $CO_2$ to mitigate against oxidation. These were both set at the same parameters as for the first hour of blending at 1,300 rpm and 5 L/min, respectively. Unmodified C170 was also blended following the same protocol to understand any thermo-oxidative ageing impacts.

Soxhlet extraction was used to extract the crumb rubber from the binders after digestion following Austroads AGPT-T142:2020 *Rubber Content of Crumb Rubber Modified Bitumen: Soxhlet Method*. Approximately 10 g of binder was placed in a thimble. Prior to the placement of each specimen in the Soxhlet extractor, they were immersed in a beaker of toluene for 48 hours, as noted in test method WA 238.1:2022 *Rubber Content of Bitumen Rubber Blends*.

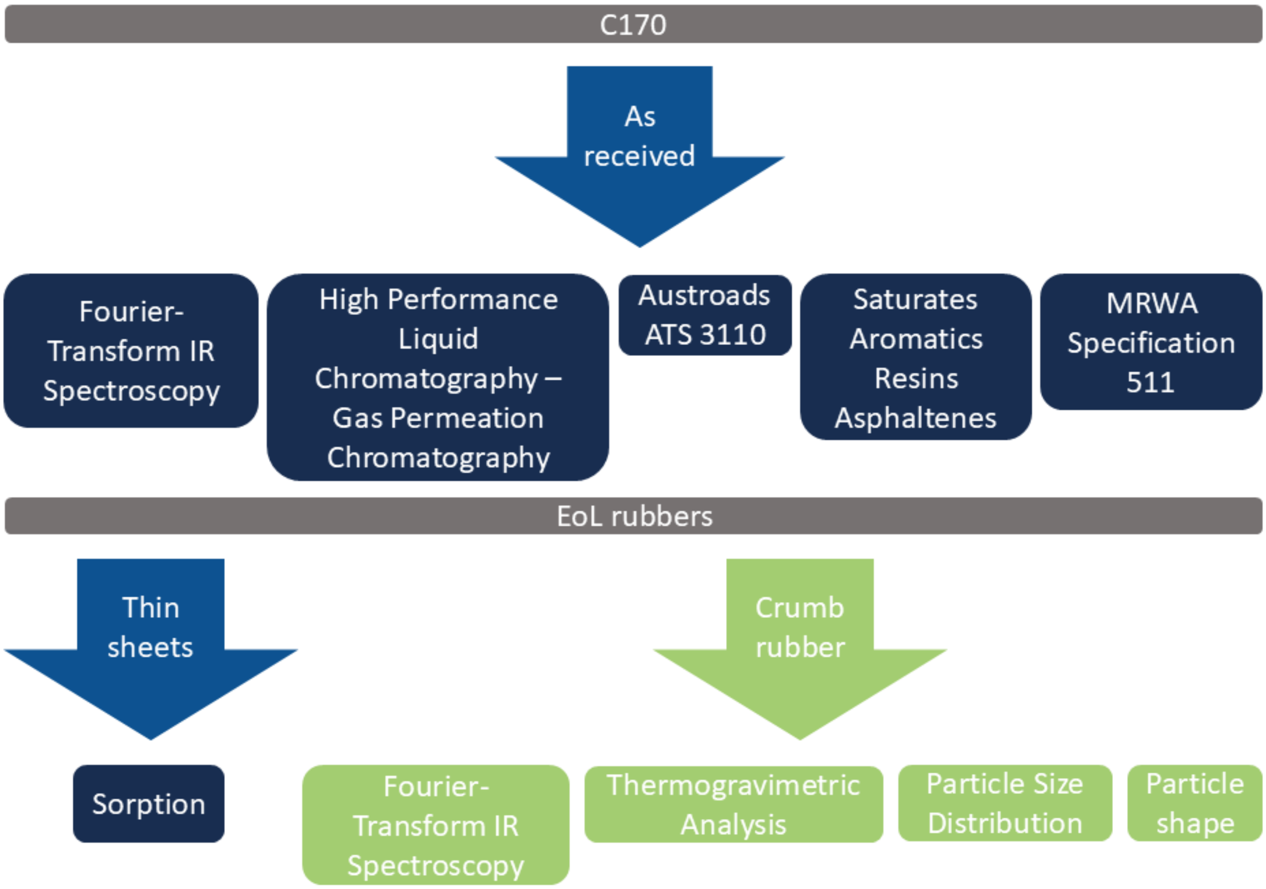
To analyse the liquid phase of the binders, 2–3 g of the CRMBs were put into silicone trays on a 75 µm mesh, and a collector tray was placed underneath. To avoid further oxidation of the bitumen, the extraction and collection trays were wrapped in an oven bag. The assembly was then placed in an oven at 165 °C for 30 minutes. It was then removed and cooled by placing it in a refrigerator for rapid cooling.

## 3.3 Experimental Methods

Partially digested CRMBs are not a homogeneous material; rather, they are a composite of a liquid and a solid phase. The liquid phase comprises the bitumen and any dissolved or degraded (< 75 µm) portion of the crumb rubber, and the solid phase comprises the rubber particle and any bitumen light components that it has absorbed. It is suspected that, due to the nature of the tests conducted, the results presented in Sections 4.2 to 4.4 are not influenced by the composite or each of its constituent phases equally. Therefore, the analytical experimental methods used to understand the impact of crumb rubber type and size and blending parameters on binder handling and performance have focused on understanding both the liquid and solid phase of the produced CRMBs.

The materials used in this research project were assessed following various methods and at different stages in the process. Figure 3.2 summarises the tests performed on the starting materials described in Section 3.1.

**Figure 3.2: Summary of properties assessed for the constituent materials prior to blending**

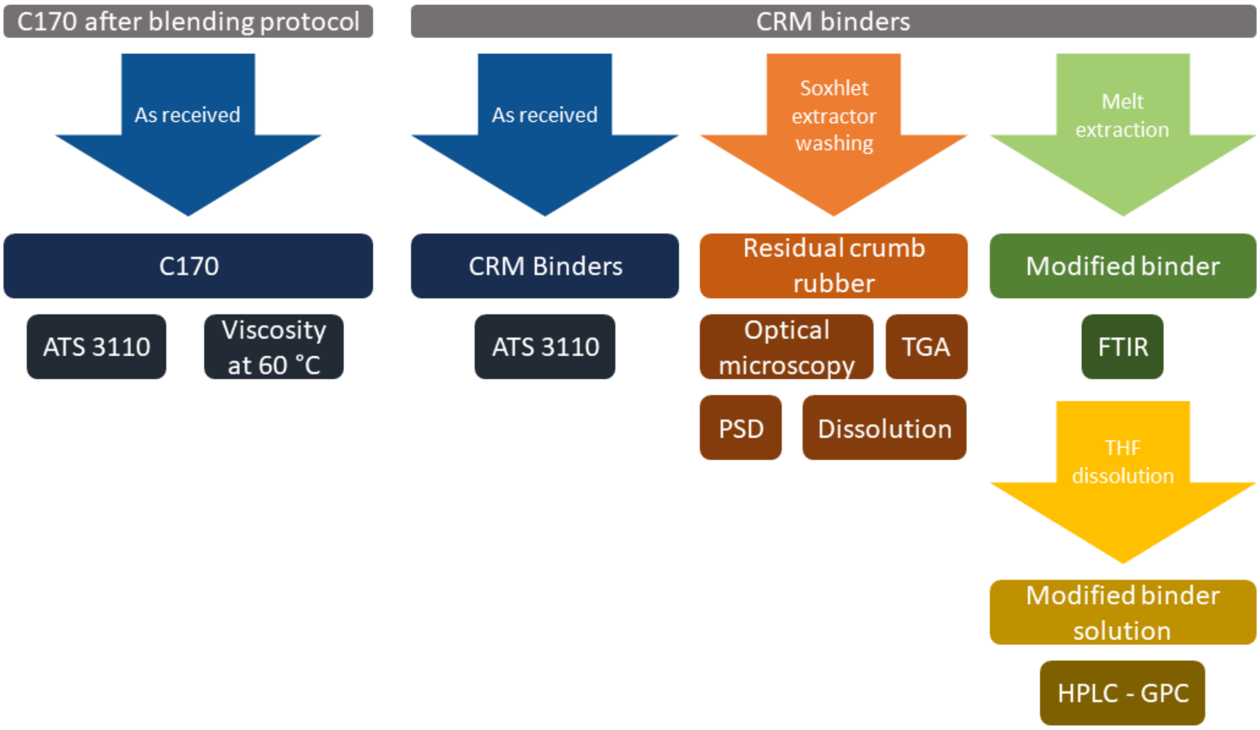



Notes:

- MRWA Specification 511 tests performed: Viscosity at 60 °C, Viscosity at 135 °C, Penetration at 25 °C, Percentage increase in viscosity at 60 °C after rolling thin film oven treatment, Viscosity at 175 °C, Resilience at 25 °C.
- Austroads ATS 3110 tests performed: Viscosity at 165 °C, Torsional recovery at 25 °C, Softening point, Consistency at 6% at 60 °C, Loss on heating.

Figure 3.3 summarises the testing protocol for the materials following the blending process described in Section 3.2. These materials were assessed in their as-received state following blending as well as following extraction of the particles and liquid phase.

**Figure 3.3: Summary of properties assessed for the materials having undergone blending**




Notes:

- Austroads ATS 3110 tests performed for the blended C170: Viscosity at 165 °C, Viscosity at 175 °C, Softening point.
- Austroads ATS 3110 and MRWA Specification 511 tests performed for the CRMBs: Viscosity at 165 °C, Viscosity at 175 °C, Torsional recovery at 25 °C, Softening point, Consistency at 6% at 60 °C, Stress ratio at 10 °C, Resilience at 25 °C, Loss on heating, Compressive limit at 70 °C.
- TGA (Thermogravimetric analysis), PSD (particle size distribution), FTIR (Fourier-transform infrared spectroscopy), HPLC-GPC (high-performance liquid chromatography – gas permeation chromatography).

# 4 Results and Discussion

## 4.1 Crumb Rubber Digestion

### 4.1.1 Rubber Swelling

Figure 4.1 presents the sorption curves for the 4 rubbers at the 2 temperatures investigated as mass increase calculated using Equation 1 plotted against $t^{1/2}/d$ (where $t$ is time in seconds and $d$ is the thickness of the rubber sample at each time $t$). A linear fit was applied to the data points using basic fitting. All fits were found to have an $R^2 > 0.95$ and, as such, were considered acceptable. From the gradient of these linear fits, it is evident that rubber swelling was, in all cases, more rapid when the bitumen was at 190 °C when compared to 165 °C. The rate of swelling at both temperatures was greatest for truck tyre followed by conveyor belt, then mining tyre was found to follow with car tyre having the lowest rate of swelling.

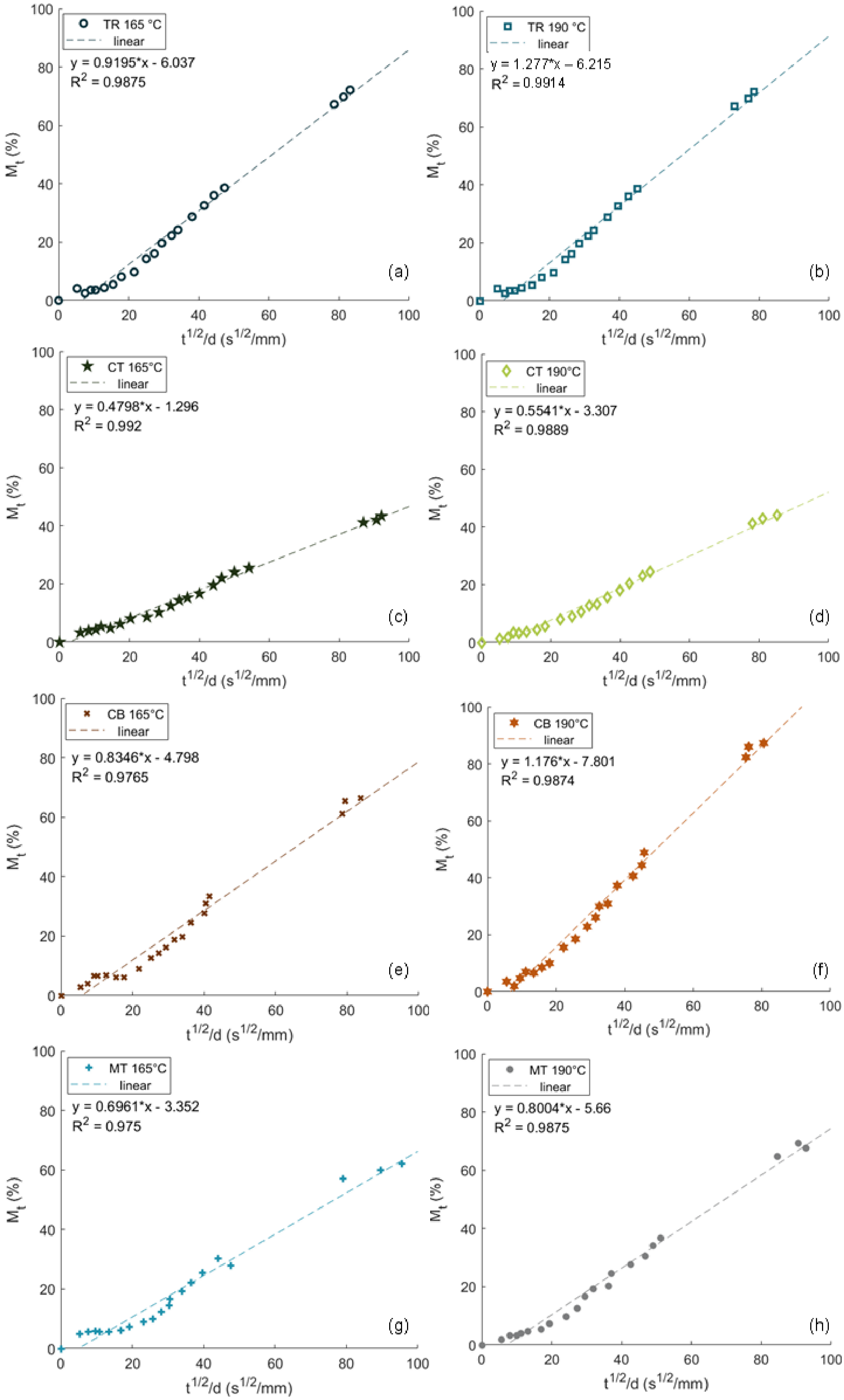
$$M_t = \left[\frac{W_t - W_0}{W_0}\right] \times 100 \qquad 1$$

where:

| | | |
|---|---|---|
| $W_t$ | = | the weight of the rubber at time t |
| $W_0$ | = | the initial weight of the rubber |
| $M_t$ | = | percentage mass increase at time t |

Source: Fazli and Ridrigue (2020).

**Figure 4.1: Mass increase as a function of $t^{1/2}/d$; (a) TR_165C, (b) TR_190C, (c) CT_165C, (d) CT_190C, (e) CB_165C, (f) CB_190C, (g) MT_165C and (h) MT_190C**



### 4.1.2 High-Performance Liquid Chromatography – Gas Permeation Chromatography

Huang et al. (2017) explained that the dissolved polymer content in the samples can be calculated through gel permeation chromatography as the area under the retention time vs intensity curves for molecular weight above 19,000 g/mol, that of asphaltenes for molecular weight between 19,000 and 3,000 g/mol, while the remaining, below 3,000 g/mol, is expected to be maltenes. To find the retention time for which the molecular weight is 19,000 and 3,000 g/mol, the retention time was calculated based on PS standards using the power fit equation in Figure 4.2.

**Figure 4.2: PS standards with power law data fit**

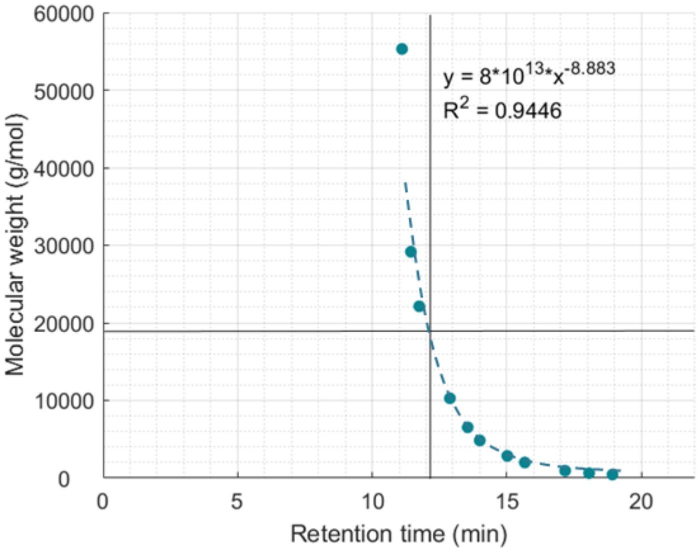



Figure 4.3 shows the concentrations of each of the components (polymer, apparent asphaltenes, and maltenes) in the melt extracted binders of all CRMBs.

Figure 4.3 (a) shows an increase in the polymer content of truck tyre-derived CRMBs with blending time from 1 to 11 and 36 hours. In all cases, the polymer concentration increase was more pronounced when blending was extended from 11 to 36 hours. Blending temperature does not appear to have notably affected the concentration of dissolved polymer.

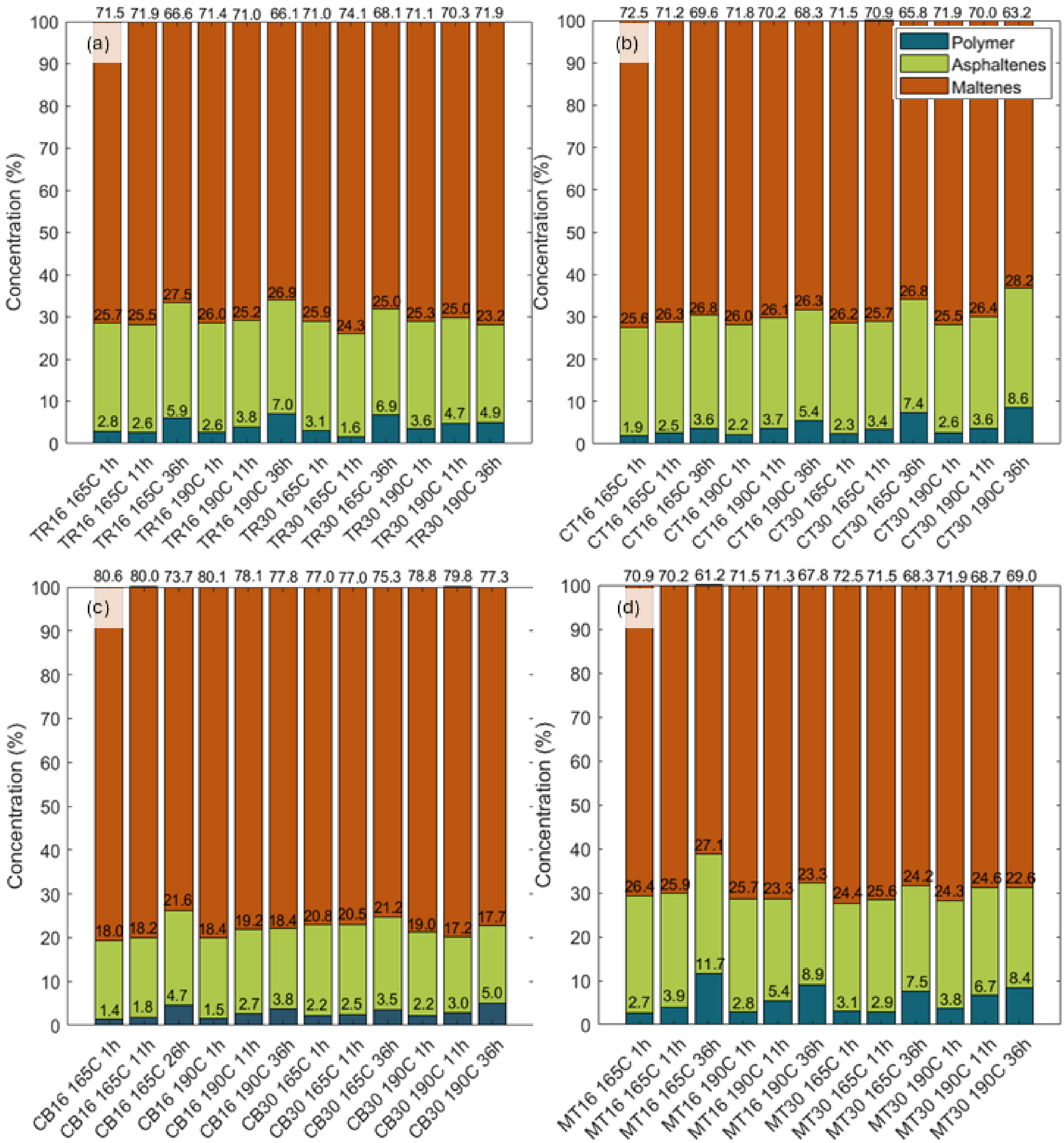
Figure 4.3 (b) shows the measured distribution of constituents in the melt extracted car tyre-derived CRMBs, where the polymer content increased with time for all samples. The polymer content was also found to be comparatively greater for size 30 CRMBs and for CRMBs blended at 190 °C.

The concentration of the different binder constituents for conveyor belt-derived CRMBs are presented in Figure 4.3 (c). As for car tyre-derived CRMBs, the concentration of polymer in the binder was found to increase with blending time. Temperature did not appear to affect the rate of polymer dissolution. This increase in polymer concentration was also more pronounced when blending was extended from 11 to 36 hours, as observed for truck tyre-derived CRMBs.

Figure 4.3 (d) presents the measured concentration of constituents of the liquid phase for mining tyre-derived CRMBs. The overall trends observed are in line with all other CRMBs in this research, whereby the polymer content increased with an increase in blending time and more so between 11 and 36 hours of blending.

Overall, the greatest concentration for dissolved polymer was measured in the liquid phase of mining tyre-derived CRMBs followed by truck tyre-, car tyre- and, lastly, conveyor belt-derived CRMBs.

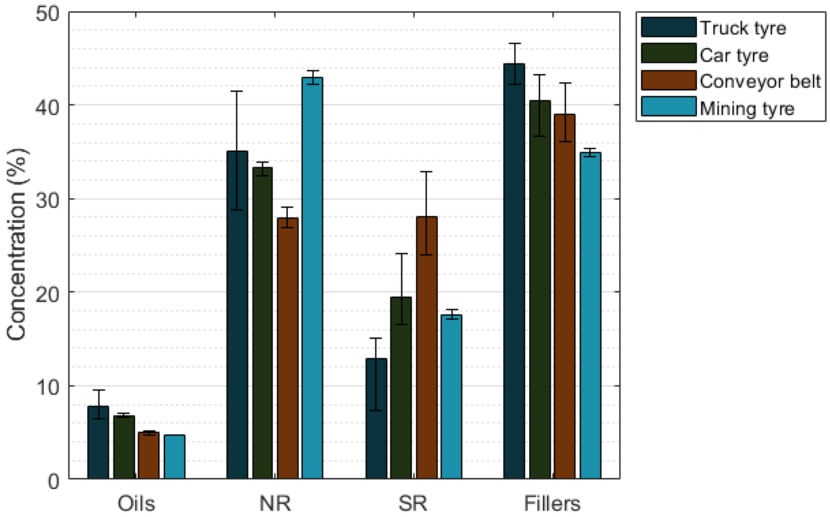
**Figure 4.3: Concentration of components based on their molecular weight for (a) truck tyre–, (b) car tyre– , (c) conveyor belt– and (d) mining tyre–derived crumb rubber-modified binders**




### 4.1.3 Thermogravimetric Analysis

Thermogravimetric analysis was used to measure the concentration of each of the rubber constituents. Mining tyre-derived rubber was found to have the greatest total rubber content at approximately 60%, followed by conveyor belt-derived rubber at approximately 56%, then car tyre-derived rubber at approximately 53% and, lastly, truck tyre-derived rubber was found to have the lowest concentration of total rubber at approximately 48%. Truck tyre-derived rubber was, however, measured to have the lowest synthetic rubber/natural rubber ratio at 0.37, followed by mining tyre-derived rubber at 0.41, then by car tyre-derived rubber at 0.58 and, lastly, by conveyor belt-derived rubber at 1.00. The concentration of each of the constituents for the 4 different types of rubber used in this research with standard deviation is presented in Figure 4.4.

Figure 4.4: Concentration of moisture and light oils, natural rubber, synthetic rubber and other polymers and carbon black and fillers for all as-received crumb rubbers

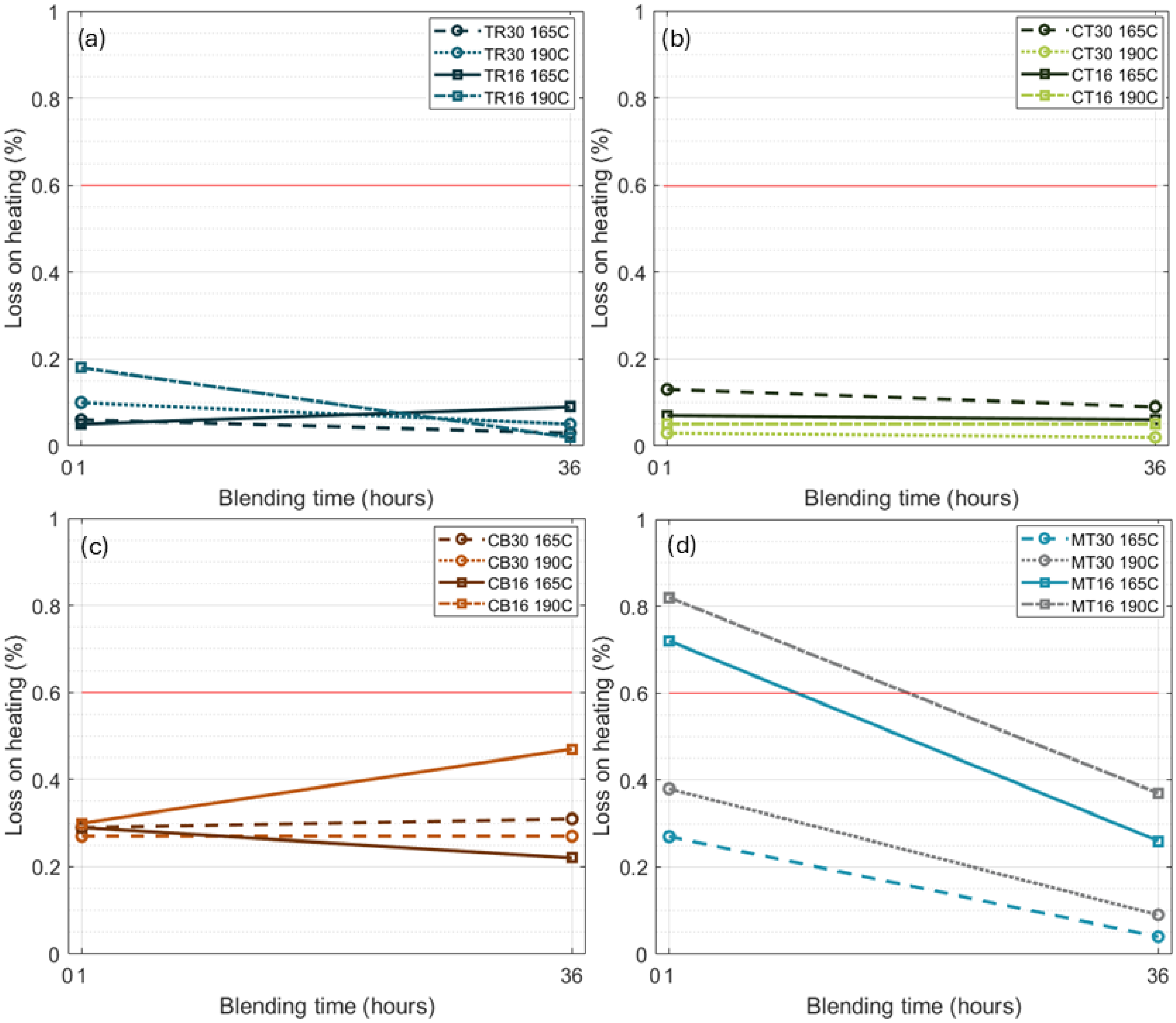



## 4.2 Handling Properties of Crumb Rubber-Modified Binders

### 4.2.1 Loss on Heating

Figure 4.5 graphically presents the results of loss on heating. All binders were found to be below the allowable limit of 0.6% by Austroads ATS 3110:2023 except MT16_165C_1h and MT16_190C_1h, which exhibited an average loss on heating of 0.72 and 0.82%, respectively.

Figure 4.5: Blending time versus loss on heating for (a) truck tyre–derived crumb rubber-modified binders, (b) car tyre–derived crumb rubber-modified binders, (c) conveyor belt–derived crumb rubber -modified binders and (d) mining tyre–derived crumb rubber-modified binders




Note: Red line denotes Austroads ATS 3110:2023 maximum requirement.

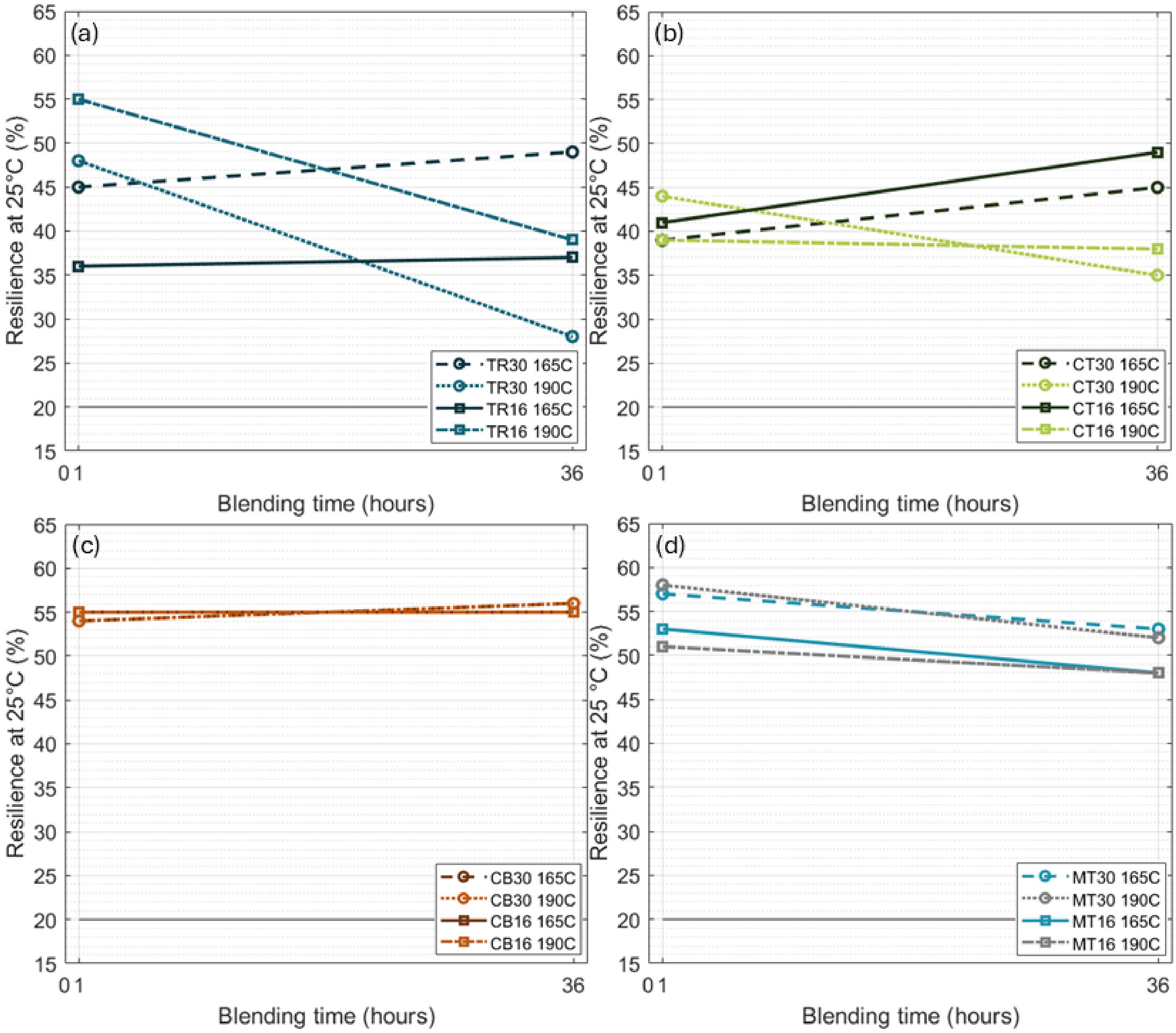
In this research, both loss on heating and viscosity at 165 and 175 °C were assessed to determine the handling properties of the produced CRMBs. Mining tyre-derived CRMBs were found to behave differently for both these properties, whereby their viscosity at 165 and 175 °C was found to be comparatively greater. However, a demonstration construction trial by Austroads (2024) focusing only on manufacturing and placing asphalt reported no need to alter work practices for any of the truck tyre-, car tyre- and mining tyre-derived CRMBs. This could either mean that the test methods used in this research are not a true representation of the handling behaviour of the binders or highlight the differences in crumb rubber source, composition and its impacts on CRMB behaviour.

## 4.3 Elasticity

### 4.3.1 Resilience at 25 °C

Figure 4.6 graphically presents the resilience at 25 °C results. All binders were found to meet the minimum requirement of 20% as per MRWA Specification 511:2025.

Figure 4.6: Blending time versus resilience at 25 °C for (a) truck tyre–derived crumb rubber-modified binders, (b) car tyre–derived crumb rubber-modified binders, (c) conveyor belt–derived crumb rubber-modified binders and (d) mining tyre–derived crumb rubber-modified binders



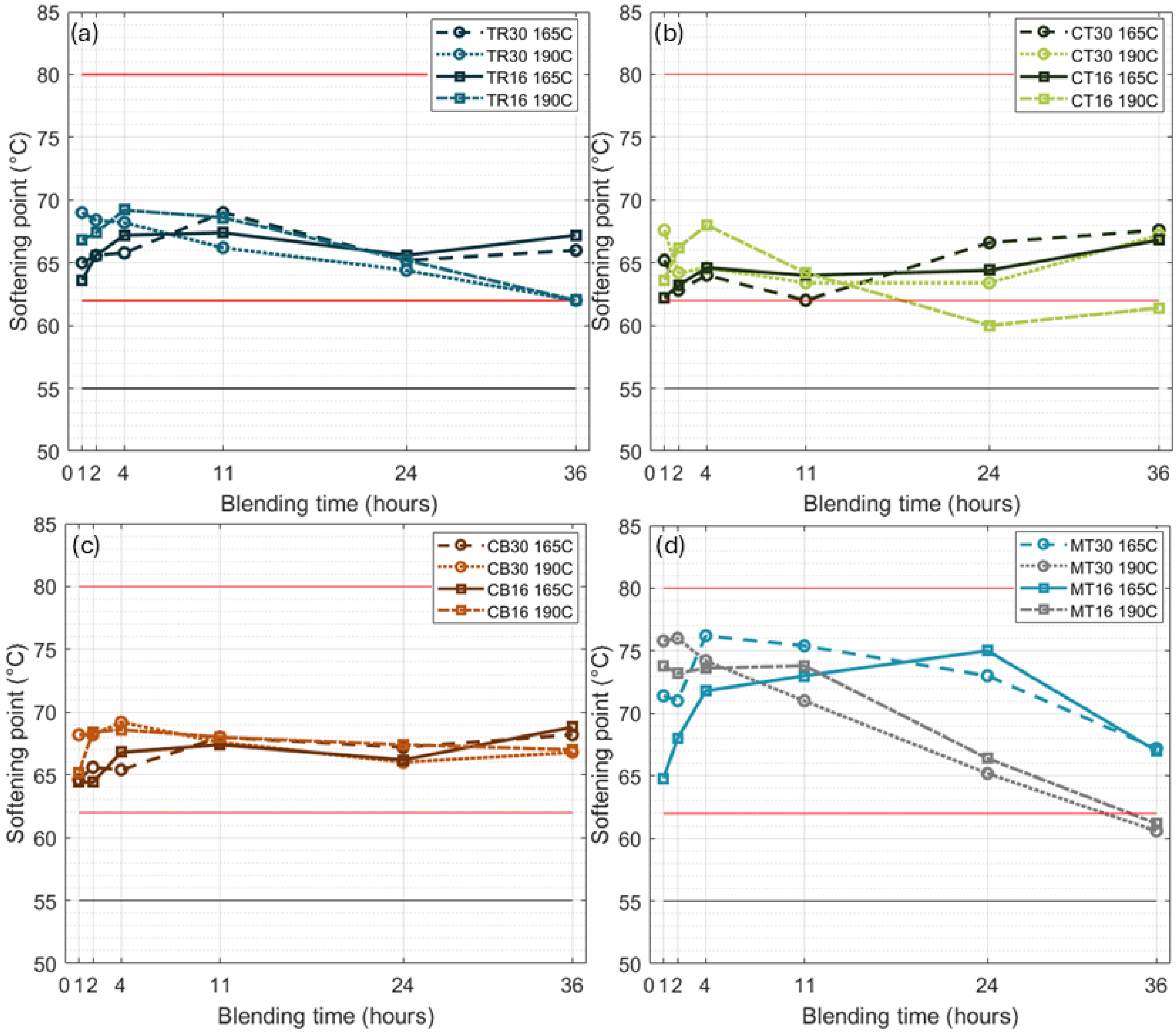

Note: Black line denotes MRWA Specification 511:2025 minimum requirement.

## 4.4 Rutting Resistance

### 4.4.1 Softening Point

Figure 4.7 graphically presents the softening point results against the limits specified by MRWA Specification 511:2025 and Austroads ATS 3110:2023. All CRMBs tested were found to have softening point values above the minimum requirement specified by MRWA Specification 511:2025.

Figure 4.7: **Blending time versus softening point for (a) truck tyre–derived crumb rubber-modified binders, (b) car tyre–derived crumb rubber-modified binders, (c) conveyor belt–derived crumb rubber-modified binders and (d) mining tyre–derived crumb rubber-modified binders**



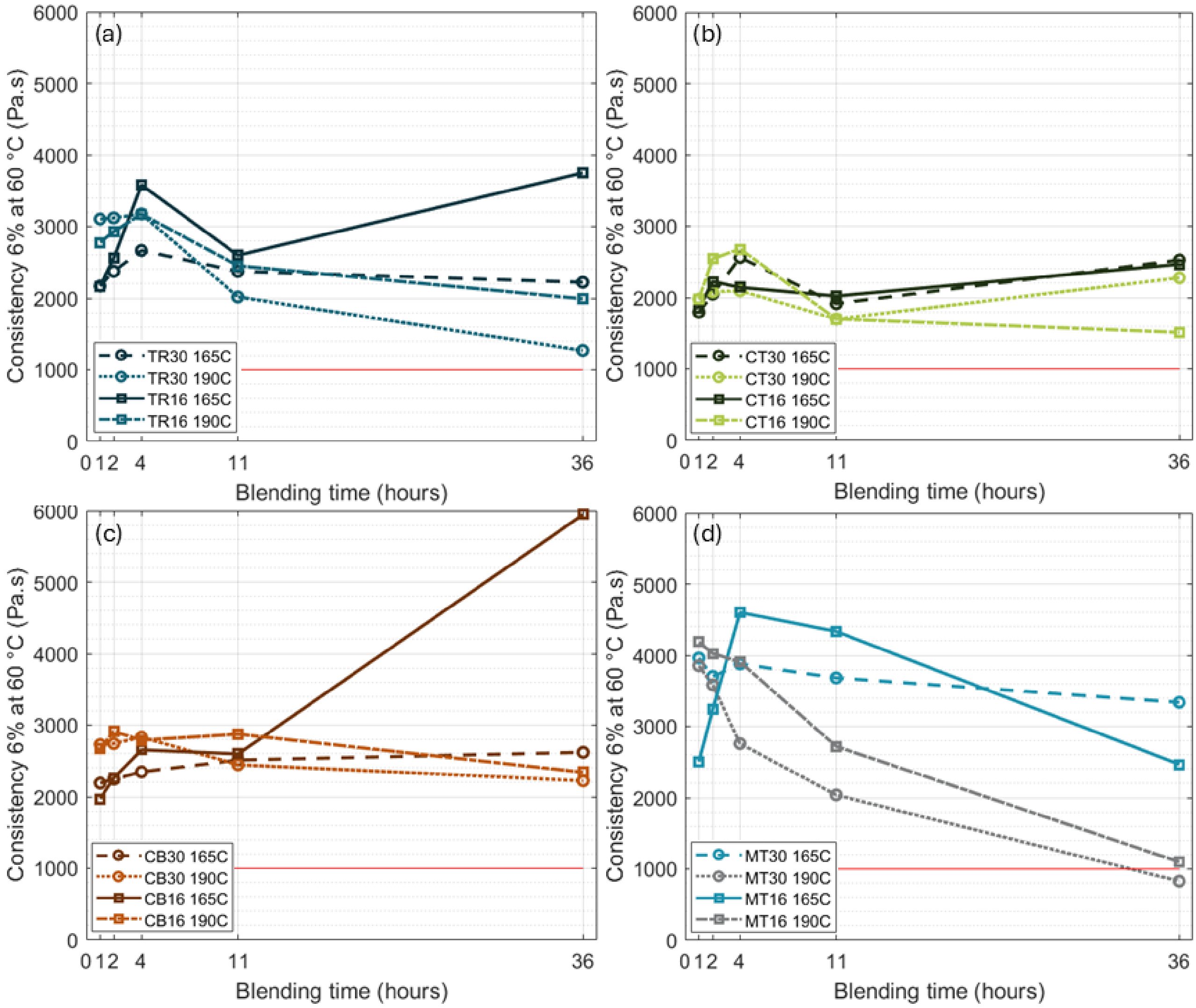

Note: Red line denotes Austroads ATS 3110:2023 limits and black line denotes minimum requirement according to MRWA Specification 511:2025.

## 4.4.2 Consistency 6% at 60 °C

Figure 4.8 graphically presents the consistency 6% at 60 °C results for all binders against the lower limit specified by Austroads ATS 3110:2023 at 1,000 Pa.s. MRWA Specification 511:2025 requires for consistency 6% at 60 °C results to be reported but does not specify any limits.

All truck tyre-derived CRMBs were found to be above the specified by Austroads ATS 3110:2023 lower limit. The results presented a 2,479 Pa.s variation depending on the investigated parameters. Car tyre-derived CRMBs were also found to meet the requirements of ATS 3110:2023. The results were found to vary by up to 1,168 Pa.s depending on the parameters. Conveyor belt-derived CRMBs were found to be above the minimum requirement of Austroads ATS 3110:2023 as well. The results presented a relatively greater variation of 3,973 Pa.s, which can be attributed to the notably greater consistency 6% at 60 °C of sample CB16_165C_36h. Mining tyre-derived CRMBs were also found to meet the minimum requirements of Austroads ATS 3110:2023, except for sample MT30_190C_36h. The consistency 6% at 60 °C of mining tyre-derived CRMBs presented a notable variation of 3,773 Pa.s depending on the investigated parameters.

**Figure 4.8: Blending time versus consistency 6% at 60 °C for (a) truck tyre–derived crumb rubber-modified binders, (b) car tyre–derived crumb rubber-modified binders, (c) conveyor belt–derived crumb rubber-modified binders and (d) mining tyre–derived crumb rubber-modified binders**




Note: Red line denotes Austroads ATS 3110:2023 minimum requirement.

# 5 Conclusion

The digestion of crumb rubber in bitumen at elevated temperatures facilitates the absorption of the light fractions of the bitumen by the crumb rubber resulting in its swelling. Over time, the crumb rubber dissolves into the bitumen, consequently reducing in size. The digestion of crumb rubber in bitumen during blending is a complex process as both crumb rubber and bitumen have different chemical compositions depending on their source and processing methods.

This research investigated the changes in conventional binder properties and binder chemical and physical properties that occur during crumb rubber digestion at different blending times and temperatures when varying the size and type of crumb rubber. Four different sources of crumb rubber were used each at 2 different gradings. Crumb rubber binders were produced at 2 different blending temperatures and 6 different blending times using 18 wt.% crumb rubber. The constituent materials, CRMBs as well as the liquid phase of the binder and extracted crumb rubber particles following digestion were assessed through a series of test methods.

This research found that:

- Analytical methods, including rubber swelling, thermogravimetric analysis and high-performance liquid chromatography – gas permeation chromatography, were found to be useful in assessing the digestion behaviour of the different rubber types under the different conditions investigated.
- It was not possible to link the findings of the analytical methods with the results of the handling and performance properties of the resultant CRMBs. This was likely due to the physicochemical complexity of the CRMBs as a result of:
  - the diversity of crumb rubber particle size meant that particles were at different stages of digestion
  - the impacts of thermo-oxidative ageing
  - the suitability of binder performance test methods used to CRMBs.
- The performance of truck tyre-, car tyre- and conveyor belt-derived CRMBs was found to be comparable and they may be used interchangeably in WA practice.
- The test results relating to handling properties, as well as observations by handling mining tyre-derived CRMBs in the laboratory, suggested that they may introduce challenges during production. In addition, they were found to not comply with MRWA Specification 511:2025 under all conditions.
- Mining tyre-derived crumb rubber may need to be considered more carefully, however, more research is required as their recycling is still in its infancy.

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