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Summary

Crumb rubber has been used in Western Australia for almost 40 years to modify bitumen used in spray sealing applications. In recent years, crumb rubber–modified binders (CRMBs) have also been used in asphalt. Regardless, a detailed understanding of how the crumb rubber and bitumen interact over time at different mixing temperatures and how the source and size of the crumb rubber affect the crumb rubber binder properties is yet to be gained.

Of particular interest in this research was how the variation of crumb rubber type and size and blending temperature and time affect digestion and, consequently, the characteristics and performance of CRMBs and so their compliance with current specifications.

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 (TR, CT, CB and MT) each at 2 different gradings (Size 16 (S16) and Size 30 (S30) in accordance with ATS 3110:2023). Crumb rubber binders 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 of the binder and extracted crumb rubber particles following digestion were assessed through a series of test methods.

The digestion behaviour of the different rubber types and crumb rubber sizes under the investigated blending conditions was assessed through a wide range of analytical methods. Of the methods used, high performance liquid chromatography – gel permeation chromatography yielded the best results. Crumb rubber dissolution was found to generally increase with blending time. The swelling of all rubber types was more rapid at the higher blending temperature of 190 °C.

A wide range of binder characterisation methods were also used, according to Austroads and Main Roads Western Australia (MRWA) specifications, to evaluate the performance of the resultant binders. Overall, results were comparable for TR-, CT- and CB-derived CRMBs and for all blending temperatures, blending times and crumb rubber size gradations investigated. Apart from minor exceptions, all CRMBs incorporating TR-, CT- and CB-derived crumb rubber were within the specified limits of both Austroads ATS 3110:2023 and 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.

The results for MT-derived CRMBs were often different and often did not meet the Austroads ATS 3110:2023 and MRWA Specification 511:2025 limits. When compared to the other 3 crumb rubber types, the production of MT-derived CRMBs is still in its infancy. For example, a demonstration construction trial from Austroads focusing only on manufacturing and placing of asphalts reported no need to alter work practices. It is, therefore, recommended that additional work be undertaken, using a more mature production stream, before MT-derived crumb rubber is considered a direct alternative to the other sources for MRWA practice.

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List of Abbreviations and Glossary of Terms

List of Abbreviated Terms

AfPA	Australian Flexible Pavements Association
ANOVA	Analysis of variance
ATR	Attenuated total reflectance
CRMB	Crumb rubber-modified binders
CB	Conveyor belt
CT	Car tyre
DGA	Dense-graded asphalt
DSR	Dynamic shear rheometer
DTG	Derivative thermogravimetry
EoL	End-of-life
FTIR	Fourier-transform infrared
GGA	Gap-graded asphalt
HPLC-GPC	High performance liquid chromatography - gel permeation chromatography
MRWA	Main Roads Western Australia
MT	Mining tyre
NR	Natural rubber
NTRO	National transport research organisation
OGA	Open-graded asphalt
OTR	Off-the-road
PSD	Particle size distribution
PVC	Polyvinyl chloride
rpm	Rotations per minute
RTFO	Rolling thin film oven
S16	Size 16
S30	Size 30
SBR	Styrene-butadiene rubber
SBS	Styrene-butadiene-styrene
SR	Synthetic rubber
TGA	Thermogravimetric analysis
TR	Truck tyre
TSA	Tyre Stewardship Australia
US	United States
WA	Western Australia
WARRIP	Western Australia Road Research and Innovation Program

Glossary of Terms

Crumb rubber digestion	The progressive change of the crumb rubber particle from a resilient particle to a gel and then an oil (Southern African Bitumen Association 2019).
Crumb rubber swelling	A physical diffusion process whereby the volume of the crumb rubber particle expands as a result of absorbing light components of bitumen (Wang, Apostolidis et al. 2021).
Crumb rubber degradation	A chemical process whereby the crosslinked chain network of the rubber particles is compromised as a result of thermal and mechanical energy input (Wang, Apostolidis et al. 2021).
Crumb rubber dissolution	The portion of crumb rubber that passes 75 µm following interaction with bitumen (Ghavibazoo & Abdelrahman 2013).

Contents

1	Introduction	1
1.1	Background	1
1.2	Project Scope and Objectives	2
1.3	Structure of the Report	2
2	Literature Review	3
2.1	Crumb Rubber Composition and Manufacture	3
2.2	Crumb Rubber Digestion in Bitumen	4
2.2.1	Overview of the Digestion Process	5
2.2.2	Effects of Crumb Rubber Content	6
2.2.3	Effects of Crumb Rubber Size, Gradation and Morphology	6
2.2.4	Effects of Crumb Rubber Type	7
2.2.5	Effects of Base Bitumen	8
2.2.6	Effects of Temperature	8
2.2.7	Effects of Digestion Time	9
2.3	Crumb Rubber Specification Requirements	9
2.3.1	International	10
2.3.2	Australia and New Zealand	11
3	Materials and Experimental Methods	13
3.1	Materials	13
3.1.1	Bitumen	13
3.1.2	Crumb Rubbers	13
3.2	Sample Preparation	14
3.2.1	Binder Blending Process	14
3.2.2	Soxhlet Extractor Washing of Crumb Rubber-Modified Binders	18
3.2.3	Manual Washing of Crumb Rubber-Modified Binders	19
3.2.4	Melt Extract of Crumb Rubber-Modified Binders	19
3.3	Experimental Methods	20
3.3.1	SARA by Column Chromatography	22
3.3.2	Viscosity at 60 °C	23
3.3.3	Crumb Rubber Swelling	23
3.3.4	Thermogravimetric Analysis	23
3.3.5	High Performance Liquid Chromatography – Gel Permeation Chromatography	24
3.3.6	Fourier-Transform Infrared Spectroscopy	25
3.3.7	Crumb Rubber Dissolution	25
3.3.8	Particle Size Distribution	26
3.3.9	Optical Microscopy	26

3.3.10	Viscosity at 165 and 175 °C	26
3.3.11	Stress Ratio at 10 °C	26
3.3.12	Torsional Recovery at 25 °C	27
3.3.13	Resilience at 25 °C	27
3.3.14	Softening Point	27
3.3.15	Consistency at 6% at 60 °C	27
3.3.16	Loss on Heating	27
3.3.17	Compressive Limit at 70 °C	27
3.4	Statistical Analysis	27
4	Results	29
4.1	Characterisation of Unmodified Bitumen	29
4.1.1	SARA by Column Chromatography	29
4.1.2	Viscosity at 60 °C	29
4.2	Crumb Rubber Digestion	30
4.2.1	Rubber Swelling	30
4.2.2	Crumb Rubber Dissolution	32
4.2.3	High Performance Liquid Chromatography – Gel Permeation Chromatography	33
4.2.4	Thermogravimetric Analysis	44
4.2.5	Fourier-Transform Infrared Spectroscopy	54
4.2.6	Optical Microscopy	66
4.2.7	Summary of Findings and Discussion	76
4.3	Crumb Rubber-Modified Binder Characteristics	76
4.3.1	Compressive Limit at 70 °C	76
4.3.2	Summary of Findings and Discussion	80
4.4	Handling Properties of Crumb Rubber-Modified Binders	81
4.4.1	Viscosity at 165 and 175 °C	81
4.4.2	Loss on Heating	89
4.4.3	Summary of Findings and Discussion	92
4.5	Elasticity	92
4.5.1	Torsional Recovery at 25 °C	92
4.5.2	Resilience at 25 °C	97
4.5.3	Summary of Findings and Discussion	100
4.6	Rutting Resistance	101
4.6.1	Softening Point	102
4.6.2	Consistency 6% at 60 °C	105
4.6.3	Summary of Findings and Discussion	109
4.7	Fatigue Life	110
4.7.1	Stress Ratio at 10 °C	110
4.7.2	Summary of Findings and Discussion	114

5	Discussion	115
5.1	Binder Oxidation	115
5.2	Effect of Crumb Rubber Type on Digestion	115
5.3	Effect of Crumb Rubber Type on Binder Properties	118
6	Conclusion	120
	References	121
Appendix A	Summary of Samples Assessed per Test	128
Appendix B	Blended Sample Morphology	136
Appendix C	Supplementary Results on Binder Performance Tests	140
Appendix D	Supplementary Results on Analytical Assessment	167
Appendix E	Specification Compliance	178

Tables

Table 2.1:	List of international, Australian and New Zealand standards and specifications reviewed	10
Table 3.1:	Results for measured properties of C170 used and summary of MRWA Specification 511:2025 requirements	13
Table 3.2:	Results for measured properties of crumb rubbers used and summary of MRWA Specification 511:2025 requirements (for S30) and Austroads ATS 3110:2023 requirements (for S16 and S30)	14
Table 3.3:	Samples prepared and naming conventions	17
Table 3.4:	Measured recovery factor for each crumb rubber type and size	18
Table 3.5:	Samples assessed according to AS/NZS 2341.2:2015	23
Table 3.6:	Instrument conditions for HPLC-GPC	25
Table 3.7:	Polystyrene standards	25
Table 3.8:	Correlation scale ranges.....	28
Table 4.1:	SARA fractions of C170 bitumen.....	29
Table 4.2:	Viscosity at 60 °C test results for the base C170 bitumen after different blending times at 165 or 190 °C; results in Pa·s	29
Table 4.3:	Optical microscope images of extracted truck tyre-derived crumb rubber after digestion at 165 °C	68
Table 4.4:	Optical microscope images of extracted truck tyre-derived crumb rubber after digestion at 190 °C	69
Table 4.5:	Optical microscope images of extracted car tyre-derived crumb rubber after digestion at 165 °C.....	70
Table 4.6:	Optical microscope images of extracted car tyre-derived crumb rubber after digestion at 190 °C.....	71
Table 4.7:	Optical microscope images of extracted conveyor belt-derived crumb rubber after digestion at 165 °C	72
Table 4.8:	Optical microscope images of extracted conveyor belt-derived crumb rubber after digestion at 190 °C	73
Table 4.9:	Optical microscope images of extracted mining tyre-derived crumb rubber after digestion at 165 °C	74
Table 4.10:	Optical microscope images of extracted mining tyre-derived crumb rubber after digestion at 190 °C	75
Table 4.11:	Compressive limit at 70 °C results for all binders following 1 and 36 hours of blending; compressive limit in mm	77
Table 4.12:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on compressive limit at 70 °C	79
Table 4.13:	Viscosity at 165 °C test results; results in Pa·s.....	82
Table 4.14:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on viscosity at 165 °C	84
Table 4.15:	Viscosity at 175 °C test results; results in Pa·s.....	85
Table 4.16:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on viscosity at 175 °C	87

Table 4.17:	Loss on heating results for all binders; loss on heating in %	89
Table 4.18:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on loss on heating.....	90
Table 4.19:	Torsional recovery at 25 °C results for all binders. Results in %	93
Table 4.20:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on torsional recovery at 25 °C	94
Table 4.21:	Resilience at 25 °C results following 1 and 36 hours of blending for the C170 and all binders; MRWA Specification 511:2025 (Table 511.6) specifies a minimum requirement of 20%; results in %.....	97
Table 4.22:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on resilience at 25 °C	98
Table 4.23:	Softening point results for all samples assessed; results in °C.....	102
Table 4.24:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on softening point	104
Table 4.25:	Results for consistency 6% at 60 °C for all binders; results in Pa.s.....	106
Table 4.26:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on consistency 6% at 60 °C.....	107
Table 4.27:	Stress ratio at 10 °C results for all binders	111
Table 4.28:	ANOVA table of the effect of crumb rubber type and size and blending temperature and time on stress ratio at 10 °C	112

Figures

Figure 2.1:	Examples of shredded end-of-life rubber products: (a) truck tyre, (b) car tyre, (c) conveyor belt, and (d) mining tyre	4
Figure 2.2:	Schematic illustration of crumb rubber swelling due to the absorption of the bitumen's light components	5
Figure 2.3:	Schematic illustration of crumb rubber degradation due to prolonged exposure to bitumen at elevated temperature.....	6
Figure 3.1:	Enclosed system for binder blending; set-up used for the first hour of blending	15
Figure 3.2:	Oven used for blending beyond the first hour	15
Figure 3.3:	Washing of CRMBs using a Soxhlet extractor	18
Figure 3.4:	Manual crumb rubber particle extraction	19
Figure 3.5:	Liquid phase extraction with filtration system and underside of filtration system with 75 µm mesh	20
Figure 3.6:	Summary of properties assessed for the constituent materials prior to blending	21
Figure 3.7:	Summary of properties assessed for the materials having undergone blending	22
Figure 3.8:	Crumb rubber composition determination through TGA	24
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	31
Figure 4.2:	Crumb rubber content (R) 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	32
Figure 4.3:	Per cent dissolution (D) 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	33
Figure 4.4:	PS standards with power law data fit	34
Figure 4.5:	Molecular weight distribution curve for baseline C170	34
Figure 4.6:	Molecular weight distribution curves for truck tyre-derived crumb rubber binders: (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C	35
Figure 4.7:	Concentration of components based on their molecular weight for truck tyre-derived crumb rubber-modified binders.....	36
Figure 4.8:	Molecular weight distribution curves for car tyre-derived crumb rubber binders; (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C	37
Figure 4.9:	Concentration of components based on their molecular weight for car tyre-derived crumb rubber-modified binders.....	38
Figure 4.10:	Molecular weight distribution curves for conveyor belt-derived crumb rubber binders: (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C	39
Figure 4.11:	Concentration of components based on their molecular weight for conveyor belt-derived crumb rubber-modified binders.....	40

Figure 4.12:	Molecular weight distribution curves for mining tyre-derived crumb rubber binders: (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C	41
Figure 4.13:	Concentration of components based on their molecular weight for mining tyre-derived crumb rubber-modified binders.....	42
Figure 4.14:	Increase of polymer content with blending time for (a) TR-derived crumb rubber, (b) CT-derived crumb rubber, (c) CB-derived crumb rubber and (d) MT-derived crumb rubber	43
Figure 4.15:	Schematic illustration of cross-linking potentially occurring due to slow digestion and the presence of elevated temperature.....	44
Figure 4.16:	TGA and DTG curves for (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	45
Figure 4.17:	Concentration of moisture and light oils, natural rubber, synthetic rubber and other polymers, and carbon black and fillers for all undigested crumb rubbers	45
Figure 4.18:	Concentration of moisture and light oils, natural rubber, synthetic rubber and other polymers, and carbon black and fillers for all Soxhlet extracted (a) S16 and (b) S30 crumb rubbers.....	46
Figure 4.19:	Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for truck tyre-derived crumb rubber following blending at 165 and 190 °C for 1, 11, and 36 hours	47
Figure 4.20:	(a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted truck tyre-derived crumb rubber following blending	48
Figure 4.21:	Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for car tyre-derived crumb rubber following blending at 165 and 190 °C for 1, 11 and 36 hours	49
Figure 4.22:	(a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted car tyre-derived crumb rubber following blending	50
Figure 4.23:	Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for conveyor belt-derived crumb rubber following blending at 165 and 190 °C for 1, 11 and 36 hours.....	51
Figure 4.24:	(a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted conveyor belt-derived crumb rubber following blending	52
Figure 4.25:	Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for mining tyre-derived crumb rubber following blending at 165 and 190 °C for 1, 11 and 36 hours	53
Figure 4.26:	(a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted mining tyre-derived crumb rubber following blending	54
Figure 4.27:	FTIR spectra for unmodified C170 bitumen	55
Figure 4.28:	FTIR spectra for truck tyre-derived crumb rubber	56
Figure 4.29:	FTIR spectra for car tyre-derived crumb rubber	56
Figure 4.30:	FTIR spectra for conveyor belt-derived crumb rubber.....	57

Figure 4.31: FTIR spectra for mining tyre-derived crumb rubber.....	57
Figure 4.32: FTIR spectra for melt extracted binders blended using truck tyre-derived crumb rubber at 165 °C; (a) S30 and (b) S16	58
Figure 4.33: FTIR spectra for melt extracted binders blended using truck tyre-derived crumb rubber at 190 °C; (a) S30 and (b) S16	59
Figure 4.34: FTIR spectra for melt extracted binders blended using car tyre-derived crumb rubber at 165 °C; (a) S30 and (b) S16	60
Figure 4.35: FTIR spectra for melt extracted binders blended using car tyre-derived crumb rubber at 190 °C; (a) S30 and (b) S16	61
Figure 4.36: FTIR spectra for melt extracted binders blended using conveyor belt-derived crumb rubber at 165 °C; (a) S30 and (b) S16	62
Figure 4.37: FTIR spectra for melt extracted binders blended using conveyor belt-derived crumb rubber at 190 °C; (a) S30 and (b) S16	63
Figure 4.38: FTIR spectra for melt extracted binders blended using mining tyre-derived crumb rubber at 165 °C; (a) S30 and (b) S16	64
Figure 4.39: FTIR spectra for melt extracted binders blended using mining tyre-derived crumb rubber at 190 °C; (a) S30 and (b) S16	65
Figure 4.40: 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.....	66
Figure 4.41: Blending time versus compressive limit at 70 °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	78
Figure 4.42: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on compressive limit at 70 °C	79
Figure 4.43: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for compressive limit at 70 °C	80
Figure 4.44: Correlation between compressive limit at 70 °C and polymer content as measured by HPLC-GPC for (a) S30 and (b) S16	81
Figure 4.45: Blending time versus viscosity at 165 °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	83
Figure 4.46: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on viscosity at 165 °C	84
Figure 4.47: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for viscosity at 165 °C	85
Figure 4.48: Blending time versus viscosity at 175 °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.....	86
Figure 4.49: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on viscosity at 175 °C	87
Figure 4.50: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for viscosity at 175 °C	88

Figure 4.51: 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	90
Figure 4.52: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on loss on heating	91
Figure 4.53: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for loss on heating.....	91
Figure 4.54: Blending time versus torsional recovery 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	94
Figure 4.55: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on torsional recovery at 25 °C.....	95
Figure 4.56: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for torsional recovery at 25 °C	96
Figure 4.57: 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	98
Figure 4.58: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on resilience at 25 °C.....	99
Figure 4.59: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for resilience at 25 °C.....	100
Figure 4.60: Correlation between (a) torsional recovery at 25 °C and (b) resilience at 25 °C with polymer content as measured by HPLC-GPC.....	101
Figure 4.61: 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	103
Figure 4.62: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on softening point.....	104
Figure 4.63: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for softening point	105
Figure 4.64: 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	107
Figure 4.65: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on consistency 6% at 60 °C	108
Figure 4.66: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for consistency 6% at 60 °C.....	108
Figure 4.67: Correlation between (a) softening point and (b) consistency 6% at 60 °C with maltene content as measured by HPLC-GPC	109
Figure 4.68: Correlation between (a) softening point and (b) consistency 6% at 60 °C with asphaltene content as measured by HPLC-GPC.....	110

Figure 4.69: Blending time versus stress ratio at 10 °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.....	112
Figure 4.70: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on stress ratio at 10 °C	113
Figure 4.71: Interaction plots of crumb rubber type, crumb rubber size, blending temperature, and blending time for stress ratio at 10 °C	113
Figure 4.72: Correlation between stress ratio at 10 °C and polymer content as measured by HPLC-GPC	114
Figure 5.1: Schematic illustration of swelling behaviour dependence on rubber type	116
Figure 5.2: Schematic illustration of rubber dissolution scenarios	117
Figure 5.3: Schematic illustration of crumb rubber/bitumen conditions for increased stiffness and elasticity of the crumb rubber-modified binders.....	118

1 Introduction

1.1 Background

To build Australia's capacity for generating high-value recycled commodities, in August 2019, the Council of Australian Governments established a timetable to ban the export of waste plastic, paper, glass and tyres (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 (EoL) 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). The performance of truck tyre–derived CRMBs has been investigated in the literature, where performance improvements have been noted when compared to unmodified bitumen (Bahia & Davies 1994; Lo Presti 2013; Palit et al. 2004).

It is expected that crumb rubber from other sources, such as car tyres, warrant investigation. In addition, it is estimated that between 60,000 and 85,000 tonnes of used conveyor belts are generated each year in Australia, 60% of which are generated in WA. Off-the-road (OTR) tyres, such as those from the mining, agriculture and aviation industries, and rubber from EoL conveyor belts account for 75% of the unrecovered EoL rubber generated annually in Australia. Currently, the annual recovery of these types of materials is less than 10% (Tyre Stewardship Australia 2023), compared to approximately 80% for car and truck tyres (Tyre Stewardship Australia 2022). This comparatively low recovery rate of OTR and conveyor belt rubber can be mainly attributed to the lack of recycling facilities within the vicinity of mining sites, the relatively large size of mining tyres (often weighting over 4.5 tonnes) and logistical challenges (Tyre Stewardship Australia 2022). If crumb rubber derived from these sources can be used in binders, it will reduce the amounts of these materials going to landfill. This research is, thus, critical to understanding a potential end market for the use of crumb rubber derived from car tyres, conveyor belts and mining tyres.

Several Western Australian Road Research and Innovation Program (WARRIP) projects have been recently undertaken to transfer United States (US) CRMB technology relating to open graded asphalt (OGA) and gap graded asphalt (GGA) to WA. These projects have focused on using crumb rubber derived from truck tyres.

Hunter et al. (2015) expressed that the digestion of the crumb rubber in bitumen during the production of a CRMB is affected by:

- the temperature of the binder
- the surface characteristics of the rubber particles
- the size of the rubber particles
- the period for which the rubber and bitumen are kept at the reaction temperature.

Therefore, Main Roads Western Australia (MRWA) has identified a need to gain a better understanding of the effects of these factors on the rheological properties of a CRMB when crumb rubber is derived from truck, car and mining tyres as well as conveyor belts. Having this understanding is expected to enable further development of WA specifications to ensure the quality of the CRMBs that are produced and to maximise potential performance improvements when these types of binders are incorporated into flexible pavements. In addition, the mechanisms affecting rheological performance are expected to be better understood by comparing the chemical and physical characteristics of the different crumb rubbers with the resulting binders.

1.2 Project Scope and Objectives

This research project aimed to investigate the impacts of crumb rubber type and size as well as digestion temperature and time on the rheological properties of the derived binders. In addition, it sought to provide an understanding of the effects of the chemical and physical characteristics of the different crumb rubbers on the properties of the binders as well as on their digestion during blending. 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 MRWA specifications to be undertaken to ensure the optimum use of crumb rubbers.

1.3 Structure of the Report

In this report:

- Section 2 presents a literature review that briefly describes the composition and manufacture of crumb rubber, explains the process of crumb rubber digestion in bitumen and the various parameters that impact it and lists the available international and national specifications for crumb rubber and CRMBs.
- Section 3 describes the materials, sample preparation procedures and experimental and analysis methods used to assess the impact of crumb rubber type, size, blending temperature and blending time on the digestion process.
- Section 4 presents the results.
- Section 5 introduces a discussion surrounding the impact of oxidation on the results, provides a summary of observations regarding the impact of crumb rubber type on the digestion process and presents the compliance of the investigated binders against MRWA Specification 511:2025 *Materials for Bituminous Treatments* and Austroads Technical Specification ATS 3110:2023 *Supply of Polymer Modified Binders*.
- Section 6 presents the conclusions from this research and provides some recommendations.

The report also includes a series of appendices providing supplementary information and test results. Conveyor belt rubber– and mining tyre–derived CRMBs were also subjected to additional testing due to the novelty of their use in bituminous binders. These results are presented in Appendix C.6.

2 Literature Review

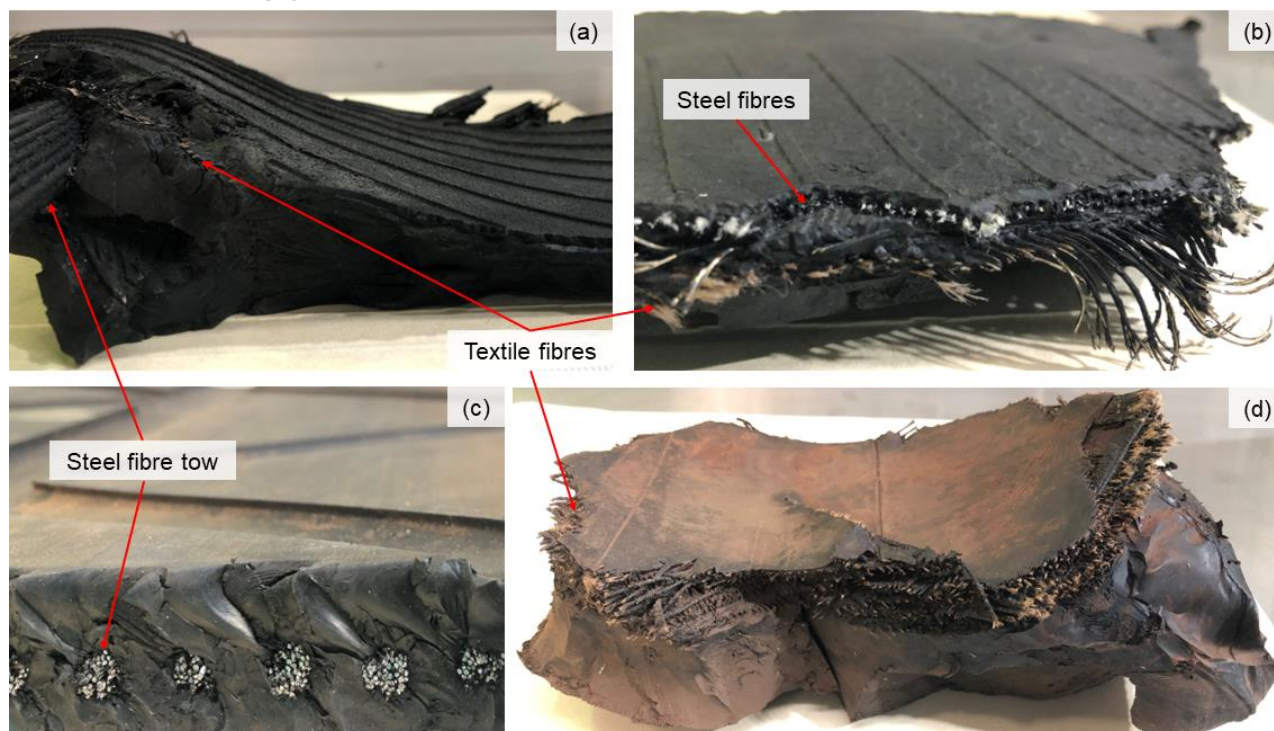
This literature review focused on published work that investigated the effects of different parameters on the digestion of crumb rubber in bitumen used in road pavement applications. Literature relating to the effects of crumb rubber on the performance of binders and the resulting pavements was beyond the scope of this review. The literature review also included a summary of international and Australian/New Zealand specification requirements that relate to the source of crumb rubber used in road construction.

2.1 Crumb Rubber Composition and Manufacture

Tyre and conveyor belt rubber products typically comprise natural (NR) and synthetic rubber (SR), steel, fabric/textiles, and chemical additives. The ratio of these different components varies depending on the rubber application (e.g. for car or truck tyres), as different ratios produce different characteristics. NR is derived from the sap of the *Hevea brasiliensis* tree. SR is typically a styrene-butadiene polymer, which is manufactured using petroleum or coal as a feedstock. Rubbers also contain carbon black, usually in concentrations around 30 wt.%, which is added as a reinforcement that increases the strength of the tyre and its resistance to abrasion. SR and NR are vulcanised prior to their use in tyres. Vulcanisation is carried out by adding sulphur to the rubber. Vulcanization is a thermo-chemical process that creates cross-links between the individual polymer chains in the rubber. Zinc oxide, a chemical that serves as an activator, is also added to the rubber during vulcanisation. The vulcanisation process stabilises the rubber and makes it chemically inert. Other additives may include silica as a reinforcing agent, calcium oxide, which can improve strength and durability, copper oxide as a bonding agent and antioxidants (Jain 2016).

Car tyres contain a greater fabric content when compared to truck tyres (Harrison et al. 2019). The material composition of OTR tyres and conveyor belts is similar to that of car tyres. Overall, OTR tyres are expected to have a comparatively greater concentration of fabric reinforcement than truck tyres to ensure adequate dimensional stability, which is suited to heavy-duty mining activities (Tyre Stewardship Australia 2023). Conveyor belts, specifically, are made using either rubber (e.g. styrene-butadiene rubber (SBR)) or another type of polymer (e.g. polyvinyl chloride (PVC)), which is reinforced with layers of textile fabrics (e.g. polyamide or polyester) or steel cables, depending on performance requirements. Carbon black or antimonite oxide may also be added depending on the formulation. Antimonite oxide is incorporated for flame retardancy (Verakis 2006). Figure 2.1 shows an example of various EoL rubber products that could be converted to crumb rubber.

Figure 2.1: Examples of shredded end-of-life rubber products: (a) truck tyre, (b) car tyre, (c) conveyor belt, and (d) mining tyre



The manufacturing process used to produce crumb rubber is an important factor that 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. During cryogenic processing, the rubber is frozen using liquid nitrogen until it becomes brittle. It is then ground into small particles using a hammer mill. Ambient grinding, on the other hand, takes place at ambient or just above ambient temperature. During this process, scrap rubber is mechanically ground using rotating blades. Cryogenic grinding produces relatively smooth and spherical crumb rubber particles that have a lower surface area, when compared to the more irregularly shaped products of ambient grinding (Lo Presti 2013).

Even though the fabric and steel fibres are removed prior to crumbing, the relative concentrations of NR, SR and other fillers are not altered by the crumbing process. A comparison between truck and car tyre-derived crumb rubber has shown that truck tyre-derived crumb rubber contains greater amounts of NR and lower amounts of SR than car tyre-derived crumb rubber (Genever et al. 2017; Ghavibazoo & Abdelrahman 2013). It has also been reported that conveyor belts typically contain greater concentrations of SR, due to their performance requirements (The QMJ Group 2023).

In Australia, crumb rubber is primarily produced by ambient grinding of truck tyres. There are challenges associated with processing car tyres due to their high fabric content, which needs to be removed before the tyres are crumbed. This requires car tyres to be processed using different equipment than truck tyres (Harrison et al. 2019). Austroads (2022c) identified a total of 16 tyre recycling facilities out of which only 4 operate fabric separating equipment across Australia and New Zealand.

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 2014b). A blend produced by the wet method where the crumb rubber is fully digested is often referred to as a terminal blend. These blends are often categorised separately. 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 GGA and OGA mixes. The wet process is most commonly used in GGA mixes. CRMBs (produced by the wet process) have also been used in dense graded asphalt (DGA), but there can be issues with asphalt compaction when CRMBs are used. Sprayed sealing applications use CRMBs that are produced by the wet process (Austroads 2021b).

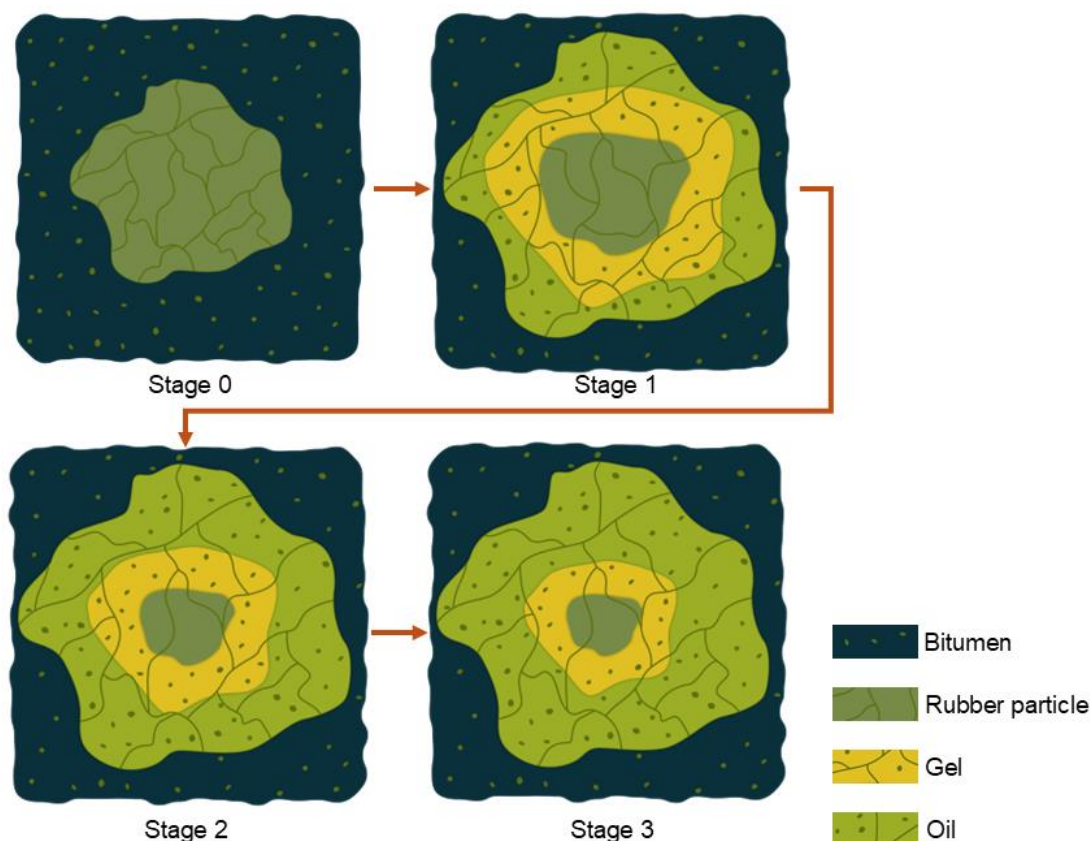
The incorporation of crumb rubber in flexible pavements is, overall, considered to be beneficial as, aside from the positive environmental impacts, it may reduce traffic noise, improve flexibility and increase skid and crack resistance (Lo Presti 2013).

2.2.1 Overview of the Digestion Process

The digestion of crumb rubber in bitumen is affected by various factors. These include the concentration of crumb rubber in the binder, its size (and gradation) and its chemical composition as well as the properties and chemical composition of the base bitumen and the blending time and temperature (Shen et al. 2009). These are further explored in Sections 2.2.2 to 2.2.7.

When crumb rubber is added to hot bitumen, 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 phenomenon was also supported by the measured decrease of aromatics in the base bitumen following crumb rubber digestion; however, care needs to be taken when interpreting these results, as the same phenomenon may also be a manifestation of bitumen aging (Ould-Henia & Dumont 2008). The amount of swelling that occurs when the crumb rubber is added to the bitumen depends on the blending conditions and the presence of other added components. The swelling of the crumb rubber is rapid in the early stages of digestion and then reaches an equilibrium when the light fraction concentration in the tyre rubber reaches a maximum (Picado-Santos et al. 2020). The process of crumb rubber swelling is schematically illustrated in Figure 2.2.

Figure 2.2: Schematic illustration of crumb rubber swelling due to the absorption of the bitumen's light components

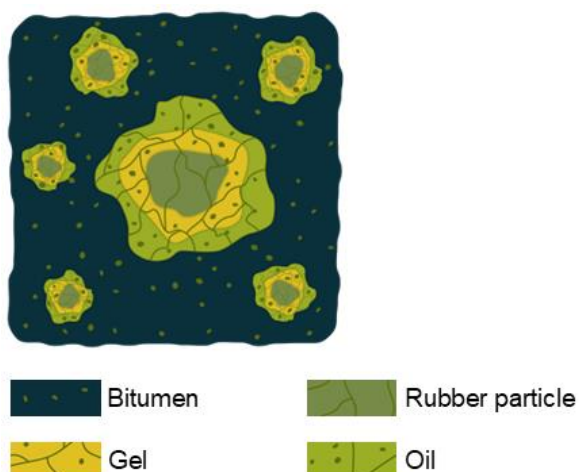


Source: Adapted and recreated from Southern African Bitumen Association (2019).

The addition of crumb rubber particles to bitumen causes changes to the 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). In WA, Stage 1 level of digestion, as schematically represented in Figure 2.2, is targeted.

Upon further blending at elevated temperatures, the crumb rubber particles begin to dissolve in the bitumen and reduce in size. The dissolution of the crumb rubber causes a reduction in high temperature rheological properties (Billiter et al. 1997), which would be expected to reduce the resistance of the CRMB-containing asphalt to permanent deformation (Choi & Urquhart 2019). The dissolution of the crumb rubber is measured by extracting the particles from the matrix and measuring the mass of the particles retained in a 75 µm mesh. Dissolution, therefore, describes a physical change in the blend. Crumb rubber degradation, which is defined by the scission of the rubber's chemical bonds, may also occur under severe interaction conditions (Wang, Apostolidis et al. 2021). The degradation and complete dissolution of crumb rubber particles is schematically illustrated in Figure 2.3.

Figure 2.3: Schematic illustration of crumb rubber degradation due to prolonged exposure to bitumen at elevated temperature



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

2.2.2 Effects of Crumb Rubber Content

As noted in Section 2.2.1, the addition of crumb rubber to bitumen causes swelling of the crumb rubber particles due to the absorption of the light fractions of the bitumen. Ould-Henia and Dumont (2008) observed that a change in the crumb rubber content from 10 to 20 wt.% significantly affected the measured extent of swelling. A greater swelling was observed when the crumb rubber content was lower, and it was proposed that the swelling extent was affected by the volume of each material in the binder (Ould-Henia & Dumont 2008). Gawel et al. (2006) investigated digestion in CRMBs containing 5 and 10 wt.% crumb rubber over 8 hours at 200 °C and also found increased swelling of the crumb rubber when it was added at lower concentrations. Similar findings were also obtained by Airey et al. (2003), who submerged 5 g of crumb rubber in 20, 30, and 40 g of bitumen, consequently changing the concentration of the crumb rubber in the binder. They proposed that the swelling of the crumb rubber was dependent on the available aromatics present in the bitumen rather than the crumb rubber concentration in the binder, suggesting a significant effect of the base bitumen, which is further discussed in Section 2.2.5.

2.2.3 Effects of Crumb Rubber Size, Gradation and Morphology

The effects of crumb rubber size, gradation (Dantas Neto et al. 2006b; Shen et al. 2009) and morphology (Shen et al. 2009) on CRMB properties have previously been studied, suggesting a more notable change in the rheological properties of a CRMB when crumb rubber particles with greater surface area are used

(Dantas Neto et al. 2006b; Shen et al. 2009). The mechanism of these effects could be understood following a study of crumb rubber digestion and dissolution in bitumen.

To investigate the effect of size on rubber swelling, Gawel et al. (2006) obtained cylindrical rubber pieces that were 25 mm in diameter and 0.5 or 0.85 mm in thickness and submerged them in bitumen for various time intervals up to 8 hours. The swelling was quantified by weighing the rubber pieces following washing in toluene. It was found that the smaller pieces increased in mass more rapidly than the larger pieces and that the overall increase was greater for smaller than larger pieces. These observations were thought to be due to the extended time required for the larger particles to swell, which promoted further cross-linking of the rubber, which in turn hindered swelling (Gawel et al. 2006).

Ould-Henia and Dumont (2008), by contrast, did not report a significant effect of particle size on the swelling behaviour of the crumb rubber, rather, as discussed in Sections 2.2.2 and 2.2.5, the content of crumb rubber in the bitumen and the concentration of light fractions of the bitumen were more significant in determining swelling (Ould-Henia & Dumont 2008).

Oliver (1981) investigated the effect of the morphology of crumb rubber with high SR content on the elastic properties of the resulting CRMBs. The variances in morphology were due to the different manufacturing methods used for the crumb rubbers, namely laboratory ground, laboratory drilled and cryogenically ground. It was found that the morphology of the crumb rubber particles had a significant effect on the elastic properties of the CRMBs, with the highly porous surface of the laboratory-ground particles resulting in greater binder modification. These particles were also found to further increase binder elastic recovery with a decrease in size, possibly attributed to their greater surface area. The smoother and more regular cryogenically ground particles, however, were not found to notably modify the CRMBs irrespective of their size (Oliver 1981).

2.2.4 Effects of Crumb Rubber Type

As expressed in Section 1.1, the use of truck tyre-derived crumb rubber is common practice in Australia. This is related to the demonstrated performance of truck tyre-derived crumb rubber, the availability of truck tyres (Austroads 2022), as well as the capacity for processing truck tyres (Harrison et al. 2019). In this section, research where truck tyre-derived crumb rubber and car tyre-derived crumb rubber have been used for the modification of bitumen is summarised. Relevant research investigating the use of conveyor belt-derived crumb rubber and mining tyre-derived crumb rubber is yet to become available.

Typically, truck tyres contain a greater concentration of NR when compared to car tyres, which have a greater content of SR. The capacity of different types of rubber to absorb bitumen components depends on the free volume available, which is determined by the total number and distribution of cross-links as well as the overall polymer chain flexibility (Artamendi & Khalid 2006).

To understand the effects of different concentrations of NR and SR on the swelling rate of crumb rubber, Artamendi and Khalid (2006) used rectangular pieces of truck and car tyre with known masses and dimensions of 15 x 10 x 1 mm³ and immersed them in bitumen at 180 °C. The capacity of the samples to absorb bitumen was quantified by weighing them after wiping at various time intervals. It was found that the bitumen uptake was more pronounced for the truck tyre rubber at the early stages of digestion. Following that initial uptake, the mass increase plateaued, reaching an equilibrium where the overall mass increase was greater for the truck tyre-derived rubber. Upon further exposure to the bitumen, the rubber pieces started degrading. The onset of degradation occurred earlier for the truck tyre-derived rubber than the car tyre-derived rubber (Artamendi & Khalid 2006).

Aside from the capacity of crumb rubber to absorb the light fractions of bitumen, crumb rubber dissolution into bitumen is also part of the modification process and, as such, warrants investigation (Ghavibazoo & Abdelrahman 2013). As noted by Artamendi and Khalid (2006), the degradation of truck tyres was more pronounced than that of car tyres. Similarly, when Ghavibazoo and Abdelrahman (2013) compared the dissolution of a blend of car tyre-derived crumb rubber with that of truck tyre-derived crumb rubber, they observed greater dissolution of the latter. It was expressed that these differences were only significant when

dissolution took place at higher temperatures (160 vs 200 °C). Based on these results, it was thought that the use of higher temperatures released the polymeric components of the crumb rubber into the bitumen. The use of lower temperatures only affected the oily components in the crumb rubber (Ghavibazoo & Abdelrahman 2013).

2.2.5 Effects of Base Bitumen

Bitumen is a complex mixture primarily composed of hydrocarbons with small amounts of nitrogen, sulphur and oxygen. As it derives from crude oil, the relative types and proportions of the different chemical components are dependent on its origin (Airey et al. 2003). The mechanism of crumb rubber digestion, as described in Section 2.1, highlights the significance of the base bitumen constitution in the process.

It has been found that when crumb rubber is added to bitumen with a greater aromatic content, it exhibits greater swelling than when it is added to bitumen with a lower aromatic content (Ould-Henia & Dumont 2008). Similar findings were discussed by Artamendi and Khalid (2006), who explained that the high molecular weight asphaltenes are less likely to diffuse into the high molecular weight rubber structure. Ghavibazoo and Abdelrahman (2013) also found that there was greater dissolution of crumb rubber particles when digestion was undertaken in softer bitumen rather than hard bitumen.

Although Airey et al. (2003) also found that swelling of crumb rubber was more pronounced when digestion was undertaken in softer bitumen, they noted that the maximum swelling observed at 160 °C after 48 hours was more heavily dependent on the nature of the crumb rubber. The chemical composition and viscosity of the bitumen, however, was found to significantly affect the initial rate of absorption, with lower viscosity bitumen yielding a greater rate of absorption (Airey et al. 2003).

To investigate the effect of the base bitumen on the dissolution of crumb rubber particles from 2 different sources and various sizes, Billiter et al. (1997) used 2 different grades of base bitumen and 1 bitumen that they had modified with a highly aromatic rejuvenating agent. It was found that the bitumen modified by the rejuvenating agent interacted with the crumb rubber particles faster when compared with the base bitumens. It was shown, however, that the light aromatics in the modified bitumen were only really effective in dissolving the crumb rubber during the first 10 hours of digestion (Billiter et al. 1997).

2.2.6 Effects of Temperature

Ghavibazoo et al. (2013), following an assessment of various factors and their effects on crumb rubber dissolution, highlighted that temperature was the most significant. As discussed in Section 2.2.4, temperatures near 200 °C appear to release the polymeric components of the crumb rubber into the bitumen, while lower temperatures only appear to affect the oily components of crumb rubber. At even higher temperatures, degradation of the rubber chains and desorption of the light bitumen components from the crumb rubber would be expected to occur, which would cause a deterioration in the physical properties of the CRMB (Ghavibazoo et al. 2013).

To examine the effect of temperature on the rate of rubber swelling, Artamendi and Khalid (2006) used the swelling quantification method and the rectangular rubber pieces described in Section 2.2.4 to investigate digestion at 150, 180 and 210 °C after various time intervals. It was found that the diffusion of the light bitumen components into the rubber specimens increased with an increase in temperature. The mechanism for this phenomenon was proposed to be twofold. It appeared to be a result of both the decreased viscosity of the bitumen at higher temperatures, which allowed the light bitumen components to more easily diffuse into the rubber pieces, and increased the oscillations of the molecular chains in the rubber as a result of the higher temperature, which increased the free volume of the rubber pieces and, hence, their diffusivity to light bitumen fractions (Artamendi & Khalid 2006). An increase in the digestion temperature from 180 to 200 °C was also found to result in an increase in the equilibrium crumb rubber swelling (Gawel et al. 2006).

2.2.7 Effects of Digestion Time

Airey et al. (2003) demonstrated that there was an increase in the absorption of light bitumen fractions by crumb rubber with an increase in digestion time for 1, 4, 24 and 48 hours of exposure at 160 °C. Notably, they showed that absorption was more pronounced during the earlier stages of exposure (Airey et al. 2003). Findings by Gawel et al. (2006) also indicated that there was more rapid swelling during the earlier stages of digestion. This increased swelling at shorter exposure times occurred irrespective of the crumb rubber concentration and particle thickness.

Billiter et al. (1997) and Ghavibazoo and Abdelrahman (2013) showed that the extent of crumb rubber dissolution as a function of digestion time was dependent on the type and size of crumb rubber, the digestion temperature, blending speed and the grade of the base bitumen used to produce the CRMB. Overall, it was found that the rate of crumb rubber dissolution could be decreased by using larger rubber particles or by decreasing the blending temperature or mixing speed (Billiter et al. 1997; Ghavibazoo & Abdelrahman 2013). Truck tyre-derived crumb rubber was found to dissolve faster than car tyre-derived crumb rubber when blending was conducted at 200 °C, but both types of crumb rubber dissolved at a similar rate when blending was undertaken at 160 °C (Ghavibazoo & Abdelrahman 2013).

The trends described above affect the properties of the CRMBs. Several studies have shown that binder properties, such as high temperature viscosity, softening point, penetration and complex modulus (G^*), show an increase at short digestion times, reach a maximum value and then decrease as the blending time is further increased. The early improvement in these properties has been associated with the swelling of the crumb rubber particles in the binder. The later deterioration of binder properties has been associated with dissolution of the crumb rubber particles into the bitumen in the binder (Cao et al. 2011; Oliver 1981; Sybhy et al. 2016).

2.3 Crumb Rubber Specification Requirements

As the incorporation of crumb rubber in road pavements has been common around the world, specifications regarding its use have been developed. This section summarises the recommendations relating to the source of crumb rubber to understand whether existing standards and other relevant documents could hinder the adoption of crumb rubber derived from sources other than EoL truck tyres. Austroads (2021b) provides extensive details about the crumb rubber specifications used in different regions. The relevant documents reviewed for this research project are listed in Table 2.1.

Table 2.1: List of international, Australian and New Zealand standards and specifications reviewed

Department/Agency	Relevant documents	Reference
International		
Arizona Department of Transportation	Standard specifications for road and bridge construction	Arizona Department of Transportation (2021)
California Department of Transportation (Caltrans)	Standard specifications	California Department of Transportation (2022)
Florida Department of Transportation	Standard specifications for road and bridge construction	Florida Department of Transportation (2022)
Texas Department of Transportation	Standard specifications for construction and maintenance of highways, streets, and bridges	Texas Department of Transportation (2014)
ASTM International	Standard terminology relating to rubber	ASTM D1566-21a
	Standard specification for asphalt rubber binder	ASTM D6114/D6114M-19(2023)
	Standard classification for rubber compounding materials – Recycled vulcanizate rubber	ASTM D5603-23
Southern African Bitumen Association (Sabita)	Guidelines for the design, manufacture and construction of bitumen-rubber asphalt wearing courses	Southern African Bitumen Association (2019)
Australia and New Zealand		
Austroads	AP-T359-21 National specification for crumb rubber binders in asphalt	Austroads (2021b)
	Supply of polymer modified binders	ATS 3110:2023
Australian Flexible Pavement Association	Crumb rubber modified open graded and gap graded asphalt pilot specification	Australian Asphalt Pavement Association (2018)
Queensland Department of Transport and Main Roads	Sprayed bituminous treatments (excluding emulsions)	MRTS11:2023
	Polymer modified binder (including crumb rubber)	MRTS18:2025
Main Roads Western Australia	Bituminous surfacings	MRWA Specification 503:2018
	Materials for bituminous treatments	MRWA Specification 511:2025
	Crumb rubber open graded asphalt	MRWA Specification 516:2024
	Crumb rubber gap graded asphalt	MRWA Specification 517:2024
South Australia Department for Infrastructure and Transport	Master Specification RD-BP-S1 Supply of bituminous materials	RD-BP-S1:2024
Transport for New South Wales	Crumb rubber asphalt	QA Specification R118:2020
	Polymer modified binder for pavements	QA Specification 3252:2023
	Crumb rubber	QA Specification 3256:2020
Victoria Department of Transport and Planning	Sprayed bituminous surfacings	Section 408:2022
	High binder crumb rubber asphalt	Section 421:2020
	Light traffic crumb rubber asphalt	Section 422:2019

2.3.1 International

US specifications for crumb rubber suitable for flexible pavements include requirements for the source of the crumb rubber, which vary among jurisdictions, and general requirements for contamination. The overarching ASTM D6114/D6114M-19 (2023) specification provides guidelines for use of crumb rubber in bituminous binders. It references ASTM D1566-21a (for the appropriate terminology to be used) and ASTM D5603-23 (which provides a classification system for all rubbers to be used in bituminous binders). Overall, it is stated that crumb rubber can be sourced from car, truck, bus, agriculture or OTR tyres for Classification 1 or it may be sourced from non-tyre products for Classification 8, leaving the specifics to the discretion of the departments of transport across the USA.

The Arizona Department of Transportation (2021) allows for tyre rubber from trucks, automobiles or other equipment. It includes an exception where 0.5 wt.% textiles/fabric (depending on the specified type) may be permitted (Arizona Department of Transportation 2021). Caltrans (2022) specifies a combination of high-

content (40.0–48.0%) NR crumb rubber and scrap tyre rubber, where scrap tyre can be from vehicle tyres or tyre buffings. The crumb rubber mix is to be composed of 75 ± 2 wt.% scrap tyre–derived crumb rubber and 25 ± 2 wt.% high content NR crumb rubber. It is also specified that it is to contain less than 0.01 wt.% wire and 0.05 wt.% textiles (Caltrans 2022). The Texas Department of Transportation (2014) stipulates that crumb rubber refers to tyre rubber produced by the ambient grinding of car or truck tyres. The Florida Department of Transportation (2022) notes that the crumb rubber should be within the department’s Approved Product List.

The reviewed specifications from the USA commonly specify the requirement for the crumb rubber to be derived from equipment owned and operated within the country (Arizona Department of Transportation 2021; Florida Department of Transportation 2022). Another common denominator across the reviewed US specifications is the requirement for the crumb rubber to be free from non-rubber contaminants (Arizona Department of Transportation 2021; Florida Department of Transportation 2022; Texas Department of Transportation 2014), while the addition of 4 wt.% calcium carbonate is often permitted to promote flowability (Arizona Department of Transportation 2021; Florida Department of Transportation 2022).

The Southern African Bitumen Association (2019) recommends that crumb rubber is produced via ambient grinding and is composed of a blend of truck and car tyres. Radial truck tyres are preferred due to their greater content of NR. It is noted, however, that high concentrations of NR can lead to binders with high viscosity and short shelf life. Similarly to specifications from the USA, the Southern African Bitumen Association (2019) also recommends that crumb rubber is free from non-rubber contaminants except for the addition of up to 4 wt.% calcium carbonate.

2.3.2 Australia and New Zealand

In Australia, CRMBs have been extensively used in sprayed seals in WA, Victoria, New South Wales, Queensland and South Australia (Austroads 2021b), and as a result, the Austroads specification ATS 3110:2023 includes 5 CRMB grades to be used in sprayed sealing applications. The latest version of the national specification also includes one CRMB grade that can be used in asphalt applications. Crumb rubber has also been added via the dry method to asphalt in Victoria and New South Wales. ATS 3110:2023 includes the requirements for crumb rubber used for both the dry and wet mix methods (Austroads 2021b). ATS 3110:2023 requires that the crumb rubber derives from waste tyres generated within Australia, is free from deleterious materials including wire, cord and fluff, and is processed by suppliers accredited by Tyre Stewardship Australia (TSA) or another supplier approved by the Principal.

Although in some cases car and truck tyre–derived crumb rubber is specified, the incorporation of OTR crumb rubber is not explicitly excluded from use in most states in Australia. A pilot specification developed by the Australian Asphalt Pavement Association (2018) (now the Australian Flexible Pavement Association (AfPA)) stipulates that the crumb rubber is to be derived from EoL tyres, must be dry to not inhibit flowability, and must not cause foaming when combined with bitumen. In addition, the use of devulcanised or uncured rubber is prohibited (Australian Asphalt Pavement Association 2018).

MRWA Specification 511:2025 indicates that crumb rubber used in the manufacture of crumb rubber binders needs to consist of EoL tyres or other suitable rubber products. Similarly to the AfPA specification, it prohibits the use of uncured and devulcanised rubber. MRWA Specification 511:2025, like Austroads ATS 3110:2023, specifies that the crumb rubber needs to be sourced from a TSA-accredited supplier or a MRWA-approved supplier. MRWA Specification 511:2025 also requires that the crumb rubber is free from foreign materials such as sand, fibres or aggregate.

In South Australia, Specification RD-BP-S1:2024 *Supply of Bituminous Materials* does not include any requirements relating to the source of the crumb rubber. This specification indicates that crumb rubber needs to be free of cord, wire fluff and other deleterious materials, similarly to Austroads ATS 3110:2023. RD-BP-S1:2024 also requires that the crumb rubber is free of lumps and is capable of being poured freely.

The Victoria Department of Transport and Planning in Section 408:2022 *Sprayed Bituminous Surfacing*, in Section 421:2020 *High Binder Crumb Rubber Asphalt*, and in Section 422:2019 *Light Traffic Crumb Rubber Asphalt* express that the crumb rubber should be sourced from waste tyres generated in Australia and

supplied by TSA-approved suppliers. Section 421:2020 and Section 422:2019 further specify that the crumb rubber must be sourced from truck or car tyres, or a mixture of the 2 and that they are to comply with Austroads AGPT-T190:2019 *Specification Framework for Polymer Modified Binders* (now replaced by Austroads ATS 3110:2023).

Transport for NSW specifies that crumb rubber should be derived from retreader's buffings or obtained from mechanically recycled car or truck tyres in QA Specification 3256:2023 *Crumb Rubber*.

3 Materials and Experimental Methods

This section describes the binder constituent materials (crumb rubber and bitumen) used for this research along with the preparation methods for the blends. In addition, the experimental methods used for the assessment of both the constituent materials as well as the binders are described in detail.

3.1 Materials

3.1.1 Bitumen

This project investigated the digestion potential of different types and sizes of crumb rubber in bitumen. To achieve this, a Class 170 bitumen (henceforth referred to as C170), commonly used as a base for CRMBs in Australia, was sourced from WA. Key properties of the C170 were assessed following MRWA Specification 511:2025 to ensure the compliance of the C170. These properties were measured following the referenced test methods as summarised in Table 3.1.

Table 3.1: Results for measured properties of C170 used and summary of MRWA Specification 511:2025 requirements

Property	Test method	C170 results	MRWA Specification 511 requirements
Viscosity at 60 °C (Pa·s)	AS/NZS 2341.2	178	160–230
Viscosity at 135 °C (Pa·s)	ATM 111	0.36	0.30–0.50
Penetration at 25 °C (pu)	AS 2341.12	66	55–78
Percentage increase in viscosity at 60 °C after rolling thin film oven (RTFO) treatment (%)	AS/NZS 2341.10 and AS/NZS 2341.2	199	300 max.

3.1.2 Crumb Rubbers

Four different types of crumb rubber were supplied from 4 different EoL rubber sources, namely truck tyres (TR), car tyres (CT), conveyor belts (CB), and mining tyres (MT). All 4 were graded to meet Size 16 (S16) (coarse grading) and Size 30 (S30) (standard grading) requirements of ATS 3110:2023. The properties measured for these crumb rubbers are summarised in Table 3.2.

In addition to crumb rubber particles, rectangular pieces of 1–2 mm in thickness, 15 mm in length and 10 mm in width were cut from each of the EoL rubbers.

Table 3.2: Results for measured properties of crumb rubbers used and summary of MRWA Specification 511:2025 requirements (for S30) and Austroads ATS 3110:2023 requirements (for S16 and S30)

Property	Test method	TR	CT	CB	MT	MRWA Specification 511 requirements	Austroads ATS 3110 requirements
S30							
Particle size distribution	AGPT-T143						
passing 2.36 (mm)		100.0	100.0	100.0	100.0	100	100
passing 1.18 (mm)		100.0	100.0	100.0	100.0	100	100
passing 0.6 (mm)		69.0	71.0	66.0	64.0	60–100	60 min.
passing 0.3 (mm)		14.0	16.0	16.0	17.0	0–22	30 max.
passing 0.15 (mm)		0.0	1.0	3.0	2.9	–	–
passing 0.075 (mm)		0	0	0	0	0–2	–
Particle length (mm)	AGPT-T143	0.5	0.5	0.5	0.5	3 max.	3 max.
Bulk density (kg/m ³)	AGPT-T144	349	347	327	294	< 350	Report
Water content (%)	AGPT-T143	0.51	0.54	0.97	0.46	1 max.	1 max.
Foreign materials – other than iron (%)	AGPT-T143	0.000	0.060	0.05	0.01	–	–
Foreign materials – metallic iron (%)	AGPT-T143	0.053	0.036	0.003	0.02	0.1 max.	0.1 max
S16							
Particle size distribution	AGPT-T143						
passing 2.36 (mm)		100.0	100.0	100.0	100.0	–	100
passing 1.18 (mm)		87.0	88.0	90.0	89.9	–	80 min.
passing 0.6 (mm)		5.0	4.0	6.4	6.9	–	10 max.
passing 0.3 (mm)		1.0	0.0	1.2	1.5	–	–
Particle length (mm)	AGPT-T143	2.0	0.5	0.5	1.0	–	3 max.
Bulk density (kg/m ³)	AGPT-T144	332	402	357	417	–	Report
Water content (%)	AGPT-T143	0.67	0.02	1.01	0.43	–	1 max.
Foreign materials – other than iron (%)	AGPT-T143	0.010	0.050	0.08	0.00	–	–
Foreign materials – metallic iron (%)	AGPT-T143	0.040	0.010	0.103	0.010	–	0.1 max

3.2 Sample Preparation

The materials presented in Section 3.1 were used to prepare CRMBs as per the process described in Section 3.2.1. Further processing necessary to conduct some of the testing required is described in Sections 3.2.2 and 3.2.4.

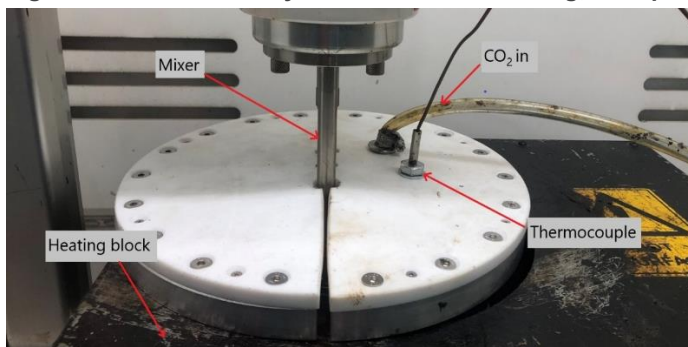
3.2.1 Binder Blending Process

CRMBs of 3,100 g were prepared at the laboratory at the National Transport Research Organisation (NTRO) using the equipment depicted in Figure 3.1. Approximately 2,542 g of C170 was measured, comprising 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 3 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.

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 the heating block depicted in Figure 3.1 with a blanket of CO₂ 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 (rotations per minute).

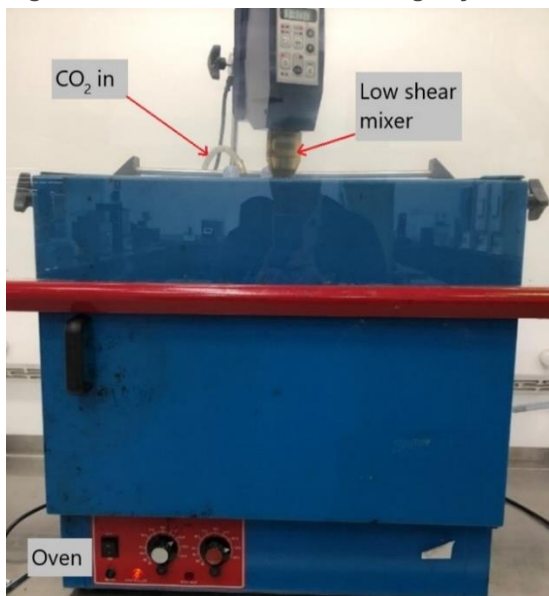
Figure 3.1: Enclosed system for binder blending; set-up used for the first hour of blending



Source: NTRO.

Following the first hour of blending, the CRMBs were transferred to the oven depicted in Figure 3.2. This oven was also equipped with a low shear mixer and an inlet for CO₂ 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.

Figure 3.2: Oven used for blending beyond the first hour



Source: NTRO.

Samples of unmodified C170 (3,100 g) were also exposed to the same regime of blending and were tested to provide a baseline for this research. A total of 35 blends were prepared, 2 for each crumb rubber type and size at each of the temperatures and 3 for the C170 that was used to set the baseline. To investigate the effects of various digestion times, the first blend was subsampled at 1, 2 and 4 hours and the second blend was subsampled at 11, 24 and 36 hours. Blending of the C170 at 190 °C was conducted following the same protocol as the blending of the CRMBs, while one blend was prepared at 165 °C, which was subsampled

after 1 and 36 hours of blending. For testing following subsampling, all samples were re-heated at 180 °C for 1.5 hours as per the method described in ATM 102:2022 *Protocol for Handling Polymer Modified Binders in the Laboratory*. The samples prepared, with their naming conventions as used henceforth in the report, are summarised in Table 3.3.

Table 3.3: Samples prepared and naming conventions

Tyre type	Tyre size	Blending temperature	1h	2h	4h	11h	24h	36h
TR	S30	165 °C	TR30_165C_1h	TR30_165C_2h	TR30_165C_4h	TR30_165C_11h	TR30_165C_24h	TR30_165C_36h
		190 °C	TR30_190C_1h	TR30_190C_2h	TR30_190C_4h	TR30_190C_11h	TR30_190C_24h	TR30_190C_36h
	S16	165 °C	TR16_165C_1h	TR16_165C_2h	TR16_165C_4h	TR16_165C_11h	TR16_165C_24h	TR16_165C_36h
		190 °C	TR16_190C_1h	TR16_190C_2h	TR16_190C_4h	TR16_190C_11h	TR16_190C_24h	TR16_190C_36h
CT	S30	165 °C	CT30_165C_1h	CT30_165C_2h	CT30_165C_4h	CT30_165C_11h	CT30_165C_24h	CT30_165C_36h
		190 °C	CT30_190C_1h	CT30_190C_2h	CT30_190C_4h	CT30_190C_11h	CT30_190C_24h	CT30_190C_36h
	S16	165 °C	CT16_165C_1h	CT16_165C_2h	CT16_165C_4h	CT16_165C_11h	CT16_165C_24h	CT16_165C_36h
		190 °C	CT16_190C_1h	CT16_190C_2h	CT16_190C_4h	CT16_190C_11h	CT16_190C_24h	CT16_190C_36h
CB	S30	165 °C	CB30_165C_1h	CB30_165C_2h	CB30_165C_4h	CB30_165C_11h	CB30_165C_24h	CB30_165C_36h
		190 °C	CB30_190C_1h	CB30_190C_2h	CB30_190C_4h	CB30_190C_11h	CB30_190C_24h	CB30_190C_36h
	S16	165 °C	CB16_165C_1h	CB16_165C_2h	CB16_165C_4h	CB16_165C_11h	CB16_165C_24h	CB16_165C_36h
		190 °C	CB16_190C_1h	CB16_190C_2h	CB16_190C_4h	CB16_190C_11h	CB16_190C_24h	CB16_190C_36h
MT	S30	165 °C	MT30_165C_1h	MT30_165C_2h	MT30_165C_4h	MT30_165C_11h	MT30_165C_24h	MT30_165C_36h
		190 °C	MT30_190C_1h	MT30_190C_2h	MT30_190C_4h	MT30_190C_11h	MT30_190C_24h	MT30_190C_36h
	S16	165 °C	MT16_165C_1h	MT16_165C_2h	MT16_165C_4h	MT16_165C_11h	MT16_165C_24h	MT16_165C_36h
		190 °C	MT16_190C_1h	MT16_190C_2h	MT16_190C_4h	MT16_190C_11h	MT16_190C_24h	MT16_190C_36h
N/A	N/A	165 °C	C170_165C_1h					C170_165C_36h
		190 °C	C170_190C_1h	C170_190C_2h	C170_190C_4h	C170_190C_11h	C170_190C_24h	C170_190C_36h

3.2.2 Soxhlet Extractor Washing of Crumb Rubber-Modified Binders

Soxhlet extraction was conducted following Austroads AGPT-T142:2020 *Rubber Content of Crumb Rubber Modified Bitumen: Soxhlet Method* to extract the crumb rubber from the binders following digestion. 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*. Figure 3.3 shows the set-up used.

Figure 3.3: Washing of CRMBs using a Soxhlet extractor



To quantify the crumb rubber mass, and consequently the mass of crumb rubber dissolved in the bitumen, the crumb rubber recovery factor was firstly determined by washing approximately 10 g of the as-received crumb rubber following the same method. The recovery factor was determined for each rubber type and size. These are presented in Table 3.4.

Table 3.4: Measured recovery factor for each crumb rubber type and size

Crumb rubber type	Crumb rubber size	Recovery factor
TR	S16	0.923
	S30	0.916
CT	S16	0.887
	S30	0.889
CB	S16	0.921
	S30	0.921
MT	S16	0.957
	S30	0.928

The crumb rubber dissolution is further described in Section 3.3.7.

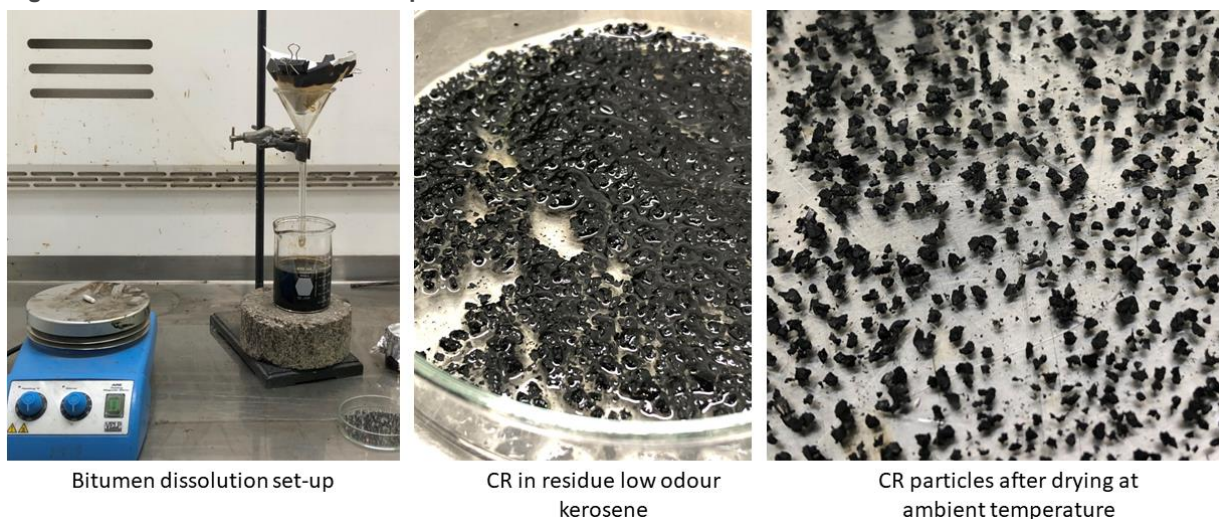
3.2.3 Manual Washing of Crumb Rubber-Modified Binders

Crumb rubber extracted using AGPT-T142:2020 was found to agglomerate, hindering further particle analysis. To facilitate particle size analysis and optical microscopy, as described in Sections 3.3.8 and 3.3.9, a manual washing method was employed as follows:

1. Approximately 6 g of the binder were placed in a glass jar and topped with approximately 30 ml low odour kerosene.
2. A magnetic stirrer was used for mixing on a hot plate set to 50 °C.
3. Stirring was undertaken for 15 minutes.
4. Time was allowed for all visible floating crumb rubber particles to settle, and then, the solution was collected with a glass pipette putting it through a 75 µm mesh filter.
5. Steps 3 and 4 were repeated until the solution had a light straw colour.
6. The crumb rubber particles with any remaining low odour kerosene were then placed in a petri dish and allowed to dry at ambient temperature overnight.

The method and results are presented in Figure 3.4. Only S16 crumb rubber particles could be effectively extracted, with S30 particles still presenting significant agglomeration.

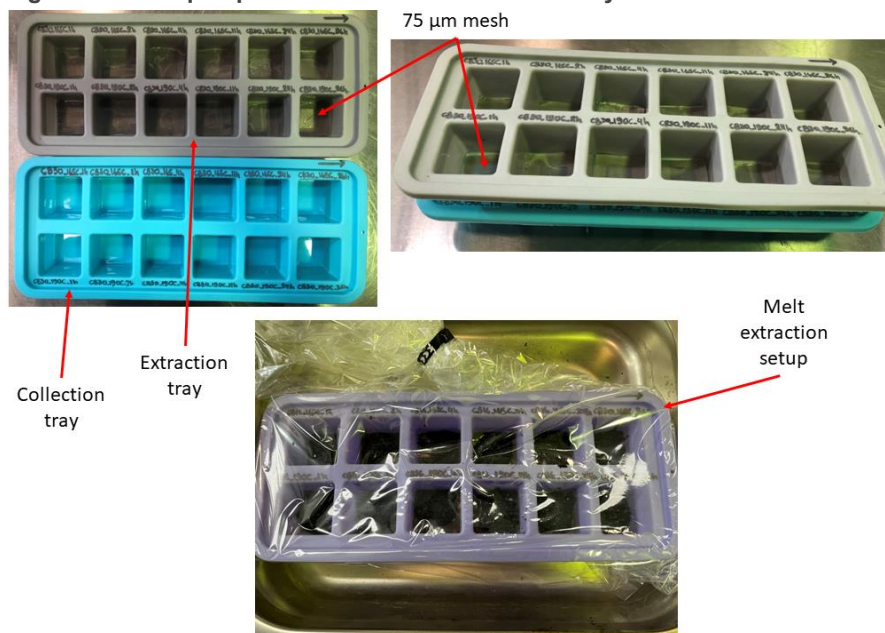
Figure 3.4: Manual crumb rubber particle extraction



3.2.4 Melt-Extract of Crumb Rubber-Modified Binders

To analyse the liquid phase of the binders, 2–3 g of the binders 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. This experimental set-up is shown in Figure 3.5. The assembly was then placed in an oven at 165 °C for 30 minutes. It was then removed and placed in a refrigerator for rapid cooling.

Figure 3.5: Liquid phase extraction with filtration system and underside of filtration system with 75 µm mesh

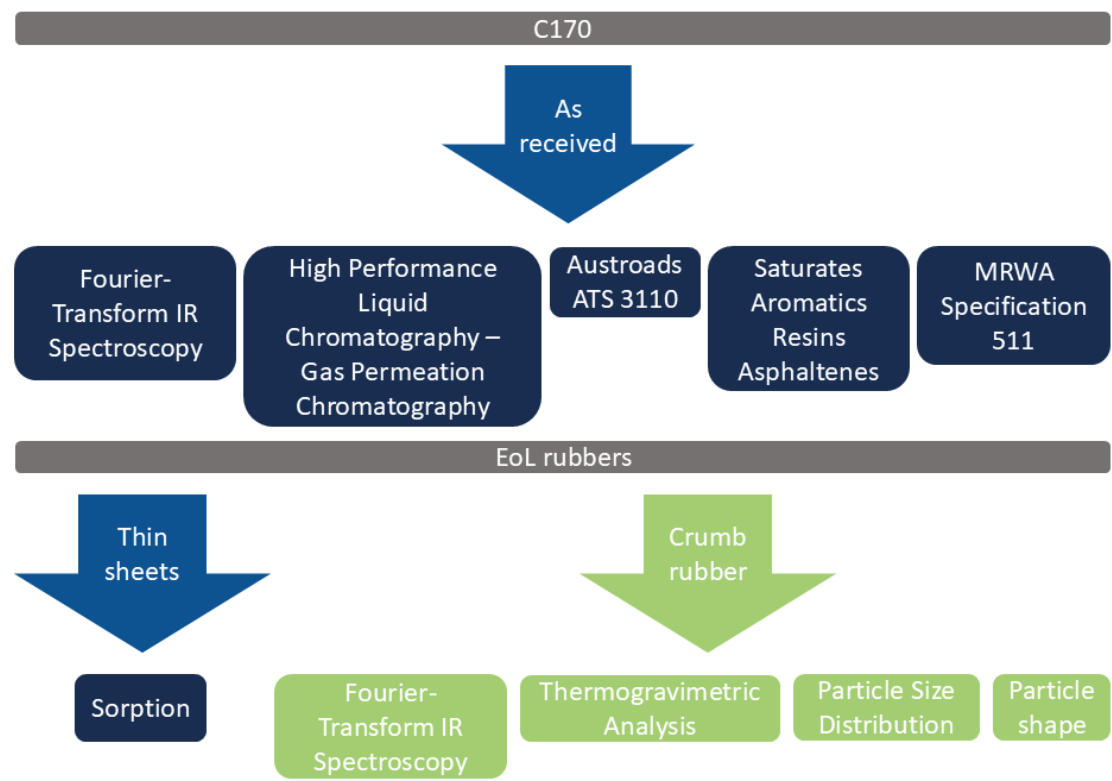


3.3 Experimental Methods

Partially digested CRMBs are not a homogeneous material, they are rather a composite of a liquid and a solid phase. The liquid phase comprises the bitumen and any dissolved or degraded ($< 75 \mu\text{m}$) portion of the crumb rubber, and the solid phase comprises the rubber particle and any bitumen light components that it has absorbed. As described in Section 2.2.1, the phases are more nuanced, but for the purposes of explaining the results of Section 4, this simplification would suffice. It is suspected that, due to the nature of the tests conducted, the results presented in Sections 4.3 to 4.7 are not influenced by the composite or each of its constituent phases equally. Rather some results might be manifesting the properties of the liquid phase and others might be influenced more by the presence of rubber particles. Therefore, the analytical experimental methods used (Sections 3.3.1 to 3.3.9) to understand the impact of crumb rubber type and size and blending parameters on binder handling and performance (Sections 3.3.10 to 3.3.17) 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 3.6 summarises the tests performed on the starting materials described in Section 3.1.

Figure 3.6: Summary of properties assessed for the constituent materials prior to blending

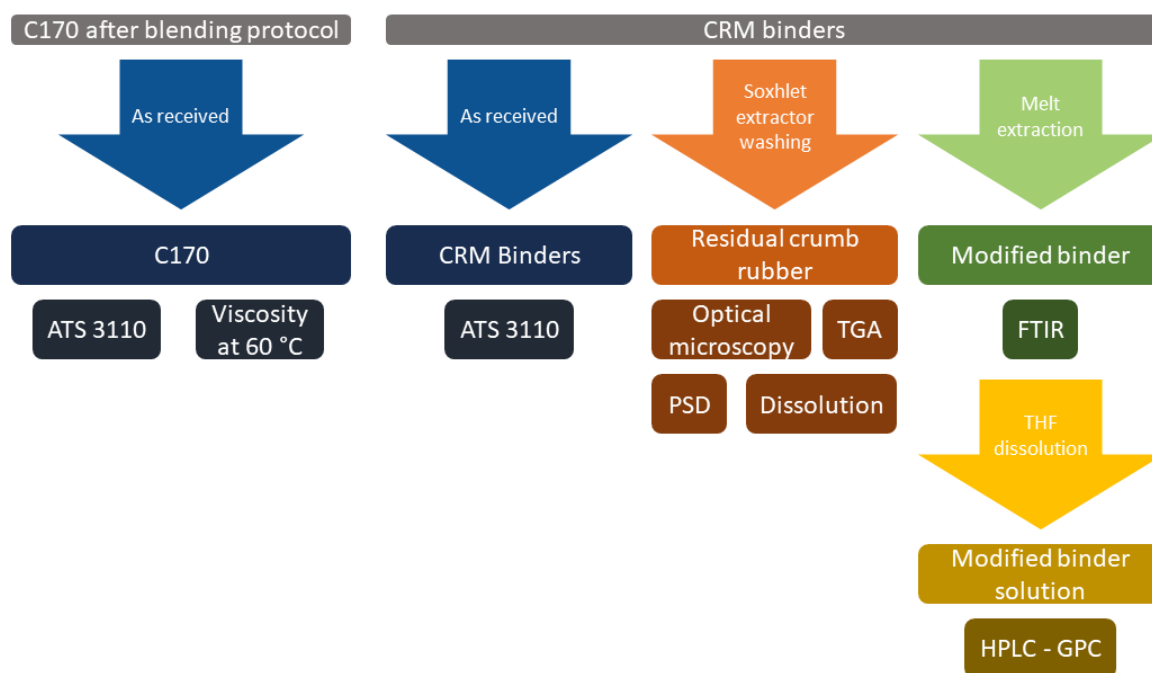


Notes:

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

Figure 3.7 summarises the testing protocol for the materials following the blending process described in Section 3.2.1. C170 and CRMBs were assessed as-blended, the crumb rubber particles and the liquid phase were assessed after having been extracted from the CRMBs following the methods described in Section 3.2.2 and 3.2.4, respectively. Specimens analysed through high-performance liquid chromatography – gel permeation chromatography (HPLC-GPC) were further prepared as appropriate for the test method. The sample preparation methods used for these tests are described in Section 3.3.5.

Figure 3.7: 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).

A list of the samples analysed per method is provided in Appendix A, and the description of each method is provided in Sections 3.3.10 to 3.3.17.

3.3.1 SARA by Column Chromatography

To determine the saturate, aromatic, resin and asphaltene (SARA) fractions, the asphaltenes were firstly separated from the maltenes (saturates, aromatics, resins) using an NTRO-developed method. The maltene constituents were then quantified using an NTRO-developed Iatroscan method.

To separate the asphaltenes, 1 g of bio-oil was added in a 40 mL beaker. To the beaker, 40 mL of AR n-heptane were also added, and the beaker was covered with aluminium foil, which was secured with a rubber band. The bitumen in n-heptane was left for approximately 19 hours and stirred using a glass rod 45 minutes before being filtered through a Hirsch funnel connected to a water vacuum. The maltene solution was collected in a beaker. The asphaltenes were subsequently washed with another 30 mL of n-heptane to remove any remaining maltenes into the beaker. The asphaltenes that remained in the Hirsch funnel were dried in an oven at 60 °C for 30 minutes and then cooled in a desiccator for another 30 minutes. The Hirsch funnel with the asphaltenes was weighed, and their content in the initial mass of bitumen was calculated. The maltene solution was left in a fume cupboard overnight to allow for the solvent to evaporate. The beaker was then put in an oven at 60 °C for 30 minutes and cooled in a desiccator for another 30 minutes. The beaker with the maltenes was then weighed and their concentration in the bitumen was calculated.

Approximately 0.2 g of the extracted maltenes were used to create a 10 mL standard solution using AR chloroform. Half the Iatroscan rods were spotted with 1 µL of reference bitumen while the other half were spotted using the maltene solution (alternating rods were spotted with each). The saturates and aromatics were developed in AR grade n-heptane followed by a development in toluene allowing a drying time of 10 minutes in between. The resins remained at the bottom of the rod. ChromStar software was used to produce the chromatograms generated by the rods containing the standard and sample C170-derived maltenes as they were run through using a hydrogen flame.

3.3.2 Viscosity at 60 °C

The viscosity at 60 °C of the as-received and blended C170 was determined using AS/NZS 2341.2:2015 *Methods of Testing Bitumen and Related Roadmaking Products, Method 2: Determination of Dynamic Viscosity by Vacuum Capillary Viscometer*. A list of the samples assessed via this method is provided in Table 3.5.

Table 3.5: Samples assessed according to AS/NZS 2341.2:2015

Samples	Digestion time						
	0h	1h	2h	4h	11h	24h	36h
C170	✓						
C170_165C_		✓					✓
C170_190C_		✓	✓	✓	✓	✓	✓

3.3.3 Crumb Rubber Swelling

To investigate the swelling of all 4 different rubbers, thin film specimens (20 mm x 40 mm x 3 mm) from each rubber (TR, CT, CB, MT) were weighed and immersed in 120 g of bitumen at 165 and 190 °C. They were then taken out from the bitumen at defined time intervals (0, 300, 600, 900, 1,200, 1,800, 2,700, 3,600, 5,400, 7,200, 9,000, 10,800, 12,600, 14,400, 18,000, 21,600, 25,200, 28,800, 86,400, 93,600, 100,800 seconds), their thickness was measured using Vernier callipers and they were weighed. To make sure that all bitumen was removed from the surface of the rubber films, they were wiped with a piece of paper and then cleaned with a piece of fabric with kerosene. The percentage of the mass increase (M_t) was then calculated as per Equation 1:

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

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).

The results were plotted against $t^{1/2}/d$, where t refers to the time at which the mass and thickness were measured after exposure and d refers to the measured thickness of the rubber at that time.

3.3.4 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) is commonly used to assess thermal stability, degradation kinetics and loss of solvent, among other parameters. In this method, the mass of a specimen is measured against time or temperature, while it is heated in either a reactive, oxidising or inert atmosphere. The samples decompose as different molecules evaporate at different temperatures. There may be multiple decomposition steps, each corresponding to the decomposition temperature of the specimens' components.

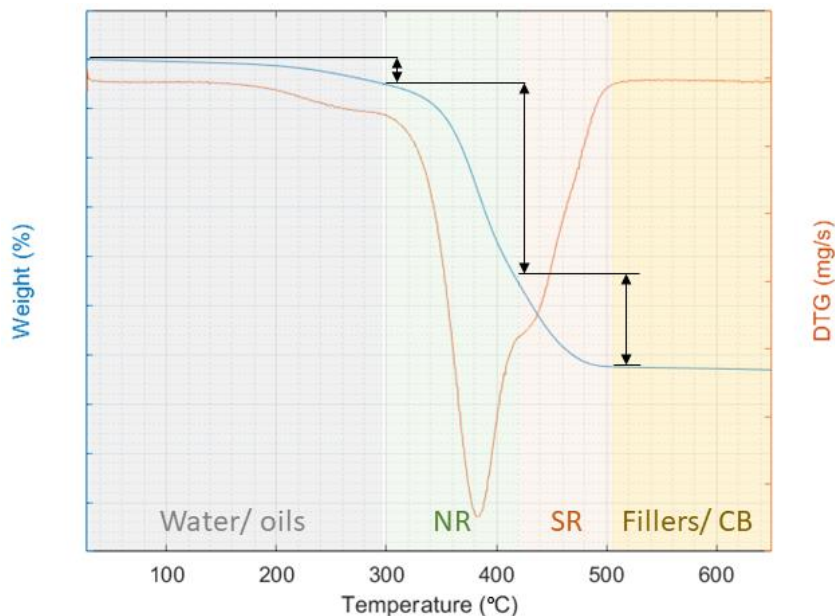
TGA was carried out using a PerkinElmer TGA 8000. Approximately 5 mg of the crumb rubber samples, as received and as extracted after washing through the Soxhlet Extractor (method described in Section 3.2.2), were loaded into a ceramic crucible and placed in the TGA. The samples analysed are summarised in Table A.1. Four specimens per sample were tested.

The samples were heated from 30 to 600 °C at a rate of 10 °C/min in a nitrogen atmosphere. The flow rate of the nitrogen was maintained at 20 ml/min. To calculate the change in the composition of crumb rubber during

interaction with the bitumen, the decomposition temperature range of each component in the crumb rubber samples was obtained using their mass loss within the derivative thermogravimetry (DTG) regions, as depicted in Figure 3.8. Mass loss up to 300 °C is attributed to the evaporation of moisture and light oils, between 300 and approximately 400 °C (depending on the crumb rubber) to the degradation of NR, between approximately 400 and 500 °C to the degradation of SR, while the mass retained beyond that point and up to 600 °C is associated with carbon black and other fillers present (Datta et al. 2017).

The mass at each temperature of interest was determined as the average of 6 measurements, 3 below the target temperature and 3 above the target temperature. The point of transition between the degradation of NR and SR was graphically determined based on the peaks of the DTG curve. The average of 4 tests with standard deviation is reported. Representative curves for each sample are provided in Appendix D.1.

Figure 3.8: Crumb rubber composition determination through TGA



Note: In this figure, CB refers to carbon black in crumb rubber.

3.3.5 High Performance Liquid Chromatography – Gel Permeation Chromatography

GPC is a liquid chromatographic method that separates the molecules in a solution by their size. This method achieves separation using porous beads packed in a column, based on the size or hydrodynamic volume of the analytes (a substance whose chemical constituents are being identified and measured). When the dissolved sample is carried through the column filled with porous material (stationary phase) by a continually flowing stream (the mobile phase), smaller analytes can enter the pores of the beads and remain in the column for a longer time. Conversely, larger analytes travel outside the pores and, thus, elute faster. Therefore, molecules are separated into different sizes that elute at different retention times.

HPLC-GPC was used to understand the size distribution of molecules present in the melt-extracted binder (prepared according to Section 3.2.4) when compared to the C170. The samples analysed through this process are listed in Table A.2.

To prepare for HPLC-GPC testing, melt-extracted samples of approximately 2 mg were weighed using a Mettler Toledo XS105 DualRange balance (readability 0.01 mg) and dissolved in 1 mL tetrahydrofuran (THF, HPLC grade), which is suitable as an HPLC mobile phase, to make 2 mg of binder per mL of THF solution. The mixtures were manually shaken and kept in the sample vials overnight for complete dissolution of the bitumen. Then, the mixtures were filtered through a 0.45 µm polytetrafluoroethylene filter prior to being analysed by GPC. To measure the molecular weight distribution of the samples, GPC analysis was carried out using a Shimadzu system. The instrument operating conditions are listed in Table 3.6.

Table 3.6: Instrument conditions for HPLC-GPC

Condition	Parameter
Effluent	Tetrahydrofuran (THF)
Effluent flow rate	0.5 mL/min
Column	Waters Styragel® HT 2, 7.8 mm I.D. × 300 mm (THF)
Column temperature	40 °C
Sample injection volume	10 µL
Detector	Refractive index detector RID-20A

Calibration was carried out with polystyrene (PS) standards with various molecular weights as shown in Table 3.7.

Table 3.7: Polystyrene standards

Vial code	Molecular weight (g/mol)	Retention time (min)
Red	55,300	11.11
	10,280	12.89
	2,850	15.02
	625	18.06
Yellow	29,160	11.44
	6,530	13.55
	2,000	15.67
	447	18.93
Green	22,130	11.75
	4,850	14
	940	17.17

3.3.6 Fourier-Transform Infrared Spectroscopy

A PerkinElmer Spectrum Two Fourier-transform infrared (FTIR) spectrometer was employed in attenuated total reflectance (ATR) mode to identify the functional groups present on the surface of melt-extracted specimens (prepared according to Section 3.2.4) and to compare them to those of the C170 and different types of crumb rubber. Due to the presence of carbon black in the crumb rubber, whose refractive index is close to that of the commonly used diamond ATR crystal, a germanium ATR crystal was used for these tests. The samples analysed are listed in Table A.3.

The samples were analysed in the mid infrared range (600–4,000 cm⁻¹). A total of 32 scans with a scan speed of 0.2 cm/s and resolution of 4 cm⁻¹ were acquired for each of the examined specimens. The results were pre-processed using the baseline correction function in Spectrum IR software.

3.3.7 Crumb Rubber Dissolution

To investigate the dissolution of crumb rubber during the digestion process, the crumb rubber was extracted from the binder following the Soxhlet extractor washing process described in Section 3.2.2 and weighed. The crumb rubber content in the binder following digestion was calculated using Equation 2 (Equation 3 in AGPT-T142:2020):

$$R = \frac{100 \times (M_3 - M_1)}{P \times (M_2 - M_1)} \quad 2$$

where:

R = rubber content of binder sample (wt.%)

- M_1 = mass of extraction thimble and plug after drying (g)
- M_2 = mass of extraction thimble, plug and binder sample before extraction (g)
- M_3 = mass of extraction thimble, plug and crumb rubber after extraction and drying to constant mass (g)
- P = rubber recovery factor

The dissolution (D) of the crumb rubber was calculated as the per cent difference between the initial concentration of crumb rubber (18 wt.%) and the rubber content in the binder sample (R) calculated using Equation 2. The samples listed in Table A.4 were analysed through this method.

3.3.8 Particle Size Distribution

The particle size distribution (PSD) of crumb rubber particles following digestion was considered a useful measure to understand not only digestion but also to inform asphalt mix design. For useful information to be extracted from such a method, though, the particles need to be free from agglomeration. Various methods for the extraction of crumb rubber particles were trialled. These included the manual washing method described in Section 3.2.3, Soxhlet extractor washing described in Section 3.2.2, and washing with mineral turpentine in an open tray until a light straw colour and filtered through a 75 μm filter followed by drying in an RTFO oven at 40 °C for 4.5 hours.

Particles that derived from manual washing were found to achieve the best outcome. The PSD of the samples listed in Table A.5 was measured using a Litesizer DIA 500 dynamic image analyser by Anton Paar. The system was equipped with a Free Fall dispersion unit with the capability to measure particles between 0.8 and 8,000 μm .

However, from the results obtained and observations from optical microscopy (see Section 4.2.6), it was evident that the collection of particles from the binders that were free from agglomeration was not possible from any of the methods used. As such, the results obtained are not reported in Section 4. Appendix D.2 shows the measured 10th, 50th and 90th percentile for the samples of Table A.5.

3.3.9 Optical Microscopy

The crumb rubber particles manually washed through the method described in Section 3.2.3 were placed on slides for optical microscopy. Images were acquired using an Olympus optical microscope to qualitatively assess their morphology following digestion. The morphology of as-received crumb rubber was also evaluated. Samples assessed are listed in Table A.6.

3.3.10 Viscosity at 165 and 175 °C

Viscosity testing at 165 and 175 °C was carried out in accordance with ATM 111:2022 *Handling Viscosity of Polymer Modified Binders (Brookfield Thermosel)* for all samples as listed in Table A.7. These tests were conducted using a Brookfield spindle SC4-29, which is recommended for use with S45R grade crumb rubber binders in MRWA Specification 511:2025 and S15R grade binders in Austroads ATS 3110:2023.

3.3.11 Stress Ratio at 10 °C

Stress ratio at 10 °C tests were conducted in accordance with AGPT-T125:2018 *Stress Ratio of Bituminous Binders Using the Dynamic Shear Rheometer* using a TA Instruments model AR 1500 ex dynamic shear rheometer (DSR). The samples examined are listed in Table A.8.

3.3.12 Torsional Recovery at 25 °C

Torsional recovery at 25 °C tests were carried out in accordance with ATM 122:2022 *Torsional Recovery of Polymer Modified Binders* for all samples listed in Table A.9.

3.3.13 Resilience at 25 °C

Resilience tests were conducted at 25 °C in accordance with ASTM D5329-20 *Standard Test Methods for Sealants and Fillers, Hot-applied, for Joints and Cracks in Asphalt Pavements and Portland Cement Concrete Pavements* (10. Resilience) for the samples listed in Table A.10.

3.3.14 Softening Point

Softening point measurements were conducted in accordance with AGPT-T131:2006 *Softening Point of Polymer Modified Binders* (this method references method AS 2341.18, which details variations to ASTM D36/D36M-14(2020) *Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)*). The samples assessed are summarised in Table A.11.

3.3.15 Consistency at 6% at 60 °C

Consistency 6% at 60 °C tests were conducted in duplicate in accordance with AGPT-T121:2014 *Shear Properties of Polymer Modified Binders (ARRB ELASTOMETER)*. These tests were performed on samples as summarised in Table A.12.

3.3.16 Loss on Heating

Loss on heating tests were performed in accordance with Austroads test method ATM 103:2022 *Mass Change of Loss on Heating of Polymer Modified Binders after Rolling Thin Film Oven (RTFO) Treatment* for the samples noted in Table A.13.

3.3.17 Compressive Limit at 70 °C

Compressive limit tests were conducted at 70 °C in accordance with ATM 132:2022 *Compressive Limit of Polymer Modified Binders* on the samples listed in Table A.14.

3.4 Statistical Analysis

A full factorial design of experiments with varying levels of investigation depending on the properties assessed (Sections 3.3.10 to 3.3.17) was used. This analysis provided an understanding of the effects, interactions and significance of blending time and temperature as well as crumb rubber size and type on these properties. The size of crumb rubber was investigated at 2 levels (S16 and S30), the type of crumb rubber was investigated at 4 levels (TR, CT, CB, MT), the blending temperature was investigated at 2 levels (165 and 190 °C) and the blending time was investigated at varying levels between 2 and 6, as shown for each property in Table A.7 to Table A.14.

An analysis of variance (ANOVA) was performed using Minitab. The ANOVA tables, main effects and interaction plots are presented in Sections 4.3 to 4.7. To consider the deviation in results for each sample group per test, each individual test result (for each specimen tested), either triplicate or duplicate depending on the test method, was included in the analysis. Even though it is recognised that for the ANOVA results to be more robust a larger number of tests would be preferred, this analysis was still deemed valuable in this

research as it provides insights into the trends, or the lack thereof, for the investigated test methods. A significance level of 5% (P-value < 0.05) was used for this analysis.

To understand the potential correlation between crumb rubber digestion and binder performance, matrix plots of polymer dissolution, as measured through HPLC-GPC against the investigated properties of the CRMBs of this research, were drawn using Pearson's correlation (95% correlation interval) using Minitab. The ranges for correlation values used in this research are summarised in Table 3.8.

Table 3.8: Correlation scale ranges

Correlation coefficient range	Value
1	Perfect
$0.70 \leq r \leq 0.99$	Strong
$0.40 \leq r \leq 0.69$	Moderate
$0.10 \leq r \leq 0.39$	Weak
$0.01 \leq r \leq 0.09$	Negligible
0	None

Source: Adapted from Akoglu (2018).

4 Results

This section presents the test results acquired following the experimental methods of Section 3.3. The performance tests were analysed using ANOVA, whereas the analytical portion of the work, along with learnings from other literature, were used to explain the observed behaviours. Pearson's correlation was also used to identify whether correlations exist between crumb rubber swelling and dissolution and binder performance.

4.1 Characterisation of Unmodified Bitumen

4.1.1 SARA by Column Chromatography

Measuring the SARA fractions of the base bitumen is fundamental for understanding the physicochemical interactions that may take place between the C170 and the different types and sizes of crumb rubber. These will further explain the performance results observed from the corresponding CRMBs. The SARA fractions of the base C170 used in this study are listed in Table 4.1.

Table 4.1: SARA fractions of C170 bitumen

SARA fractions	C170 (%)
Saturates	3.8
Aromatics	64.7
Resins	14.5
Asphaltenes	17.0

4.1.2 Viscosity at 60 °C

Table 4.2 presents the viscosity at 60 °C of the as-received and blended C170. A digestion time of 1 hour was not found to significantly affect the viscosity of the C170 at 60 °C irrespective of the blending temperature. However, after 36 hours of blending, the viscosity at 60 °C appeared to have notably increased irrespective of blending temperature. It is noted that although these results precede those of CRMBs in this report, chronologically, this testing was undertaken in retrospect to understand potential thermo-oxidative effects.

Table 4.2: Viscosity at 60 °C test results for the base C170 bitumen after different blending times at 165 or 190 °C; results in Pa·s

Samples	Digestion time						
	0h	1h	2h	4h	11h	24h	36h
C170	178	–	–	–	–	–	–
C170_165C_	–	186	–	–	–	–	957
C170_190C_	–	187	–	–	–	–	11,700
C170_190C_	–	187	198	333	246	569	19,136

The increase in viscosity at 60 °C with increased blending times indicates hardening of the bitumen due to oxidation. For sample C170_190C, which was tested at all blending times, this increase appears to be more pronounced when blending exceeded 11 hours. Unlike the findings following 1 hour of blending, after 36 hours of blending, the temperature was found to affect the viscosity at 60 °C, with blending at 190 °C resulting in almost 20 times greater viscosity when compared to that following blending at 165 °C. This increase in viscosity with increased blending temperature is in line with the rate of bitumen oxidation increasing at higher temperatures as discussed by Hunter et al. (2015).

As C170 was blended with a CO₂ blanket, the observed increase in viscosity at 60 °C was unexpected. It is suspected that oxidation occurred due to the change in the set-up from that of Figure 3.1 to that of Figure 3.2 following the first hour of blending. The flow rate selected for the CO₂ was selected following a series of experiments using a high shear mixer at the set-up of Figure 3.1, and therefore, it is possible that the changed conditions of the oven of Figure 3.2 allowed for enough oxygen to come and remain in contact with the bitumen during the remaining of the blending. It is also noted that the bitumen was subsampled at 1, 2, 4, 11, 24 and 36 hours, potentially contributing to these results. The impacts of subsampling can be understood when comparing the viscosity at 60 °C for C170_190C_36h subsampled at 1 and 36 hours and that of sample C170_190C_36h subsampled at 1, 2, 4, 11, 24 and 36 hours, whereby the viscosity at 60 °C of the latter is notably greater. This suggests that the repeated exposure of the bitumen to ambient conditions to facilitate subsampling, as well as the increased surface area of the bitumen, promoted oxidation.

Practically, when considering a commercial hot storage tank or blending facilities, oxidation does not occur. This is related to the shape of the tanks being long cylinders with a small diameter, which minimises the binder surface area in contact with air.

4.2 Crumb Rubber Digestion

In this section, the results from the methods described in Sections 3.3.3 to 3.3.9 are presented. These methods were used in an attempt to understand whether different crumb rubbers swell at different rates as a result of exposure to bitumen and to quantify the dissolution of crumb rubber components in bitumen following prolonged exposure.

4.2.1 Rubber Swelling

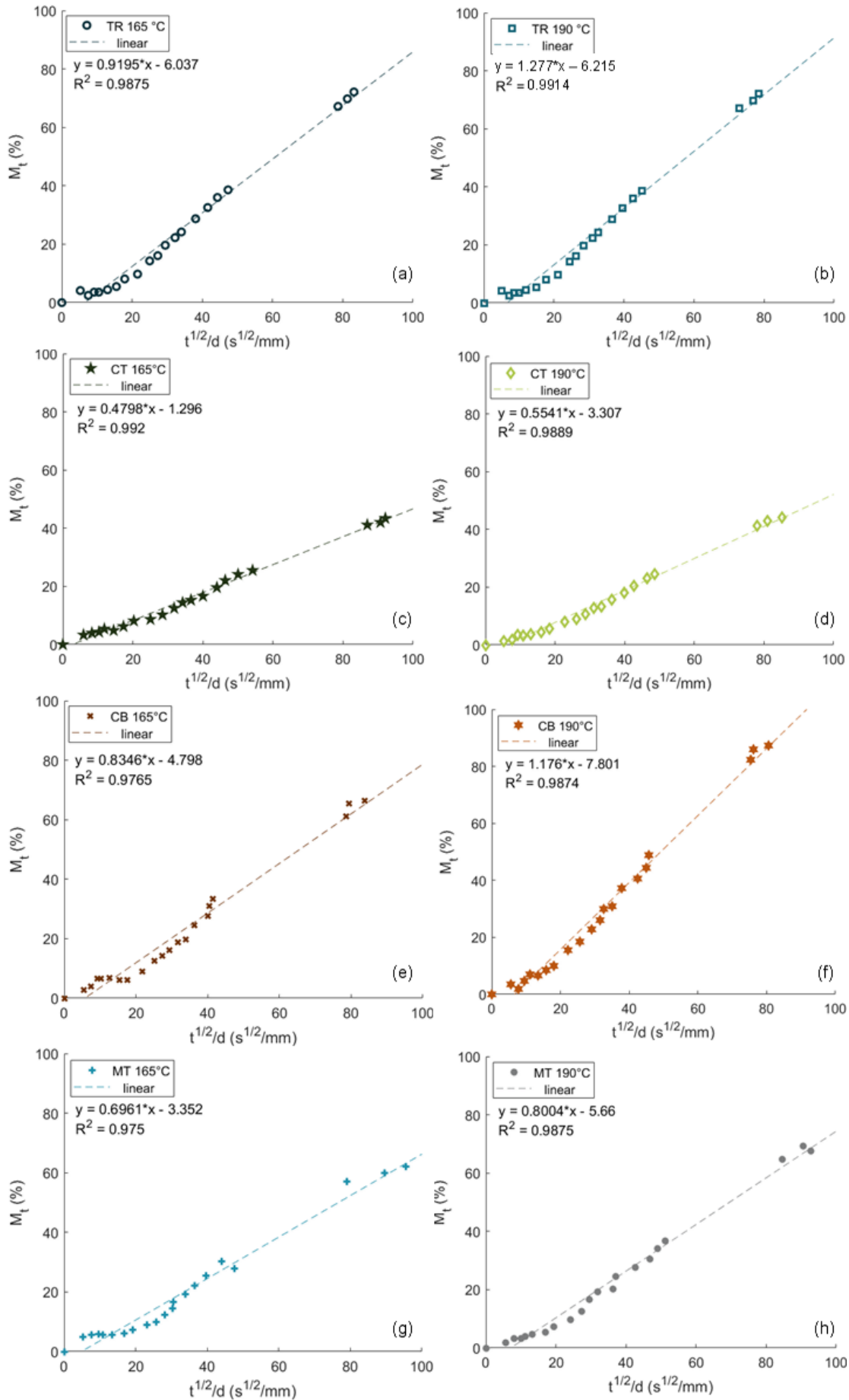
The solubility parameters of car and truck tyres have previously been quantified, and their similarity with that of bitumen has been explained to indicate that bitumen is a swelling agent for these rubbers (Artamendi & Khalid 2006). Provided that the degree of swelling of crumb rubber in bitumen is gradient from the surface to the core (Wang, Apostolidis et al. 2021), its dependence on the crumb rubber size is expected (Ren et al. 2021). To isolate these effects, the swelling of the 4 different types of rubber (TR, CT, CB and MT) at the 2 temperatures investigated (165 and 190 °C) was measured for thin films of approximately the same size, as outlined in Section 3.3.3. 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 TR followed by CB; then, MT was found to follow with CT having the lowest rate of swelling.

These findings relate to the density of the chain cross-links in each of the rubber compounds and the mobility of the molecular chains under each temperature condition, which determine the free volume available to absorb the bitumen's light components (Artamendi & Khalid 2006).

The impact of bitumen viscosity at 165 and 190 °C must also not be neglected. Artamendi and Khalid (2006) explained that the decreased bitumen viscosity at 190 °C when compared to that at 165 °C can promote its diffusivity in the rubber structure. As the same bitumen was used in all cases, however, the comparatively different swelling behaviours observed in Figure 4.1 are attributed solely to the differences in the molecular structure of the types of rubber investigated in this research.

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

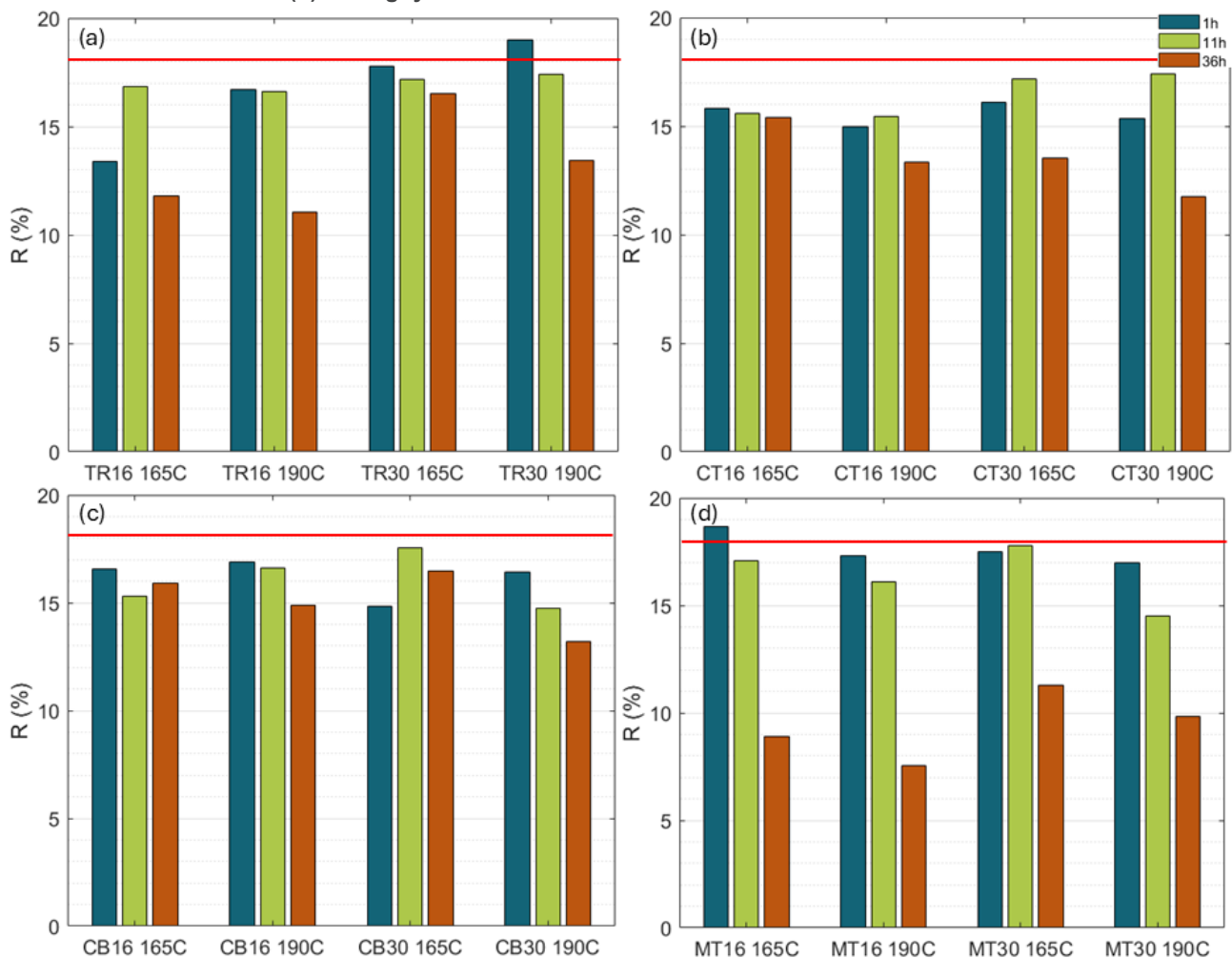


4.2.2 Crumb Rubber Dissolution

The dissolution of crumb rubber into bitumen is the result of chain disentanglement and scission within the rubber's main chain (Wang et al. 2017). This is a non-reversible process. The method described in Section 3.3.7 measures the mass of crumb rubber retained in a 75 μm mesh thimble. As such, any portion of the crumb rubber particles that is smaller than that constitutes the dissolved portion of the rubber.

The crumb rubber content in the CRMBs (undissolved portion) following blending at 165 and 190 $^{\circ}\text{C}$ for 1, 11 and 36 hours was extracted following the method described in Section 3.2.2 and calculated using Equation 2. The results are presented in Figure 4.2.

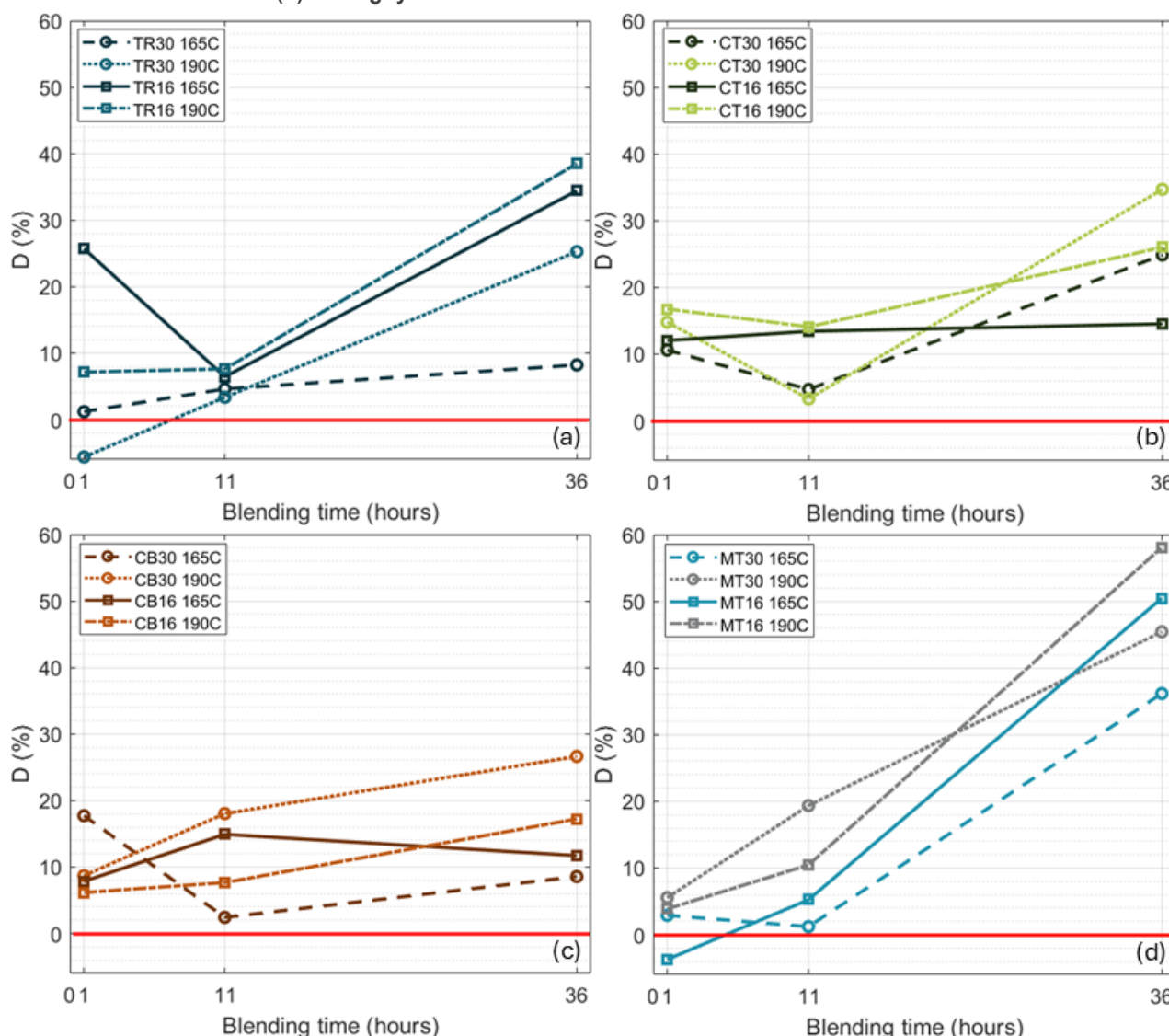
Figure 4.2: Crumb rubber content (R) for (a) truck tyre-derived crumb rubber-modified binders, (b) car tyre-derived crumb rubber-modified binders, (c) conveyor belt-derived crumb rubber-modified binders and (d) mining tyre-derived crumb rubber-modified binders



Note: Red line denotes the crumb rubber content before blending.

The crumb rubber content, as shown in Figure 4.2, was then used to calculate the dissolution of each of the crumb rubber types. These results are presented in Figure 4.3.

Figure 4.3: Per cent dissolution (D) 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



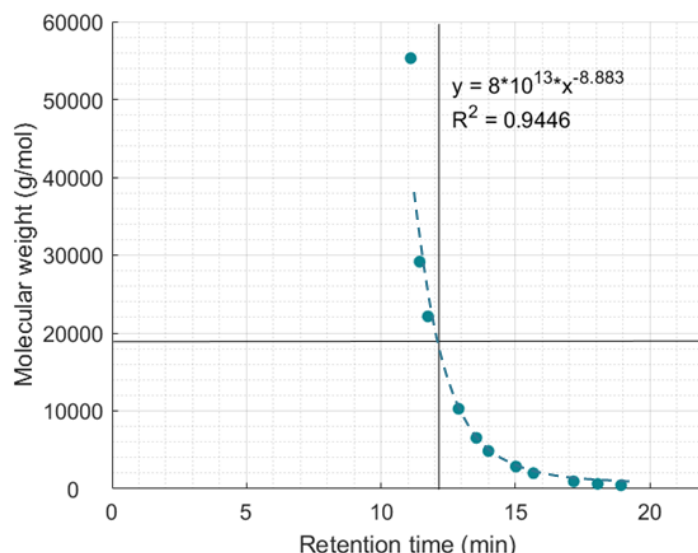
Note: Red line is at 0.

Overall, the content of rubber dissolved during blending appeared to increase after 36 hours of blending. A closer examination, however, revealed that these trends are not consistently progressive from 1 to 11 and from 11 to 36 hours of blending. In addition, the crumb rubber content after digestion for TR30_190C_1h and MT16_165C_1h was found to be greater than the 18 wt.% introduced in the binder. Consequently, the calculated digestion presents negative results. As this is not physically possible, the phenomenon is attributed to subsampling accuracy and overall binder handling consistency. It is worth noting here that the method analyses approximately 10 g of a sample deriving from a 4 L tin.

4.2.3 High Performance Liquid Chromatography – Gel Permeation Chromatography

From Section 2.2.4, it is understood that the dissolution of the crumb rubber components depends on their concentration in the rubber. 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 for the PS standards of Table 3.7 using the power fit equation in Figure 4.4.

Figure 4.4: PS standards with power law data fit



The base C170 was also analysed. The results are presented in Figure 4.5. Figure 4.5 denotes the calculated retention time where the molecular weight of the samples is above 19,000 g/mol, where an increase would suggest the dissolution of the crumb rubbers' polymer components into the bitumen. It also shows the regions between 19,000 and 3,000 g/mol, where asphaltenes are found, and the maltenes below 3,000 g/mol. All results were normalised to 100% at their maximum intensity.

Figure 4.5: Molecular weight distribution curve for baseline C170

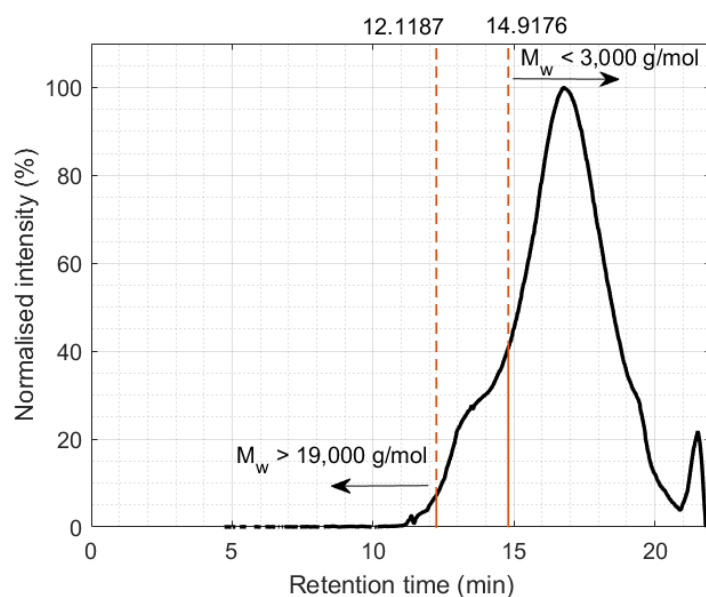


Figure 4.6 presents the curves for retention time vs normalised intensity for binders modified with TR-derived crumb rubber. Overall, an increase in the area under the curve for retention times below 12 minutes can be observed with an increase in blending time.

Interestingly, though, for 36 hours of blending, this increase is more pronounced in Figure 4.6 (d), where binders have been modified with S16 particles and blended at 190 °C (quantified in Figure 4.7). According to Section 2.2, S30 crumb rubber particles would be expected to have progressed through the digestion stages towards dissolution faster than the S16 crumb rubber particles, which means we would expect to observe TR30_190C_36h to have greater apparent polymer content concentration when compared to TR16_190C_36h. It is possible, however, that, as also suggested by Huang et al. (2017), the dissolved portion of S30 particles has been subjected to subsequent chain scission decreasing the measured polymer content.

Figure 4.6: Molecular weight distribution curves for truck tyre-derived crumb rubber binders: (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C

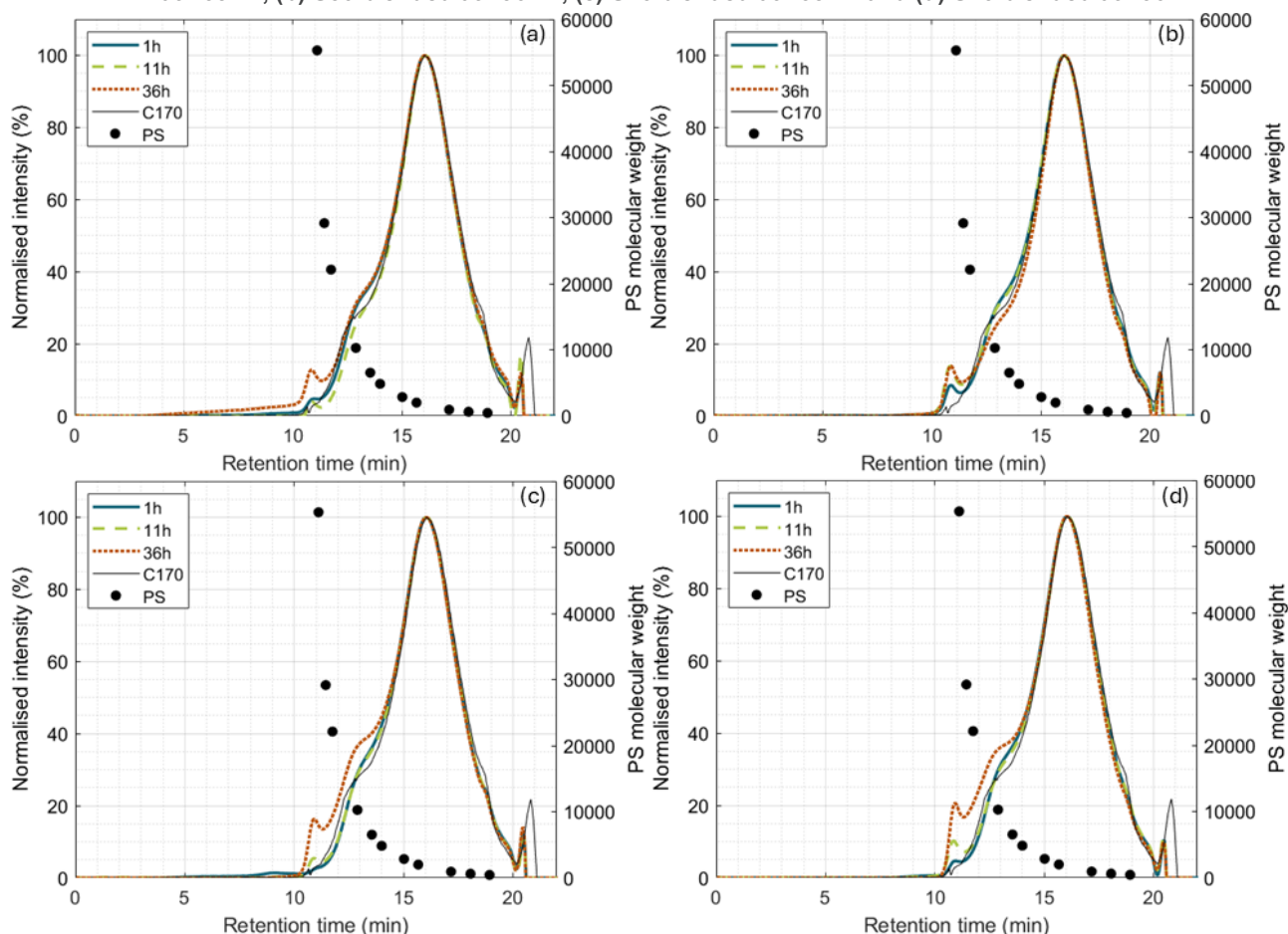


Figure 4.7 shows the concentrations of each of the components (polymer, apparent asphaltenes and maltenes) in the melt-extracted binders of TR-derived CRMBs as measured based on the corresponding areas under the curves of Figure 4.6.

For TR16_190C and TR30_190C, an increase in the polymer content was observed with blending time from 1 to 11 and 36 hours. TR16_165C_11h and TR30_165C_11h, however, were found to have a 0.2 and 1.5% lower concentration of polymer content when compared to TR16_165C_1h and TR30_165C_1h, respectively. It is worth remembering the binder blending process, where 2 different blends were prepared for each binder with the same crumb rubber type and size blended at each temperature: one for 1, 2 and 4 hours and one for 11, 24 and 36 hours. As the samples analysed through this process were part of these 4 L tins, it is noted that blends manufactured at 1 and 11 hours were from 2 different tins. Therefore, it might be suggested that a decrease in the polymer content such as that observed between TR16_165C_1h and TR16_165C_11h and TR30_165C_1h and TR30_165C_11h would not have been possible had the binders been blended as part of the same batch. This suggestion, however, is only being made here due to the relatively low blending temperature of 165 °C, especially as the same is not observed for their counterparts blended at 190 °C.

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 4.7: Concentration of components based on their molecular weight for truck tyre-derived crumb rubber-modified binders

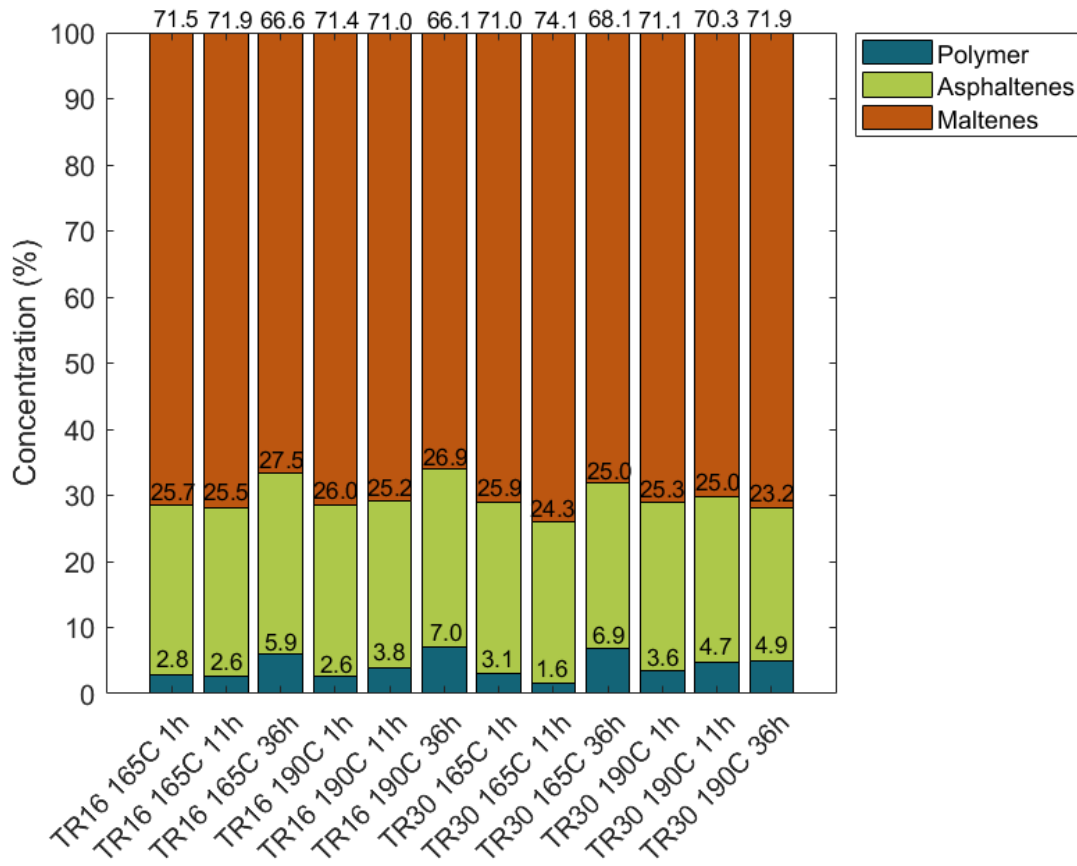


Figure 4.8 presents the retention time vs normalised intensity curves for CT-derived CRMBs. As in Figure 4.6, Figure 4.8 also shows that the concentration of higher molecular weight components in the melt-extracted binder was greater than that of the unmodified C170. Unlike what was observed for TR-derived CRMBs, though, the concentration of high molecular weight chains appears greater for binders modified with S30 CT-derived crumb rubber particles.

Figure 4.8: Molecular weight distribution curves for car tyre-derived crumb rubber binders; (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C

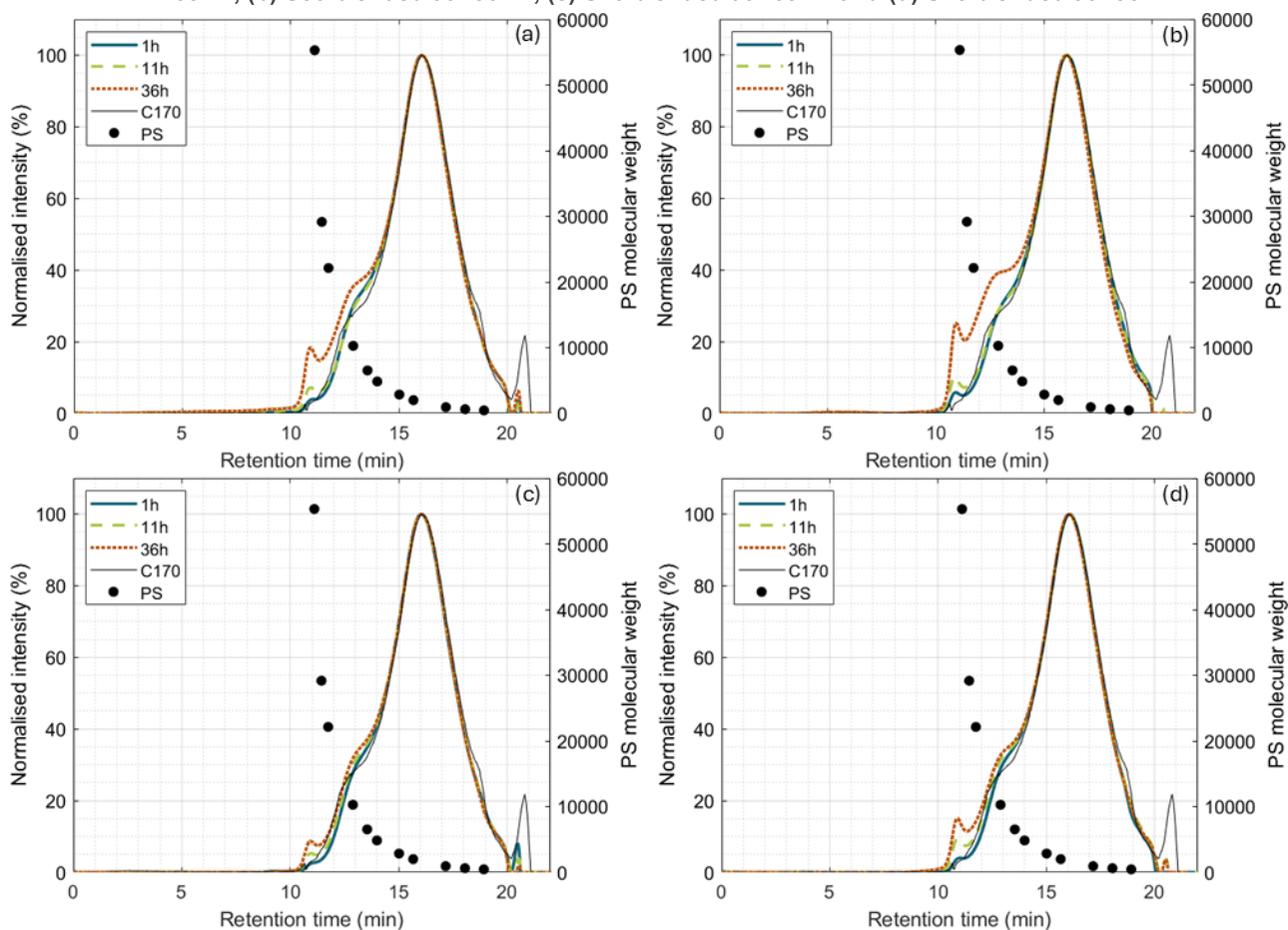


Figure 4.9 shows the measured distribution of constituents in the melt-extracted CT-derived CRMBs. For CT-derived CRMBs, the polymer content increased with time for all samples. The polymer content was also found to be comparatively greater for S30 CRMBs and for CRMBs blended at 190 °C. These observations align with the expectations set by Section 2.2.

Figure 4.9: Concentration of components based on their molecular weight for car tyre-derived crumb rubber-modified binders

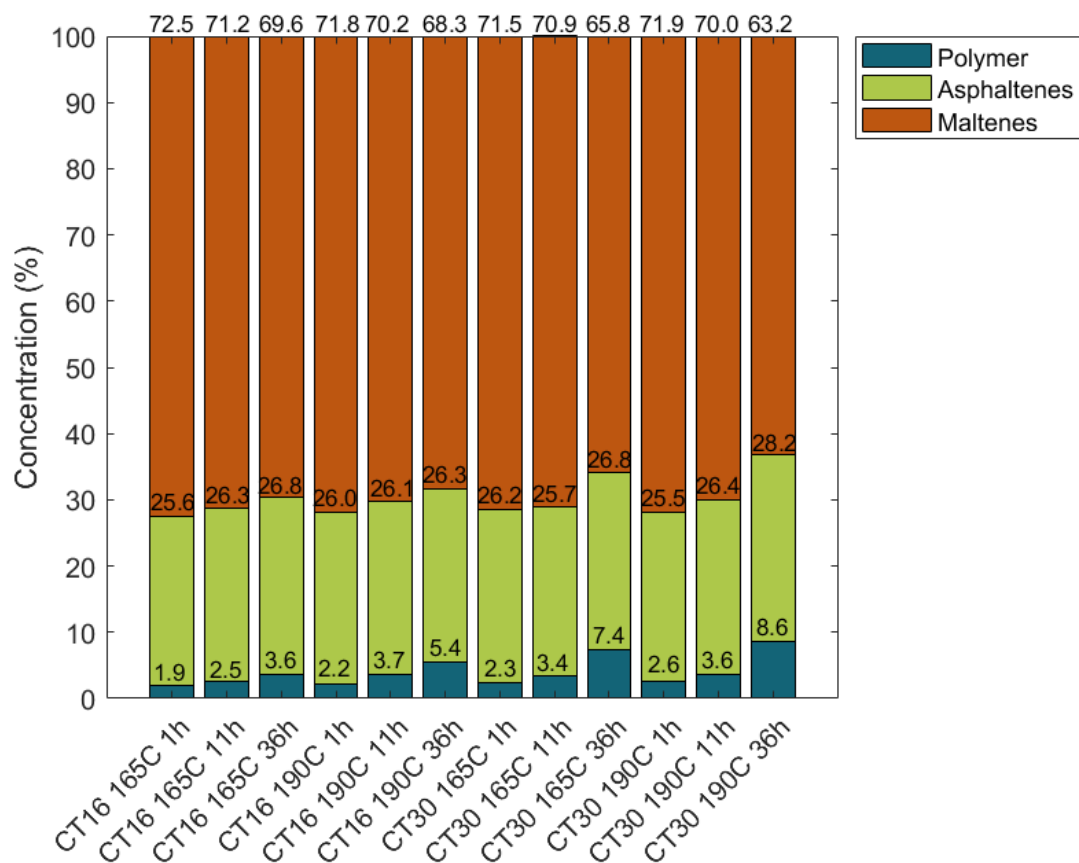
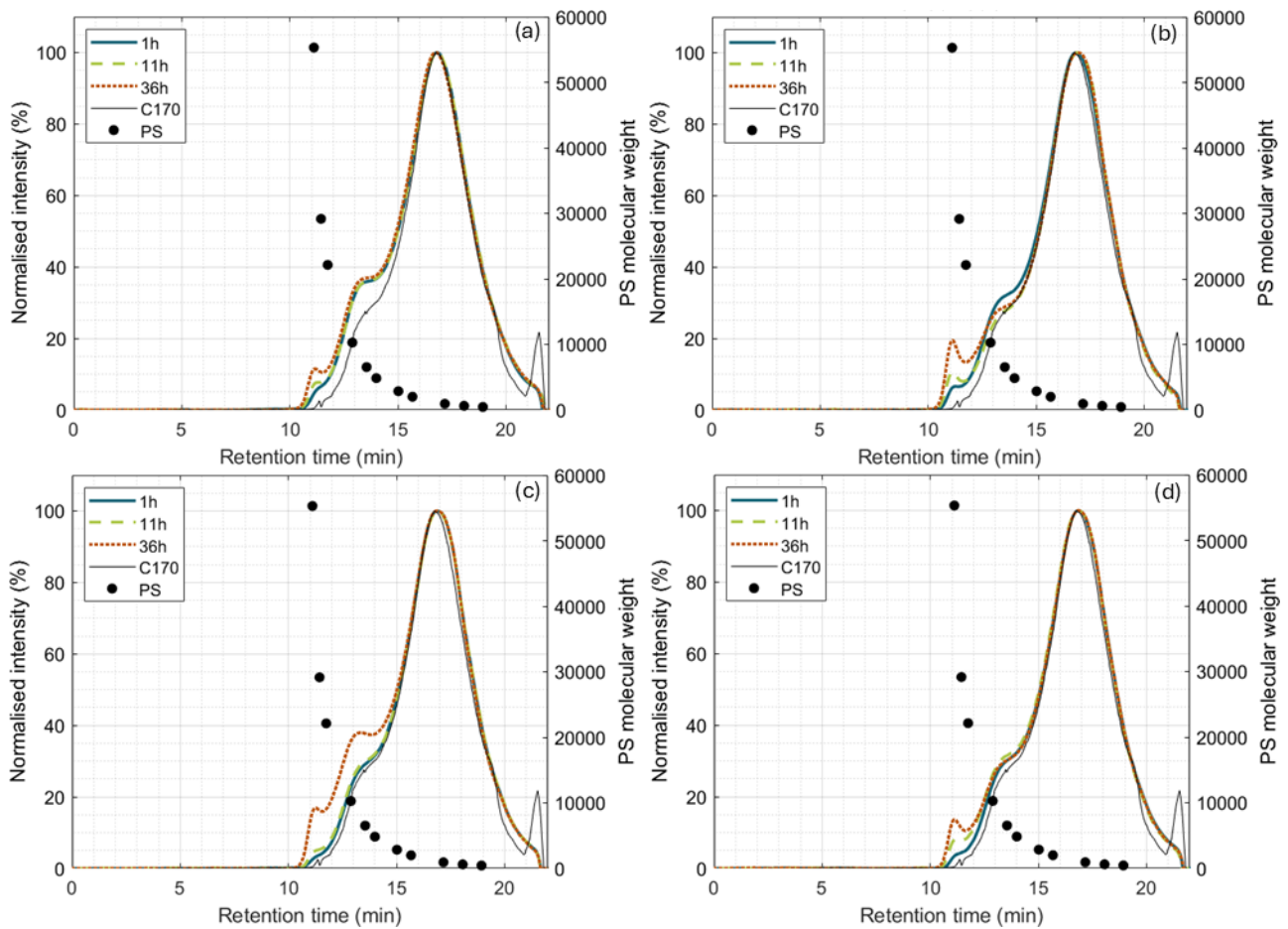


Figure 4.10 shows the retention time vs normalised intensity plots for CB-derived CRMBs. As also observed by the TR- and CT-derived CRMBs, the polymer content of CB-derived CRMBs was found to have increased when compared to the unmodified C170.

In Figure 4.10 (b), the apparent asphaltenes appear to be lower for CB30_190C_11h and CB30_190C_36h when compared to CB30_190C_1h. However, the content of asphaltenes is not anticipated to change as a result of the digestion process. The phenomenon may instead be attributed to the oxidation of bitumen, which could result in the increase of molecular weight due to oxygen uptake (Ren et al. 2021). Furthermore, as it is unlikely that oxidation decreased with blending time, remembering that CB30_190C_1h was blended as part of a different 4 L tin to CB30_190C_11h and CB30_190C_36h is once again important.

Figure 4.10 (b), therefore, suggests that the tin that produced CB30_190C_1h, CB30_190C_2h and CB30_190C_4h was subject to more extensive oxidation when compared to the tin that produced CB30_190C_11h, CB30_190C_24h and CB30_190C_36h. Daly et al. (2019) found that an increase in the apparent asphaltene fraction could also be the result of crumb rubber particle dissolution of lower molecular weight. This explanation was not considered probable in this case, however, as it would have translated to similar findings in CB30_190C_11h and CB30_190C_36h.

Figure 4.10: Molecular weight distribution curves for conveyor belt–derived crumb rubber binders: (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C



The concentration of the different binder constituents for CB-derived CRMBs are presented in Figure 4.11. Similarly to the CT-derived CRMBs in Figure 4.9, 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 TR-derived CRMBs.

Figure 4.11: Concentration of components based on their molecular weight for conveyor belt–derived crumb rubber-modified binders

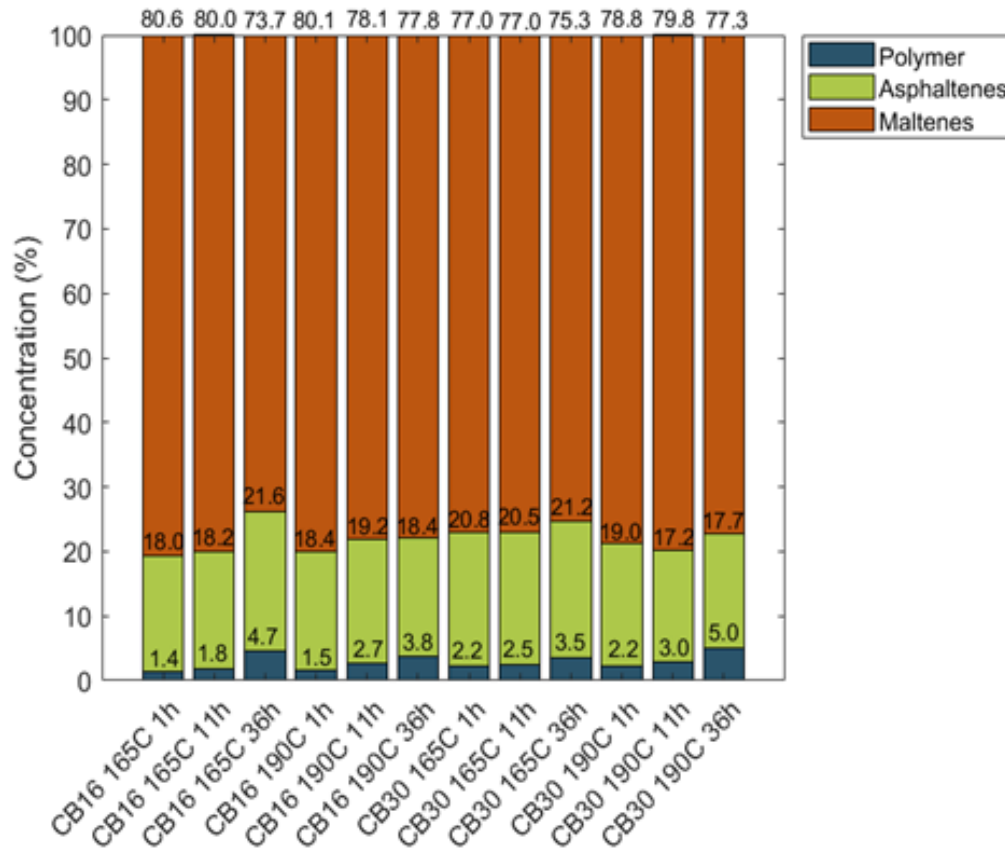


Figure 4.12 presents plots of normalised intensity against retention time for MT-derived CRMBs. The peak in intensity between 21 and 22 min of retention time was attributed to a testing artifact and was not represented in the calculations of maltene concentration shown in Figure 4.13. Similarly to what was observed by the rest of the CRMBs of this research, the polymer content of all MT-derived CRMBs was found to be greater than that of the C170 bitumen.

As was also observed for CB-derived CRMBs, MT16_190C_1h, MT30_165C_11h, MT30_190C_1h and MT30_190C_11h show an increase in the apparent asphaltenes when compared to the respective binders blended for 36 hours. As previously explained, this is likely due to different levels of oxidation experienced by the binders in the different tins during blending. However, MT30_190C_11h exhibited a greater content of apparent asphaltenes when compared to MT30_190C_36h (blends produced in the same tin). In this case, therefore, it may be suggested that a portion of those apparent asphaltenes is a result of rubber chain scission with chains smaller than 19,000 g/mol, as was also previously observed by Daly et al. (2019).

Figure 4.12: Molecular weight distribution curves for mining tyre-derived crumb rubber binders: (a) S30 blended at 165 °C, (b) S30 blended at 190 °C, (c) S16 blended at 165 °C and (d) S16 blended at 190 °C

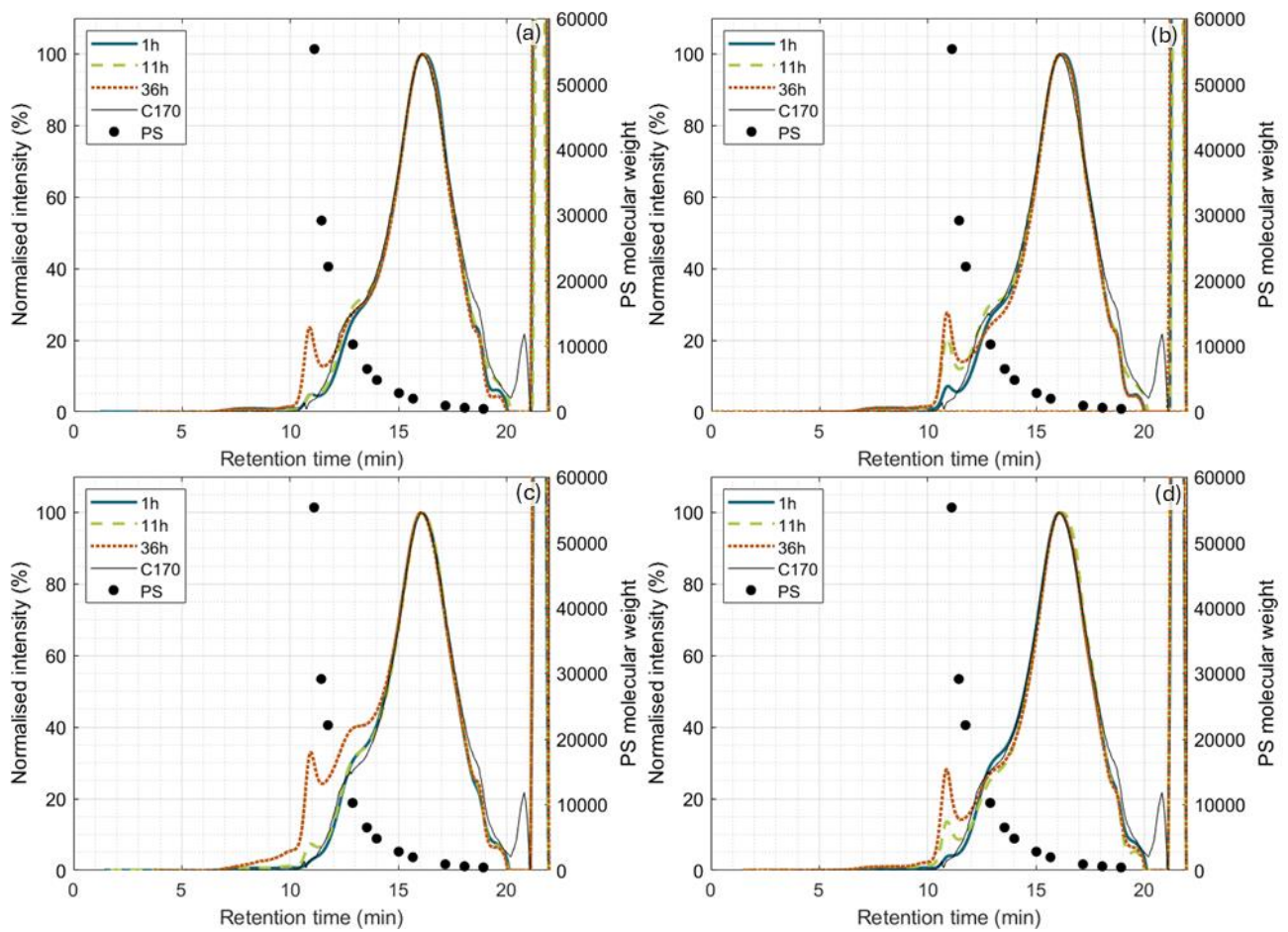
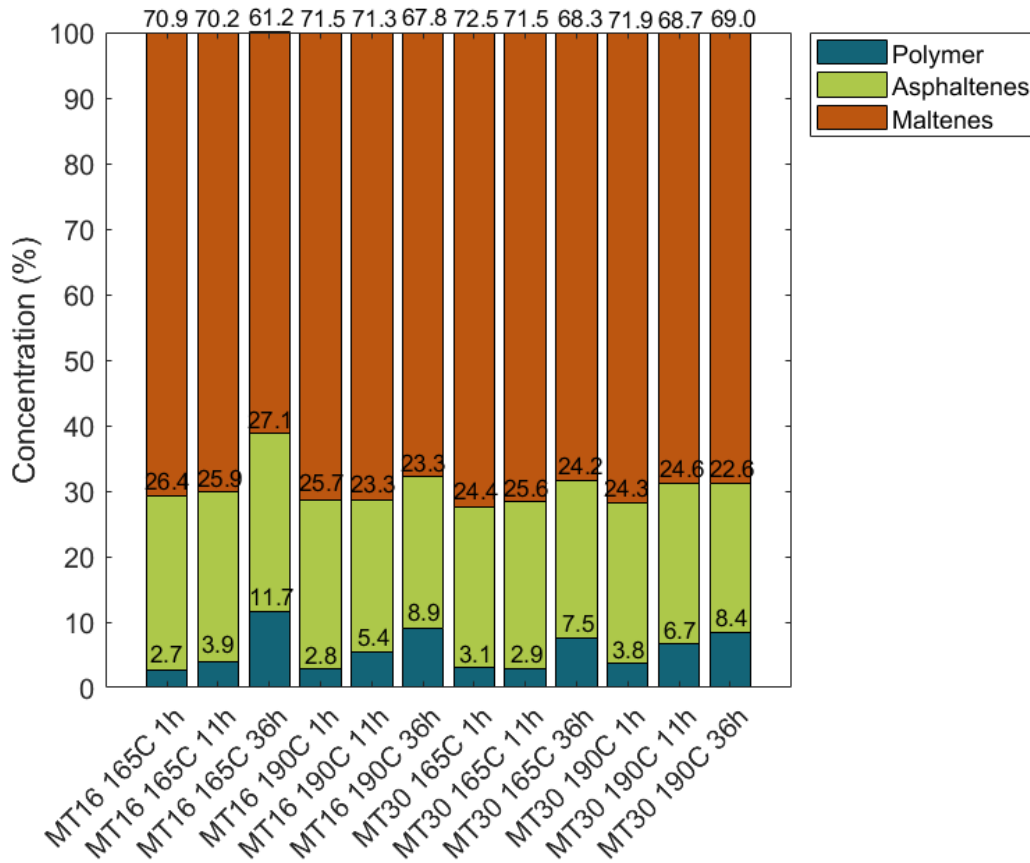


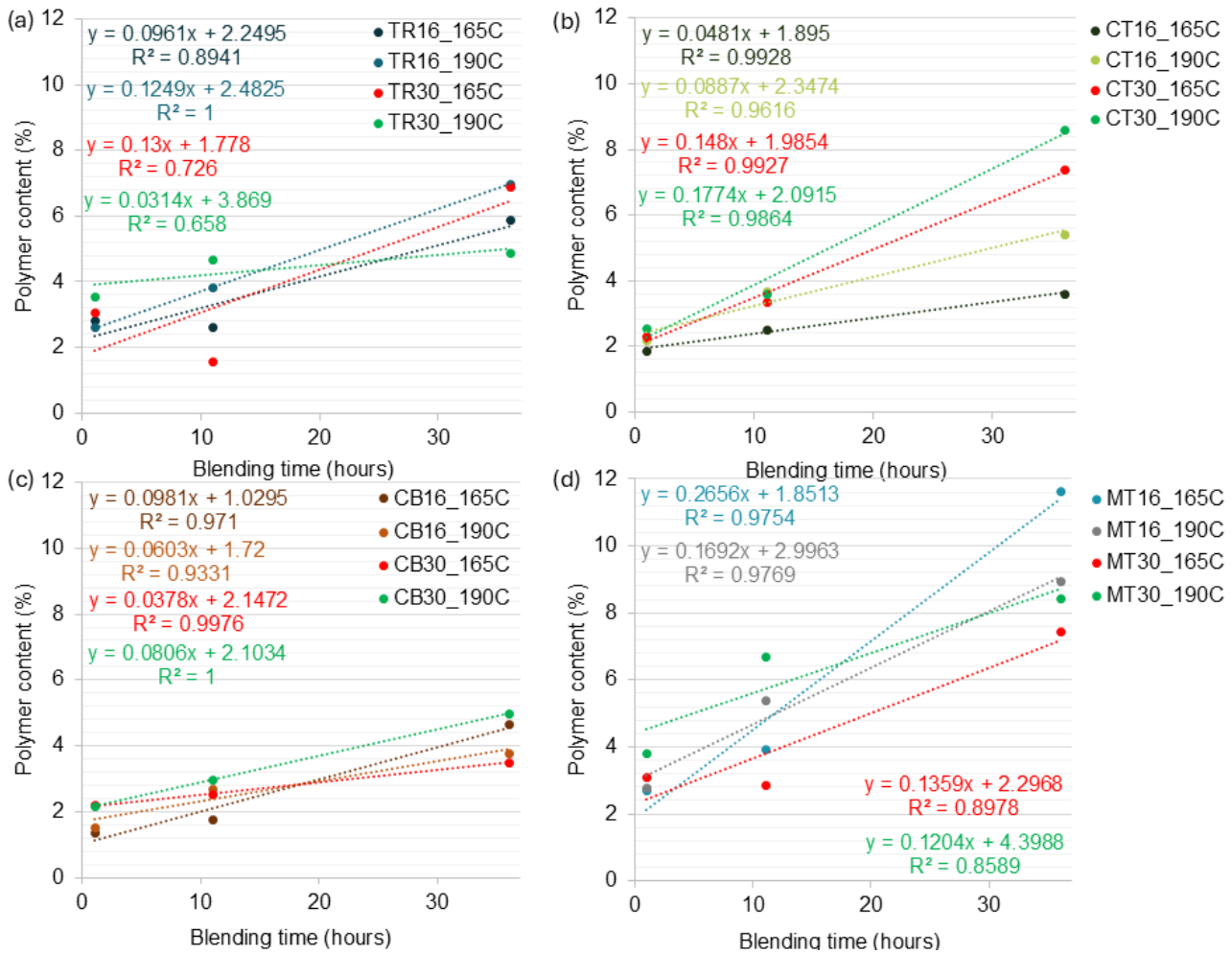
Figure 4.13 presents the measured concentration of constituents of the liquid phase for MT-derived CRMBs. The overall trends observed are in line with all other CRMBs in this research, whereby the polymer content increased with an increase in blending time and more so between 11 and 36 hours of blending. The greatest polymer content was measured for MT16_165C_36h. This suggests that some rubber chain scission following dissolution is probable for MT16_190C_36h, MT30_165C_36h and MT30_190C_36h.

Figure 4.13: Concentration of components based on their molecular weight for mining tyre-derived crumb rubber-modified binders



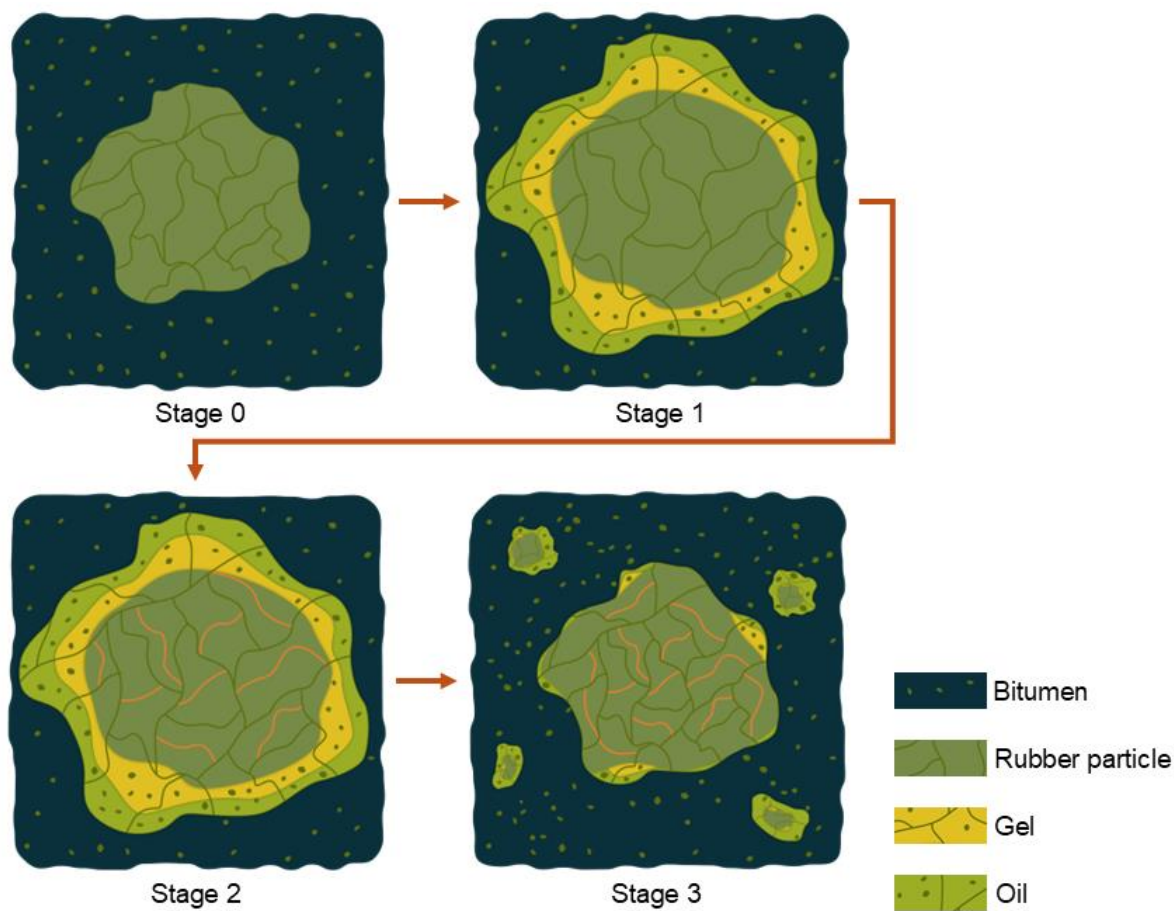
Overall, the greatest concentration for dissolved polymer was measured in the liquid phase of MT-derived CRMBs followed by TR-, CT- and, lastly, CB-derived CRMBs. Figure 4.14 presents the polymer content changes with increase in blending time and corresponding linear fits. The R^2 values were generally found to be above 0.95, with a few exceptions.

Figure 4.14: Increase of polymer content with blending time for (a) TR-derived crumb rubber, (b) CT-derived crumb rubber, (c) CB-derived crumb rubber and (d) MT-derived crumb rubber



According to Section 2.2, digestion of crumb rubber in bitumen is faster for smaller particles and at higher temperatures. However, contrary to this expectation, according to Figure 4.14 (d), sample MT16_165C was found to experience the greatest increase in polymer content in the liquid phase with increasing blending time when compared the rest of the MT-derived CRMBs. Swelling is a process that occurs from the outer layers of the crumb rubber particles and progresses inwards (Wang, Apostolidis et al. 2021). According to the findings of Section 4.2.1, and in line with the literature, swelling of MT-derived rubber is slower at 165 when compared to 190 °C. It may, therefore, be hypothesised that this slower swelling progression potentially allows for cross-linking among the chains towards the inner layers of crumb rubber particles. The potentially resultant increase in the number of cross-links towards the crumb rubber particle centre would, in turn, hinder further progressive swelling, achieving a comparatively earlier swelling equilibrium, as suggested by Artamendi and Khalid (2006). Once those outer layers have reached their swelling equilibrium, further exposure to bitumen at elevated temperatures results in their dissolution, which would support this observed behaviour. This hypothesised mechanism is schematically illustrated in Figure 4.15.

Figure 4.15: Schematic illustration of cross-linking potentially occurring due to slow digestion and the presence of elevated temperature

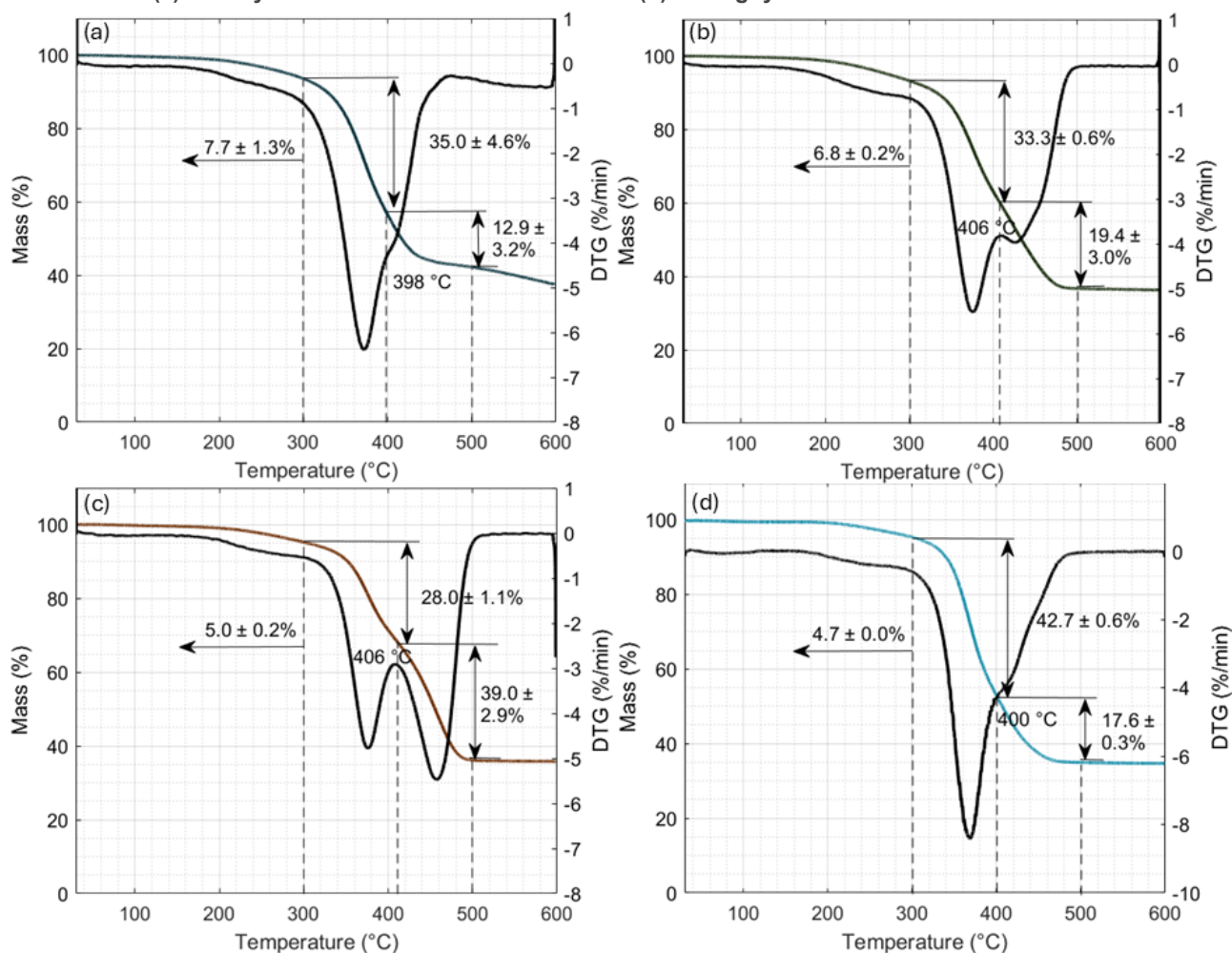


4.2.4 Thermogravimetric Analysis

TGA was used to quantify the content of moisture and oils, NR, SR and other polymers, and carbon black and fillers of the different types of undigested and digested crumb rubber. The results are detailed in Table D.1. Table D.2 to Table D.9 present a representative curve for all the digested crumb rubbers.

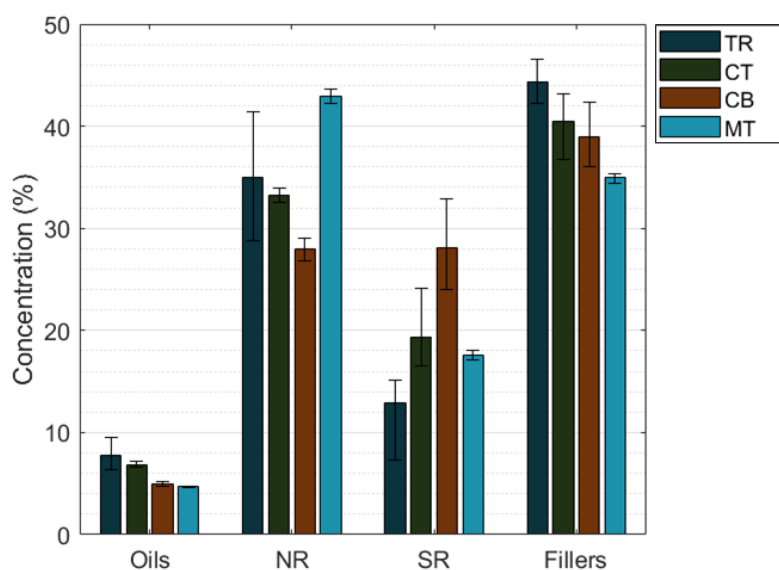
Representative TGA and DTG curves of the different types of as-received crumb rubber used in this research are presented in Figure 4.16. MT-derived rubber was found to have the greatest total rubber content at approximately 60%, followed by CB-derived rubber at approximately 56%, then CT-derived rubber at approximately 53% and, lastly, TR-derived rubber was found to have the lowest concentration of total rubber at approximately 48%. TR-derived rubber was, however, measured to have the lowest SR/NR ratio at 0.37, followed by MT-derived rubber at 0.41, then CT-derived rubber at 0.58 and, lastly, CB-derived rubber at 1.00.

Figure 4.16: TGA and DTG curves for (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



The mean concentration of each of the undigested crumb rubber constituents is presented in Figure 4.17.

Figure 4.17: Concentration of moisture and light oils, natural rubber, synthetic rubber and other polymers, and carbon black and fillers for all undigested crumb rubbers



Just as the digested crumb rubber was extracted from the binder using the Soxhlet method described in Section 3.2.2, as-received crumb rubber was also subjected to the extraction method, and the particles were then analysed through TGA to understand whether any of the observed changes could be purely attributed

to the digestion process or if the extraction method could also have an effect. The concentration of the different crumb rubber components of the extracted S16 and S30 particles is presented in Figure 4.18.

The concentration of oils was found to be impacted by the Soxhlet extraction for all types of rubber but, interestingly, not notably for MT16. On the other hand, the Soxhlet extraction process was found to impact the concentration of oils and NR in MT30 particles the most, when compared to the other crumb rubber types. These observations were reflected in the calculation of total rubber and SR/NR ratio for each of the Soxhlet extracted rubbers. The total rubber content for TR- and CB-derived rubbers following Soxhlet extraction was indistinguishable for S30 and S16 particles at approximately 56% for both; that of CT16 and CT30 presented a small difference at 51 and 54%, respectively, whereas that of MT16- and MT30-derived rubbers deviated notably at 59 and 47%, respectively.

Importantly, insights regarding the solubility of the different types of rubber in bitumen (predominantly by the aromatic component given its concentration in bitumen and its polarity, which is similar to that of toluene (Lesueur 2009)) can be gained from these findings. CB-derived rubbers were not found to be impacted by the extraction process with both the total rubber content and SR/NR ratio remaining unchanged at 56% and 1.00, respectively. For all other rubber types, the SR/NR ratio increased following the Soxhlet extraction process, except for TR30, for which it decreased. This suggests that, during blending, a portion of the NR is likely to dissolve in bitumen and more so for MT30 crumb rubber particles whose SR/NR ratio following the Soxhlet extraction process was measured to be 1.64.

Figure 4.18: Concentration of moisture and light oils, natural rubber, synthetic rubber and other polymers, and carbon black and fillers for all Soxhlet extracted (a) S16 and (b) S30 crumb rubbers

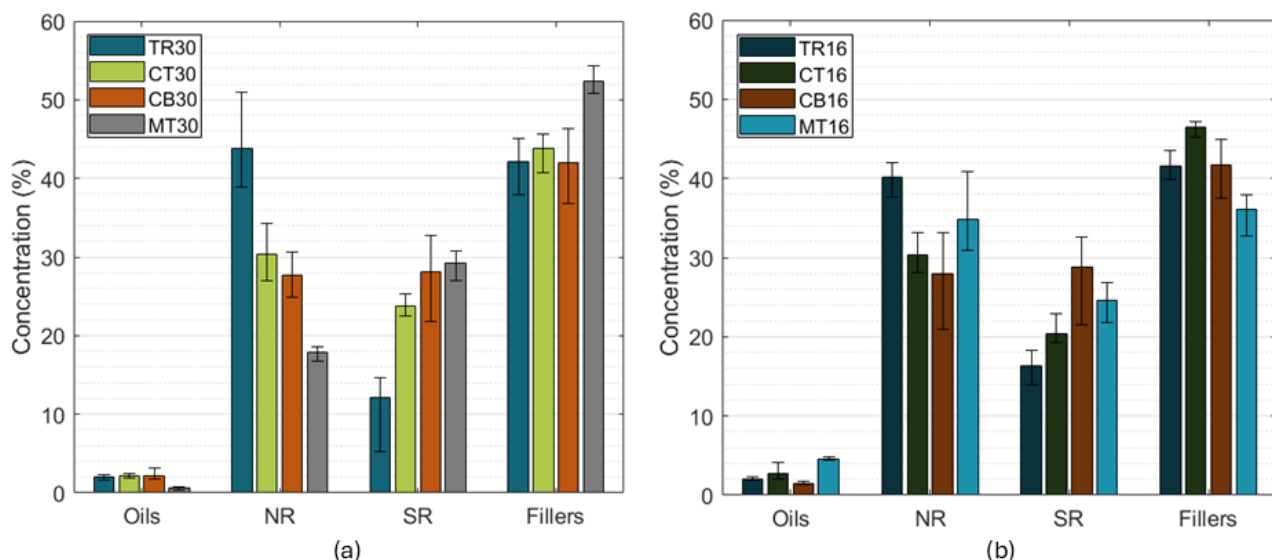


Figure 4.19 shows the mean concentration of each of the TR-derived crumb rubber constituents following blending for 1, 11 and 36 hours. The measured concentration of moisture and light oils, as shown in Figure 4.19 (a), and that of SR and other polymers, shown in Figure 4.19 (c), do not appear to follow any distinct trends relating to the crumb rubber size or blending parameters. The concentration of NR shown in Figure 4.19 (b), however, appears to decrease with blending time. The NR concentration following blending at 190 °C is lower than that following blending at 165 °C. In addition, in line with the expectations regarding NR solubility in bitumen set by the Soxhlet extraction findings, the NR content of S16 was found to be comparatively greater than that of the S30 particles.

Figure 4.19: Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for truck tyre-derived crumb rubber following blending at 165 and 190 °C for 1, 11, and 36 hours

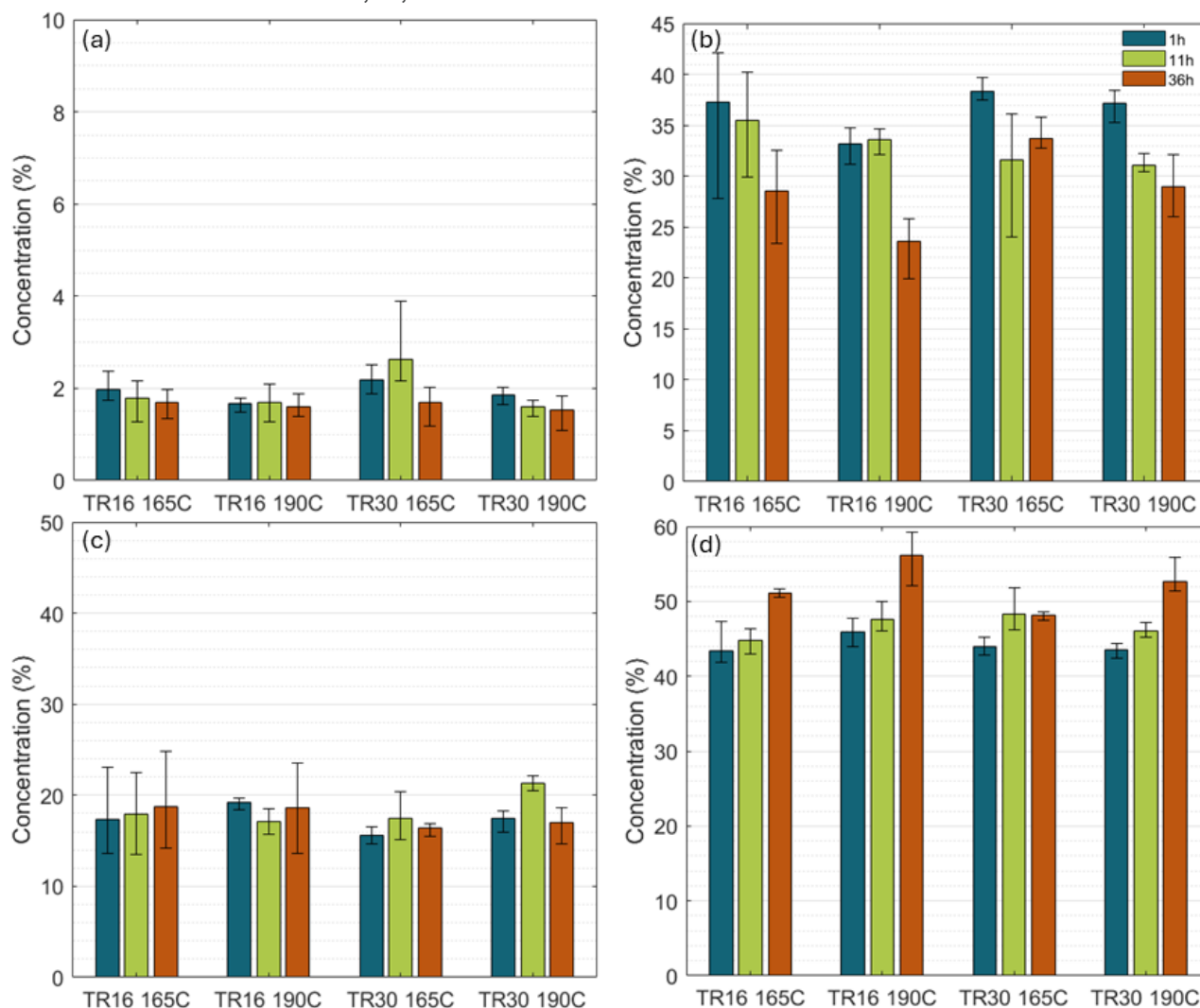


Figure 4.20 (a) shows the total content of rubber (NR plus SR and other polymers) following digestion compared to the mean total rubber content measured in the undigested TR-derived crumb rubber. Theoretically, the total rubber content for digested crumb rubber would be expected to be lower than that of the undigested crumb rubber. However, the findings presented in Figure 4.20 (a) could be attributed to the gradation of crumb rubber particles introduced during blending (particles of different size are expected to progress through the digestion stages at different times) and the expected variability in the composition of the EoL tyres recycled through this process. The impact of mass uptake due to oxidation was also considered here; however, carbonyl groups are expected to typically degrade at temperatures below the range where NR and SR are quantified.

Figure 4.20 (b) shows the ratio between SR and NR in the digested and undigested TR-derived crumb rubber. From Section 2.2, it would be expected that the SR/NR ratio would increase with blending time as the concentration of NR is expected to decrease due to dissolution. As noted above, the deviation from this expectation could be attributed to crumb rubber particle gradation and variability in tyre composition.

Figure 4.20: (a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted truck tyre-derived crumb rubber following blending

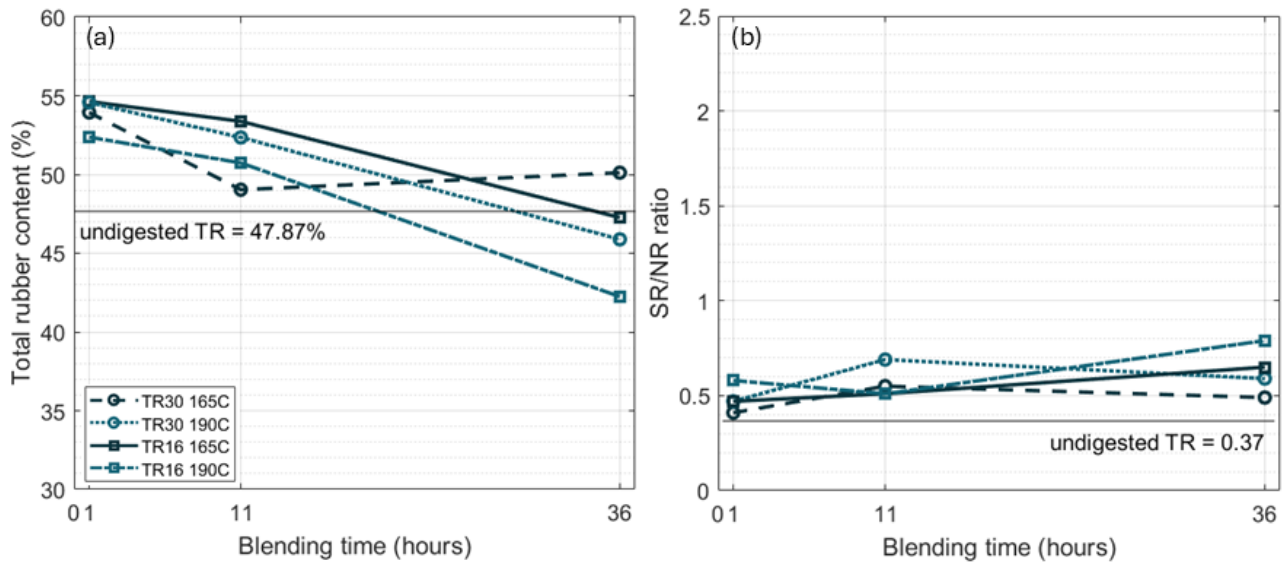


Figure 4.21 shows the concentration of CT-derived crumb rubber constituents following their digestion in bitumen for 1, 11 and 36 hours. The concentration of moisture and light oils and NR appear to generally decrease with blending time particularly following blending for 36 hours and more so for CT30-derived binders when compared to their CT16-derived counterparts. This observation is in line with the expectation set by the post-Soxhlet extraction TGA results.

Figure 4.21: Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for car tyre-derived crumb rubber following blending at 165 and 190 °C for 1, 11 and 36 hours

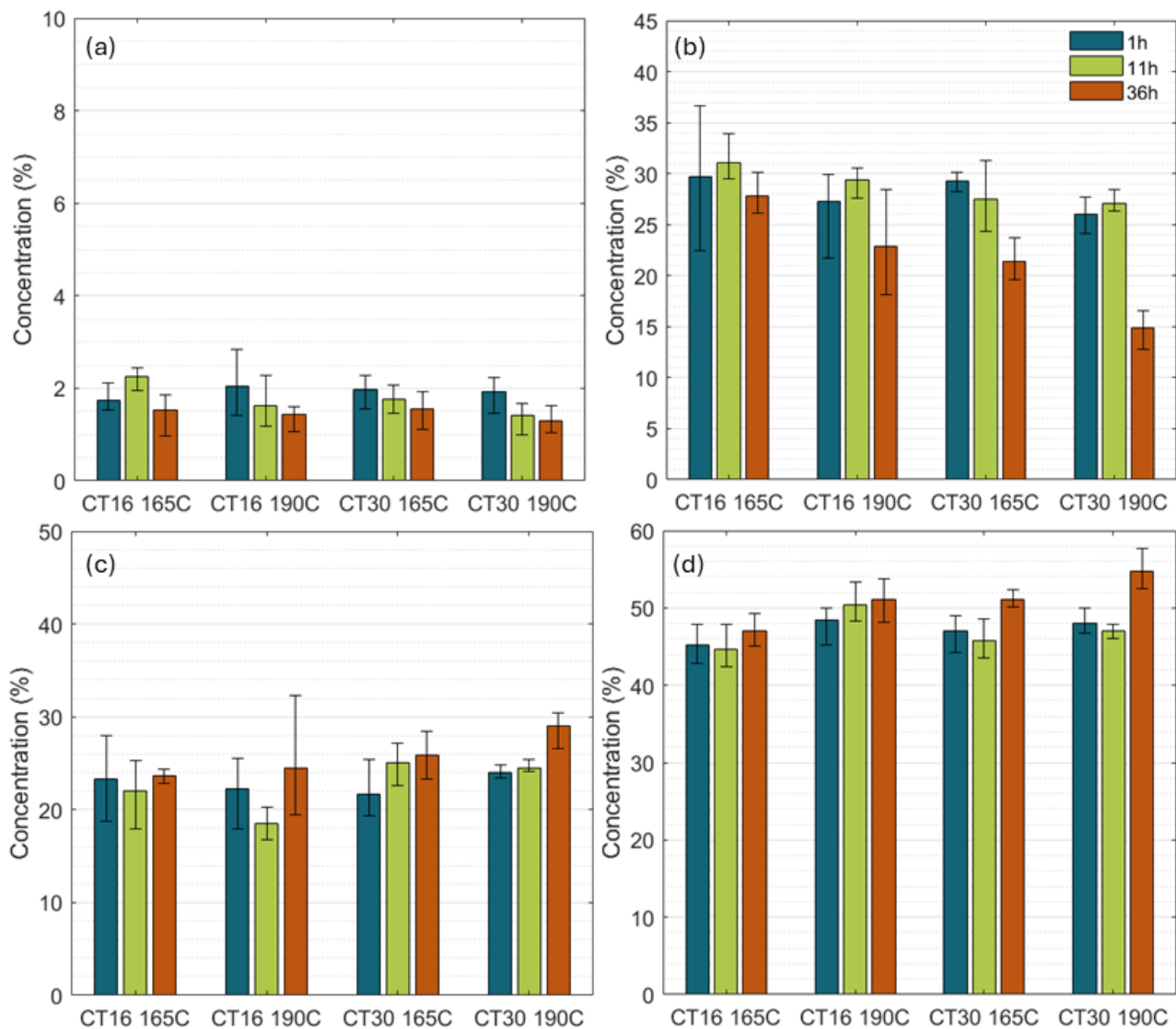


Figure 4.22 (a) shows the mean concentration of total rubber content in digested CT-derived crumb rubber compared to undigested CT-derived crumb rubber and Figure 4.22 (b) shows the ratio of SR over NR compared to that of undigested CT-derived crumb rubber. As also suggested by the findings of Soxhlet extracted CT-derived particles, these trends were more pronounced for the S30 particles and, as expected, even more so following blending at 190 °C for 36 hours.

Figure 4.22: (a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted car tyre-derived crumb rubber following blending

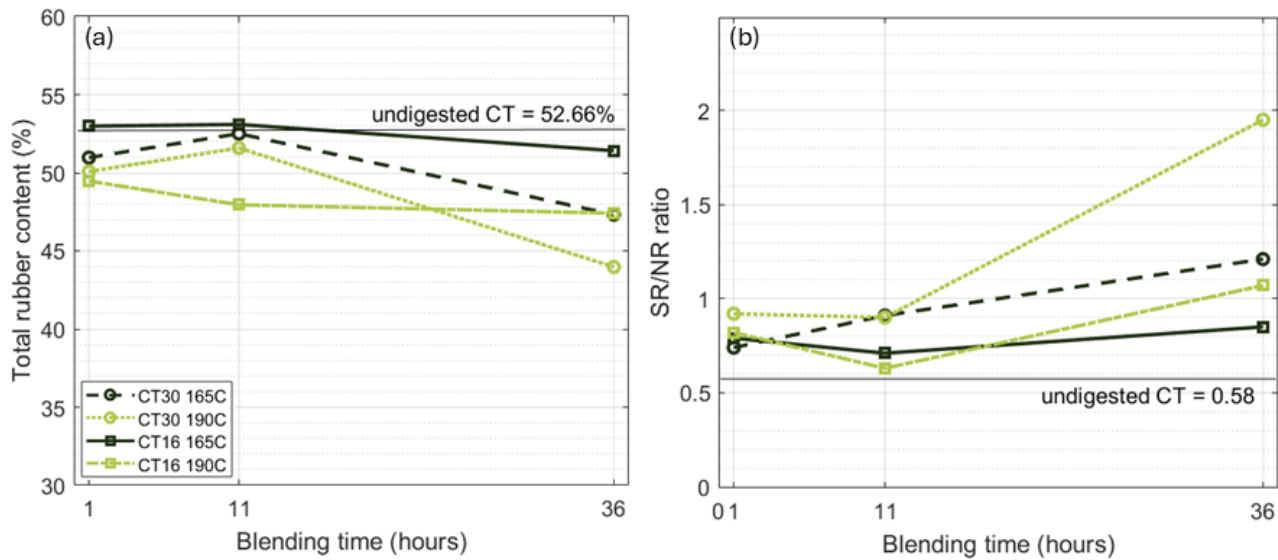


Figure 4.23 shows the mean concentration of each of the CB-derived crumb rubber constituents following digestion with error bars. The mean concentration of moisture and light oils in Figure 4.23 (a) and that of SR and other polymers in Figure 4.23 (c) were not found to follow any distinct trends. Their contents were found to be comparable irrespective of crumb rubber size and blending parameters. The concentration of NR in Figure 4.23 (b), however, can be seen to overall decrease with blending time. This suggests that NR from the crumb rubber has dissolved into the bitumen as blending progressed. At the same time, the concentration of carbon black and fillers can be seen to increase with blending time in Figure 4.23 (d). This increase is attributed to the decreased concentration of the other constituents, rather than a change in the concentration of carbon black and fillers as a result of the blending process.

Figure 4.23: Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for conveyor belt–derived crumb rubber following blending at 165 and 190 °C for 1, 11 and 36 hours

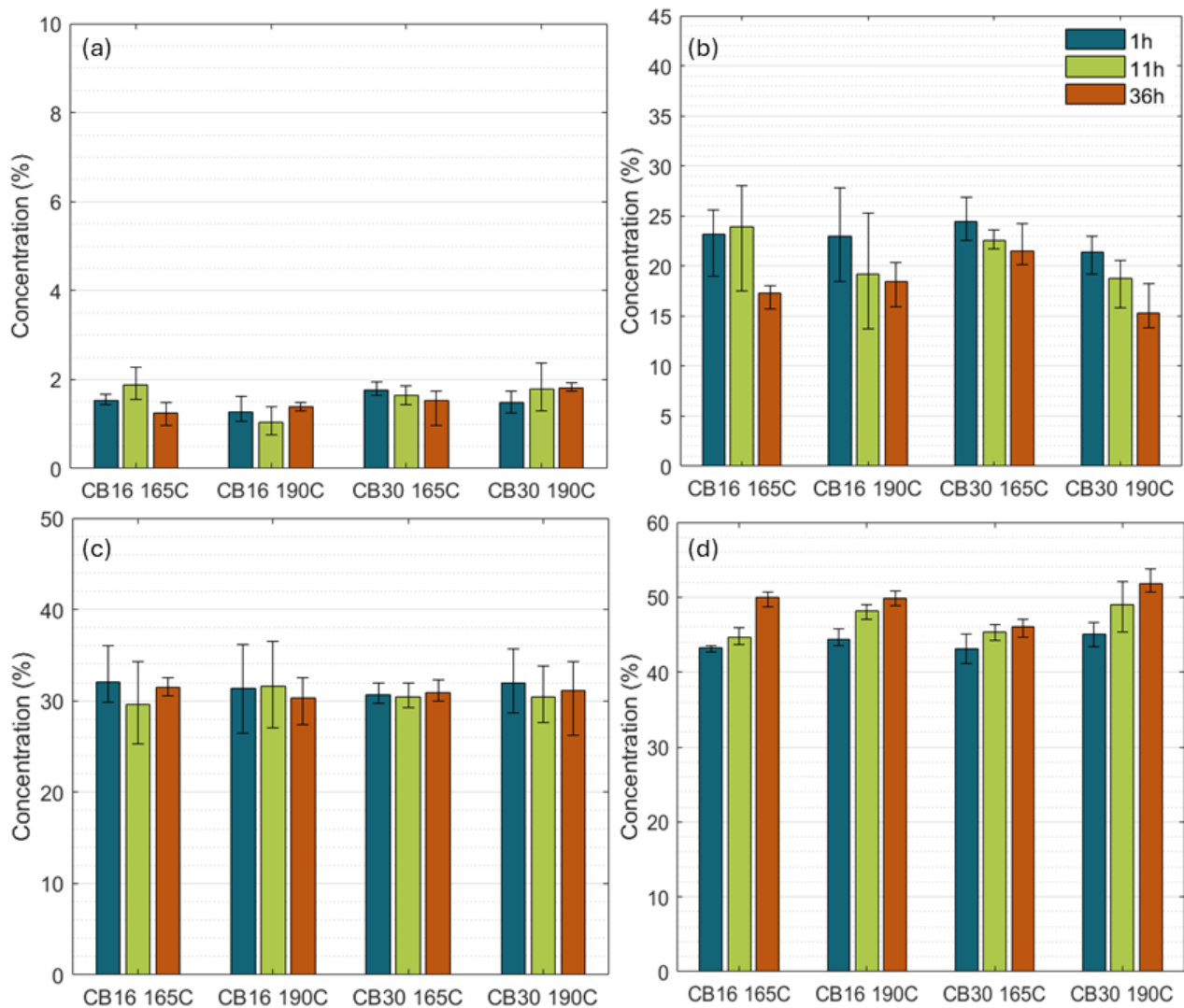


Figure 4.24 (a) shows that the total rubber content decreased in all digested CB-derived crumb rubber particles, which in all cases, was below the total rubber content of the undigested particles. It is also evident that this decrease became more pronounced as blending time increased. In addition, crumb rubber particles blended at 165 °C were found to maintain a comparatively greater concentration of rubber content after the digestion process, except for CB16_165C, which was found to have the same rubber content as CB16_190C.

Figure 4.24 (b) presents the ratio of SR and other polymer content over the content of NR. This ratio was found to generally increase with blending time, suggesting that the dissolution of NR is favoured over that of SR and other polymers during the blending process. A decrease in the SR/NR ratio for CB16_165C was calculated between 1 and 11 hours of blending. This decrease could potentially be attributed to the variability of the CB sample.

Figure 4.24: (a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted conveyor belt–derived crumb rubber following blending

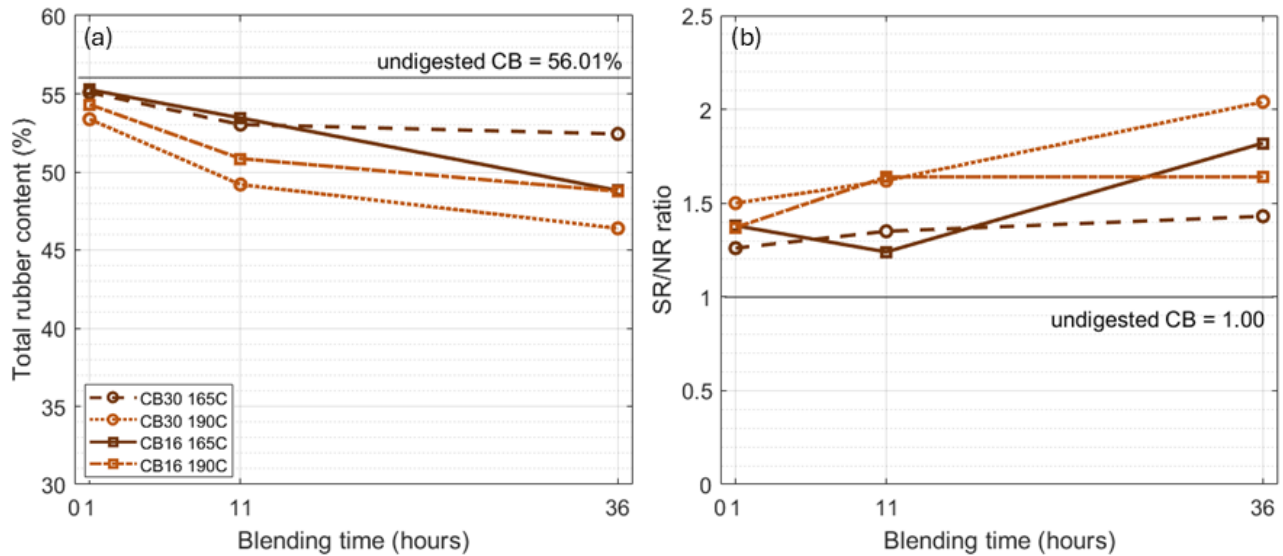


Figure 4.25 shows the constituent concentration for MT-derived crumb rubber particles digested for 1, 11 and 36 hours. All constituents present distinct trends with blending time; however, that of S16 particles blended at 165 °C contradicts expectations presenting an increase of NR content and a decrease in the content of SR and other polymers with an increase in blending time. S16 particles blended at 165 °C are expected to be going through the digestion stages presented in Section 2.2 more slowly compared to their S30 counterparts as well as particles blended at 190 °C. It may, therefore, be hypothesised that MT-derived crumb rubber particles undergo some chemical change, possibly subsequent cross-linking or chain scission, during the blending process, leading to this observed increase in apparent NR with blending time. This hypothesis would also align with observations in Section 4.2.3.

Figure 4.25: Concentration of (a) moisture and light oils, (b) natural rubber, (c) synthetic rubber and other polymers and (d) carbon black and fillers for mining tyre-derived crumb rubber following blending at 165 and 190 °C for 1, 11 and 36 hours

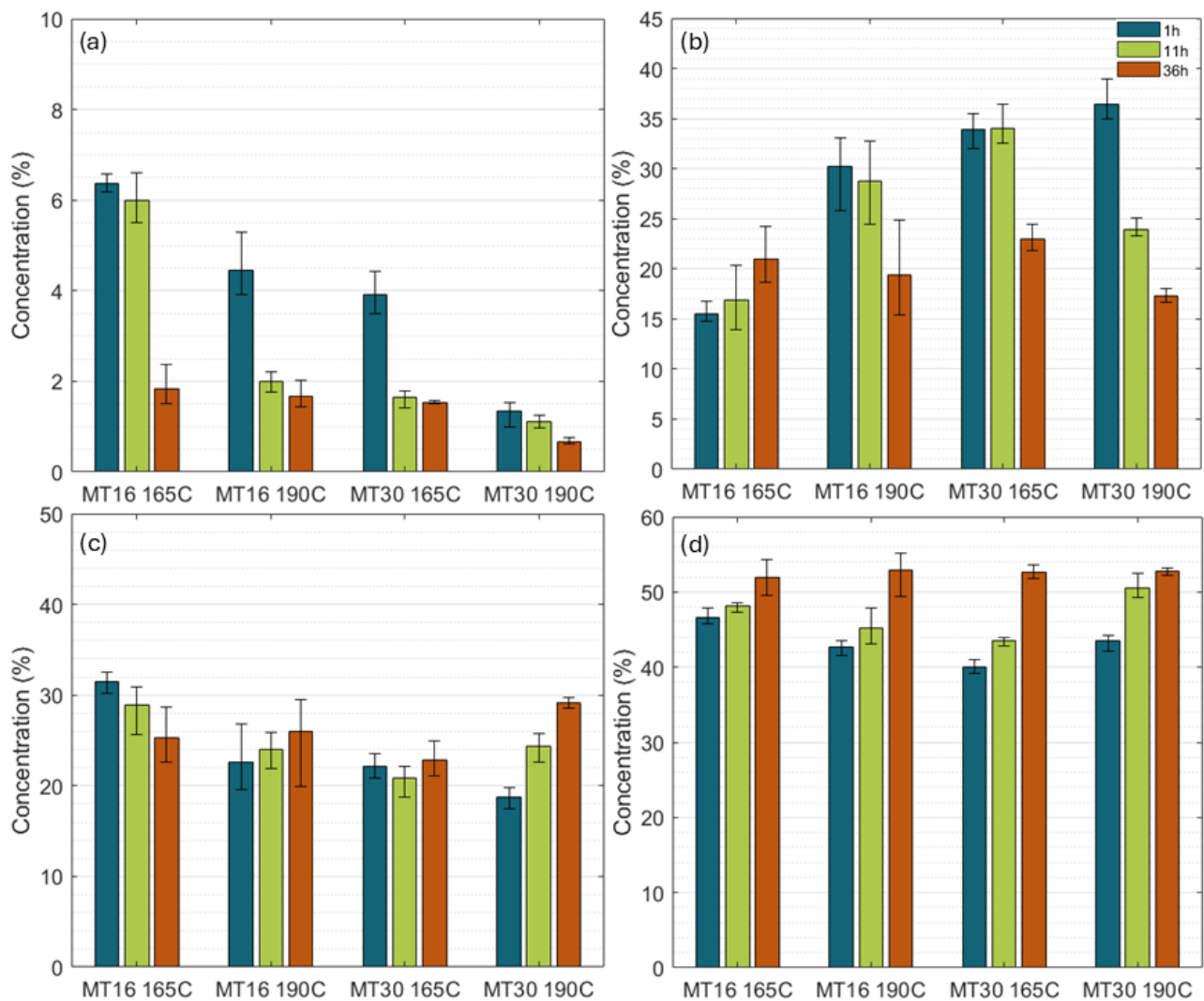
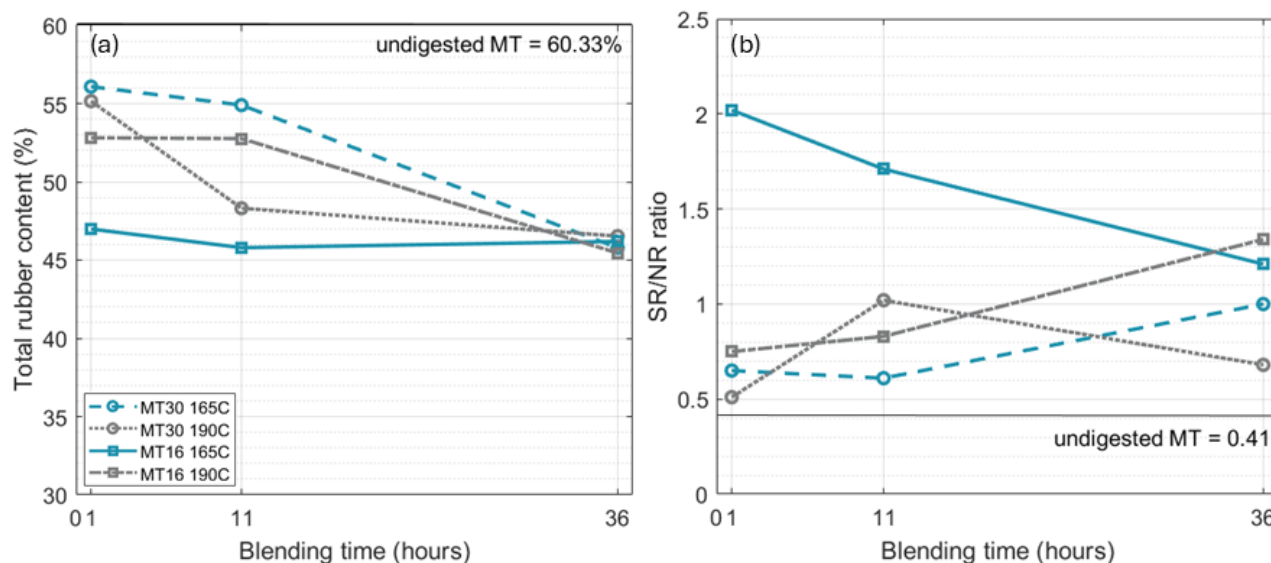


Figure 4.26 (a) shows that the total rubber content decreased in all digested MT-derived crumb rubber particles. Overall, the total rubber content was found to decrease with blending time for all digested MT-derived crumb rubber particles.

Figure 4.26 (b) presents the ratio of SR and other polymer content over the content of NR. In most cases, an increase in the SR/NR ratio is observed, suggesting the dissolution of NR, except for the S16 crumb rubber particles blended at 165 °C, as suggested by the findings of Figure 4.25 (b).

Figure 4.26: (a) Total rubber and other polymer content and (b) synthetic rubber and other polymer over natural rubber ratio for extracted mining tyre-derived crumb rubber following blending



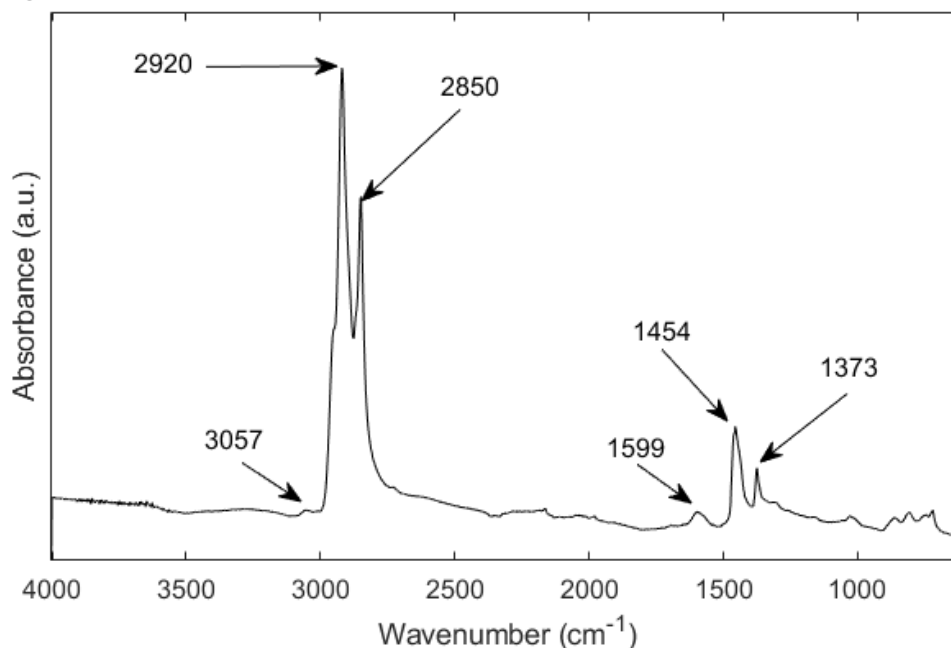
Crumb rubber is derived from EoL rubber products produced by different manufacturers. As a result, a variability within each sample, such as that evident in Figure 4.18, Figure 4.19, Figure 4.21, Figure 4.23 and Figure 4.25, is expected. In addition, TGA requires approximately 5 mg of crumb rubber per test. Therefore, depending on the size of the particles used, some variability dependent on the stage of digestion of these particles may also be introduced in the test results.

Overall, MT-derived crumb rubber had the greatest concentration of rubber content, followed by CB-derived crumb rubber, CT-derived crumb rubber and, lastly, TR-derived crumb rubber. CB-derived crumb rubber was found to comprise equal amounts of NR and SR, whereas all other types of crumb rubber had comparatively more NR. With digestion, the total rubber content of TR-derived crumb rubber was found to vary by approximately 13% (greatest for TR16_165C_1h at approximately 55% and lowest for TR16_190C_36h at approximately 42%), that of CT-derived crumb rubber was found to vary by approximately 9% (greatest for CT16_165C_11h at approximately 53% and lowest for CT30_190C_36h at approximately 44%), that of CB-derived crumb rubber was found to vary by 6.5% (greatest for CB16_165C_1h at approximately 55% and lowest for CB16_190C_36h at approximately 49%) and lastly, that of MT-derived crumb rubber was found to vary by approximately 11% (greatest for MT30_165C_1h at approximately 56% and lowest for MT16_190C_36h at approximately 45%).

4.2.5 Fourier-Transform Infrared Spectroscopy

FTIR was conducted to identify whether new functional groups were present in the binders following their modification by crumb rubber and as digestion progressed. Figure 4.27 shows the spectra of the C170 bitumen used as a base for the CRMBs. The peaks for the aliphatic groups at 1373 and 1454 cm^{-1} (corresponding CH_3 and CH_2 bends, respectively) and the aliphatic CH_2 stretches at 2850 and 2920 cm^{-1} can be clearly identified. A peak indicating a C=C stretch of aromatic ring at 1599 cm^{-1} could also be detected (Austroads 2014a, 2020; Daly 2017).

Figure 4.27: FTIR spectra for unmodified C170 bitumen



As discussed in Section 2.1, crumb rubber is primarily composed of NR and SR (typically SBR). A number of peaks have been associated with the presence of NR including 833, 885, 1370, 1378, 1450 and 1664 cm^{-1} (Nunes et al. 2018). However, ASTM D3677-10(2023) *Standard Test Methods for Rubber – Identification by Infrared Spectrophotometry* states that the 1450 cm^{-1} peak along with other peaks, is not of diagnostic value for rubbers. According to ASTM D3677-10(2023), styrene-butadiene-styrene (SBS) rubber presents very strong peaks at 699 and 962 cm^{-1} , a fairly strong peak at 990 cm^{-1} , strong peaks at 775 and 909 cm^{-1} and a medium peak at 1490 cm^{-1} . However, SBS is not typically expected to be present in truck and car tyres.

Figure 4.28 to Figure 4.31 show the spectra for as-received TR-, CT-, CB- and MT-derived crumb rubbers. Within the fingerprint region (1500–600 cm^{-1}) the spectra become rather complex as the different components overlap. Some of the more pronounced peaks present are identified with the intent to understand whether any appear in the melt-extracted binders.

Figure 4.28 presents the spectra for TR-derived crumb rubber. Some of the peaks attributed to the presence of NR according to Nunes et al. (2018) were found. These were around wavenumbers 833, 885 and 1375 cm^{-1} . A distinct peak around 1450 cm^{-1} was also identified, however; it was not used to identify rubbers according to ASTM D3677-10(2023). Peaks around 777, 909 and 966 cm^{-1} may be attributed to SBR according to ASTM D3677-10(2023). The peak around 721 cm^{-1} may be attributed to the vibration of the $(-\text{CH}_2-)_n$ chemical group (Ali et al. 2022), the peak around 1021 cm^{-1} may be due to $-\text{C}-\text{C}-$ stretching explained by the presence of carbon black commonly added to tyres, the peak around 1094 cm^{-1} may be due to the presence of SiO_2 , whereas the peaks around 1398 and 1539 cm^{-1} could be attributed to ZnO (Colom et al. 2016).

Figure 4.28: FTIR spectra for truck tyre-derived crumb rubber

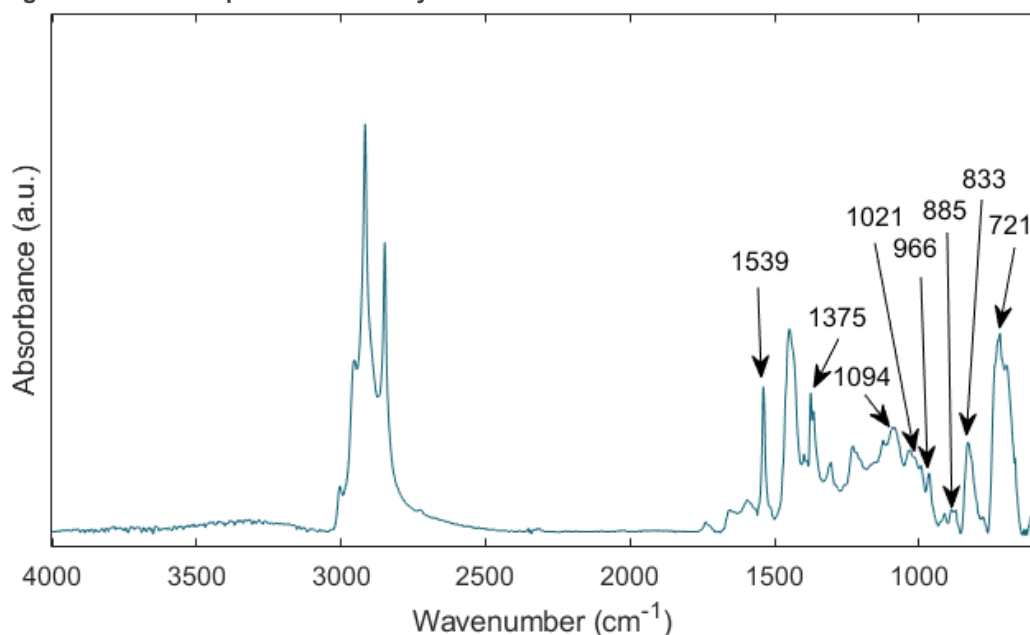


Figure 4.29 shows the spectra of the CT-derived crumb rubber. Many of the peaks found were the same as those identified for TR-derived crumb rubber in Figure 4.28, including the peaks around 722, 831, 965, 1096, 1375 and 1539 cm⁻¹. In Figure 4.29, a distinct peak at 699 cm⁻¹ was also observed, which according to ASTM D3677-10 (2023), may be attributed to SBR. The intensity and morphology of the peaks in Figure 4.29 differ from those in Figure 4.28, suggesting that the 2 rubbers differ in composition.

Figure 4.29: FTIR spectra for car tyre-derived crumb rubber

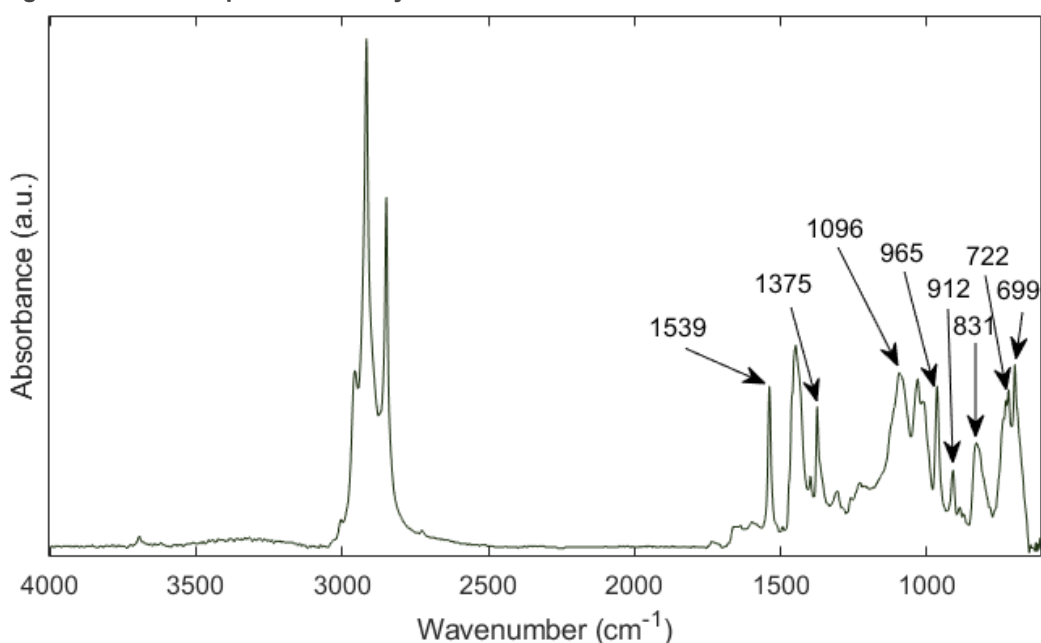


Figure 4.30 presents the spectra for the CB-derived crumb rubber. Similarly to TR- and CT-derived crumb rubber, peaks at 722, 833, 964, 1094, 1375 and 1539 cm⁻¹ suggest the presence of NR and SR were identified. Further, peaks at 699 and 909 cm⁻¹, attributed to SBR (ASTM D3677-10(2023)) and found in CT-derived crumb rubber but not in TR-derived crumb rubber, were also detected. The intensity and morphology of the peaks in Figure 4.30 were again different to that of Figure 4.28 and Figure 4.29, suggesting that the composition of the CB-derived crumb rubber varies from that of the TR- and CT-derived crumb rubbers.

Figure 4.30: FTIR spectra for conveyor belt–derived crumb rubber

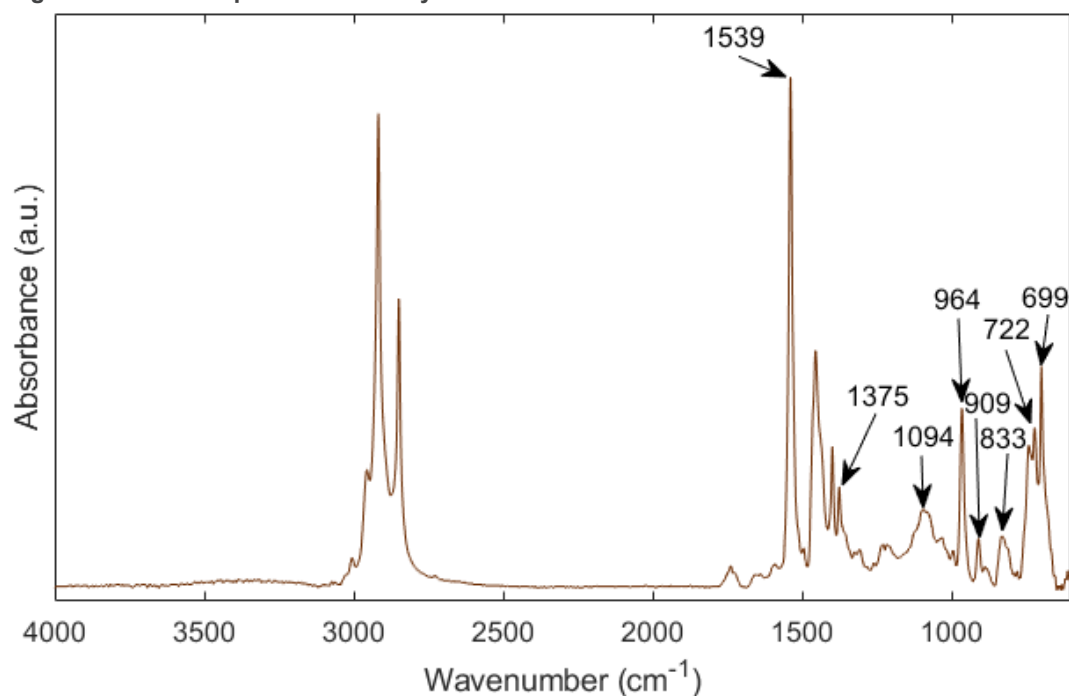
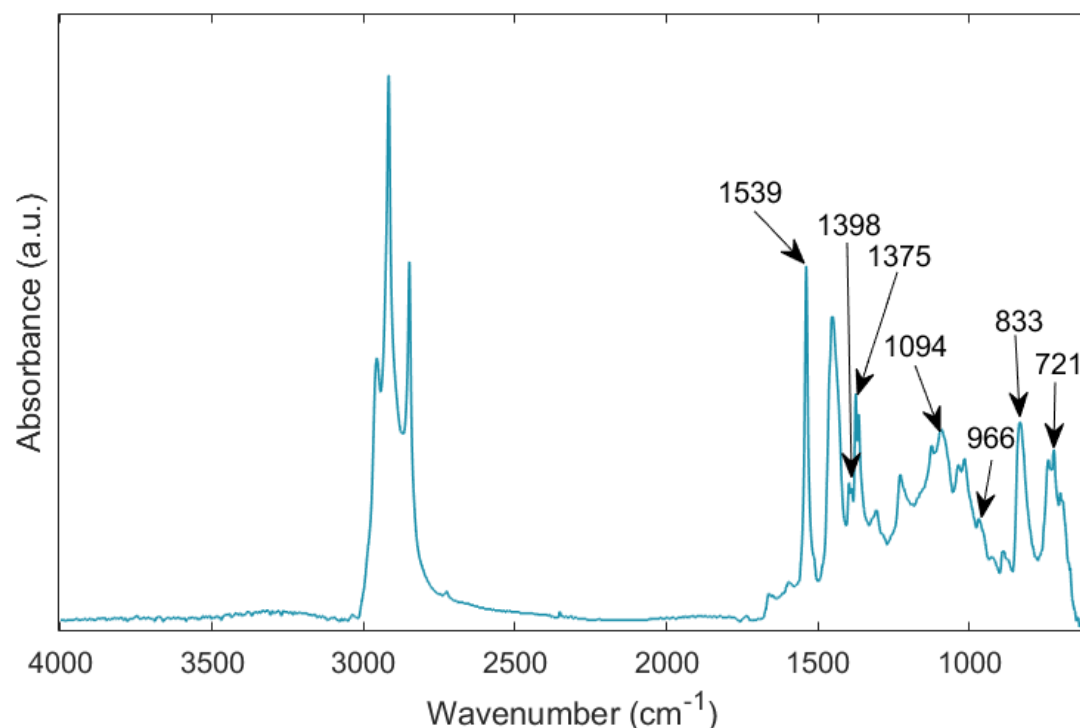


Figure 4.31 shows the spectra for MT-derived crumb rubber. Within the fingerprint region, a peak at 721 cm⁻¹ attributed to the vibration of (-CH₂)_n chemical group, which was also found in the TR-derived crumb rubber, was identified. As in the rest of the crumb rubber types, peaks suggesting the presence of NR and SR are also marked in Figure 4.31. The intensity and morphology of the peaks in the MT-derived crumb rubber spectra are evidently different to those presented in Figure 4.28, Figure 4.29 and Figure 4.30. This suggests that the chemical makeup of MT-derived crumb rubber is different to the TR-, CT- and CB-derived crumb rubber.

Figure 4.31: FTIR spectra for mining tyre–derived crumb rubber



During binder blending and following crumb rubber swelling, the crumb rubber particles may degrade. This can happen either through devulcanisation (breaking of C-S and S-S bonds formed during vulcanisation) and/or depolymerisation (breaking of the main C-C bonds) (Wisniewska et al. 2022). As a result, the spectra

of the melt-extracted binders (method described in Section 3.2.4) may not be exactly as presented in Figure 4.27, but rather they may be affected by the presence of the rubbers following blending. Figure 4.32 to Figure 4.39 show these spectra for the melt-extracted CRMB. Overall, peaks associated with crumb rubber are seldom present. It is important to note that the identification of peaks in FTIR requires that a certain concentration of these functional groups is present in the specimen.

Figure 4.32 shows the spectra for the melt-extracted binders blended using TR-derived crumb rubber at 165 °C for 1, 11 and 36 hours. For all spectra presented, the main peaks are as shown in Figure 4.27 for the base bitumen. For TR30_165C_1h, new peaks were identified around 2359 and 2333 cm^{-1} . These can be attributed to CO_2 stretching as a trace by-product of oxidation or atmospheric CO_2 (Ogura et al. 1998; Pacheco Santos et al. 2007; Zhu et al. 2015). Such spectral artifacts are rather common and were identified here even though a background scan was performed for each sample. They are not of diagnostic significance.

Figure 4.32: FTIR spectra for melt-extracted binders blended using truck tyre-derived crumb rubber at 165 °C; (a) S30 and (b) S16

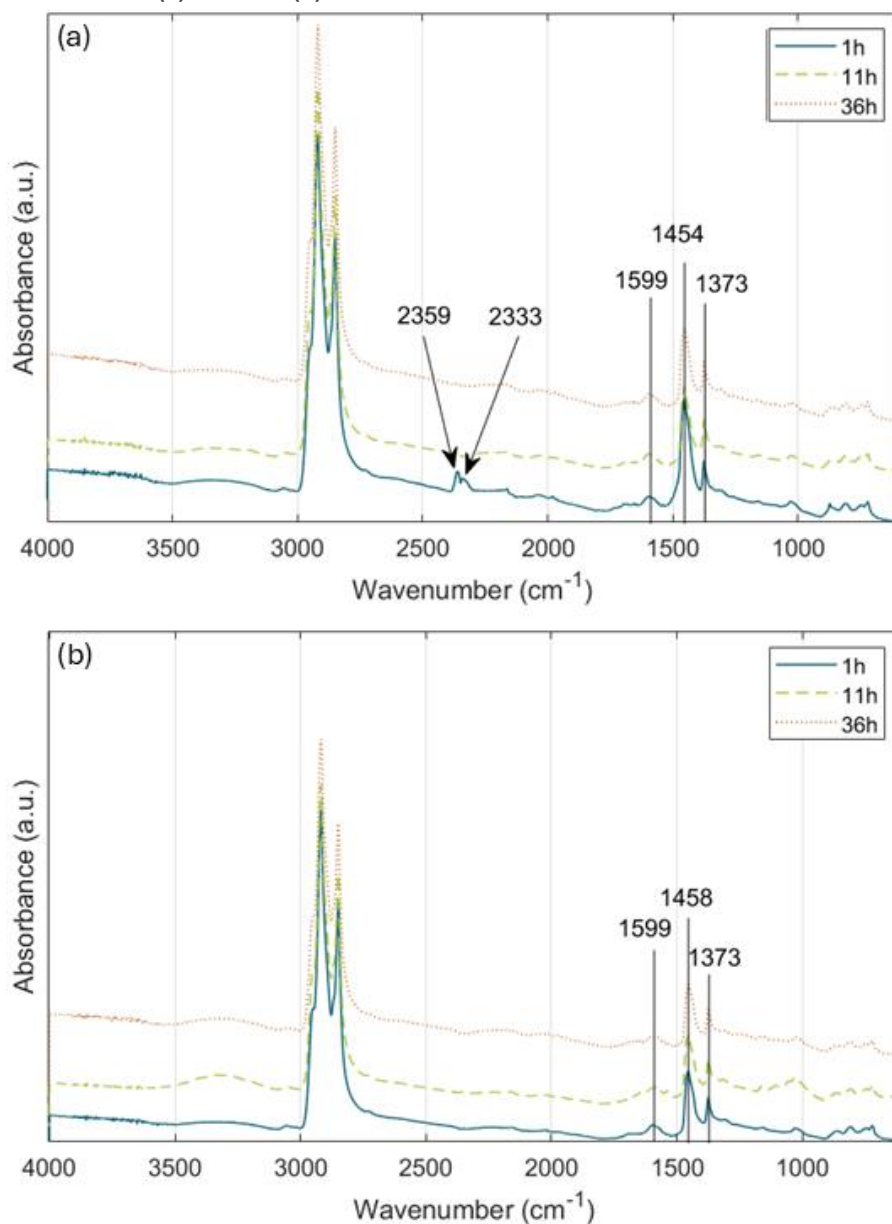
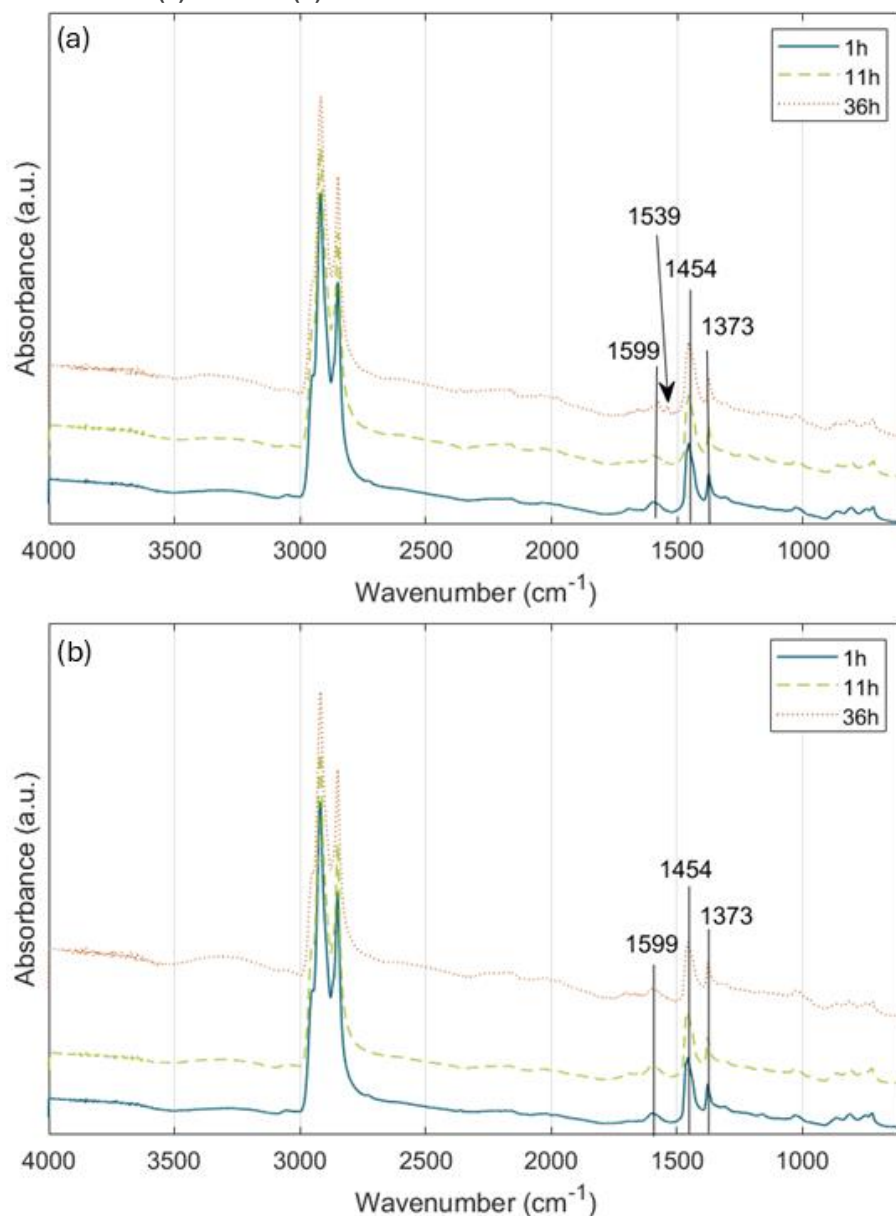


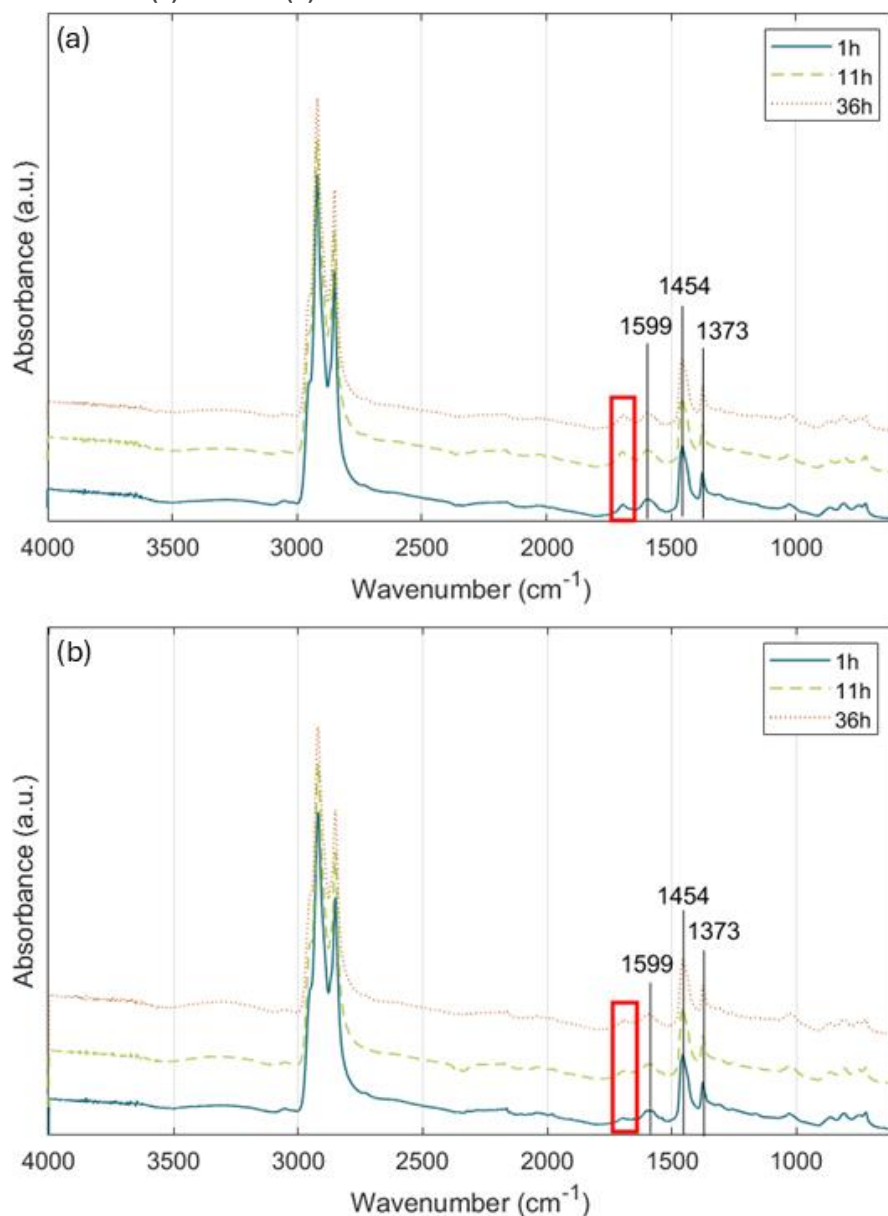
Figure 4.33 shows the spectra for TR-derived CRMBs following blending at 190 °C. The spectra of TR30_190C_36h reveal a peak at 1539 cm^{-1} , also present in the spectra of the TR-derived crumb rubber of Figure 4.28. This peak is also found in CT16_190C_1h and _11h melt-extracted binders (shown in Figure 4.35). As noted, the peak at 1539 cm^{-1} is most likely due to ZnO from the crumb rubber.

Figure 4.33: FTIR spectra for melt-extracted binders blended using truck tyre-derived crumb rubber at 190 °C; (a) S30 and (b) S16



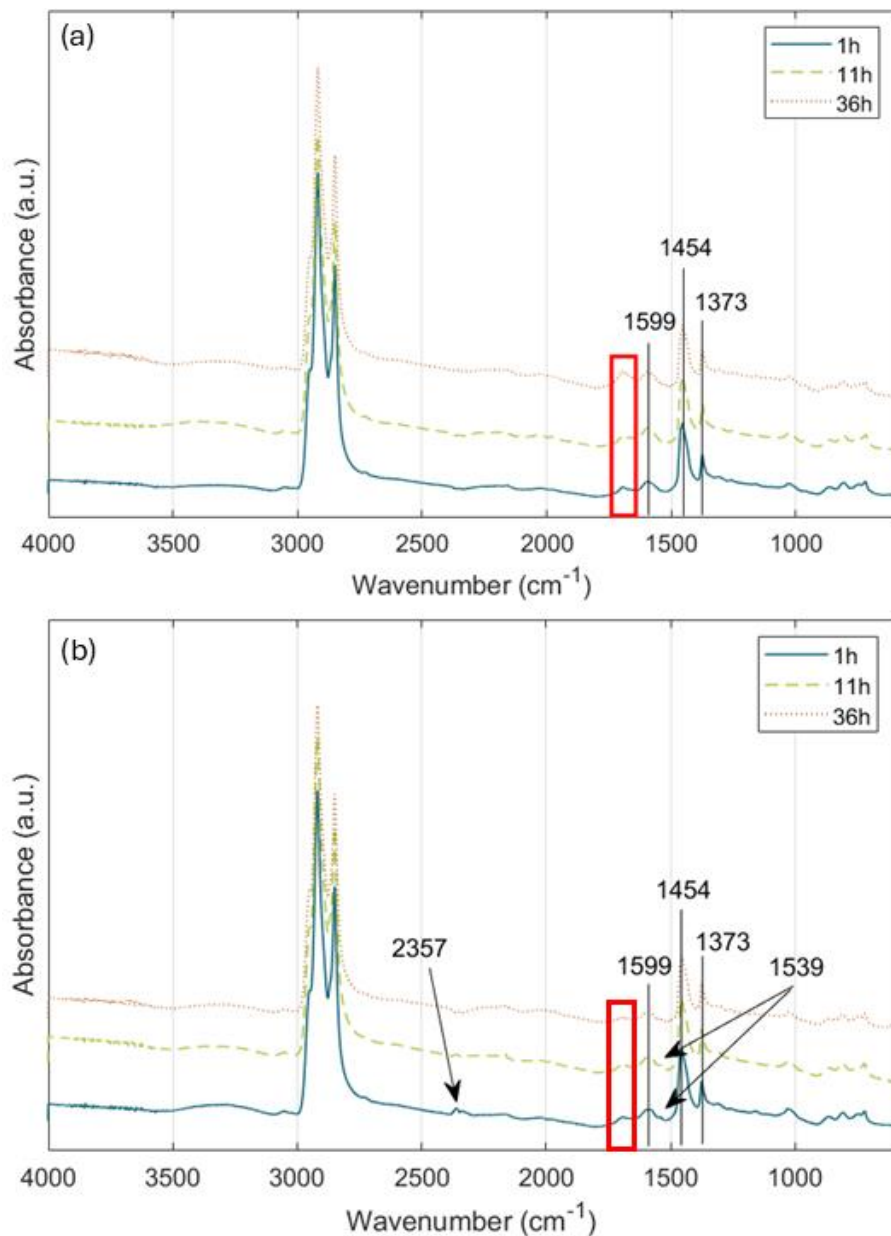
The spectra for the extracted CT-derived crumb rubber binders blended at 165 °C are presented in Figure 4.34. The 3 main peaks at 1373, 1454 and 1599 cm⁻¹ from the base bitumen are clearly identified in the spectra of all melt-extracts. The spectra of CT30_165C and CT16_165C present peaks between 1695 and 1720 cm⁻¹. The relevant region is highlighted in the red box in Figure 4.34. This peak is present for 1, 11 and 36 hours, although it is more pronounced in the S30 samples. The range of 1695 to 1720 cm⁻¹ is associated with carbonyl (C=O) stretching, thereby suggesting the bitumen in the melts has undergone some level of oxidative ageing (Harrison et al. 2020; Wang, Yang et al. 2021).

Figure 4.34: FTIR spectra for melt-extracted binders blended using car tyre-derived crumb rubber at 165 °C; (a) S30 and (b) S16



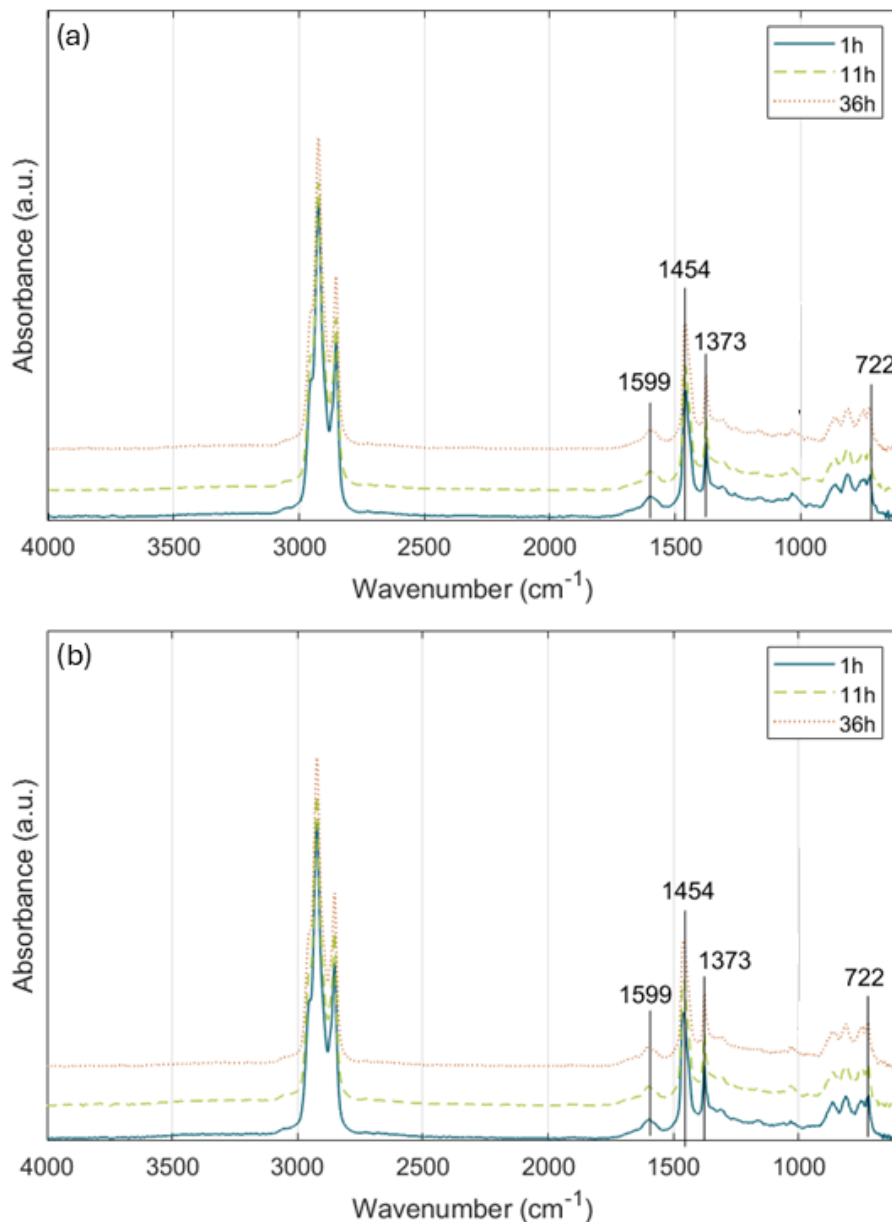
The spectra for the extracted CT-derived crumb rubber binders blended at 190 °C are presented in Figure 4.35. The 3 main peaks of the base bitumen are identified at 1373, 1454 and 1599 cm^{-1} and were found in the spectra of all melt-extracts of Figure 4.35. Consistent with the other CT-derived samples in Figure 4.34, the peak at 1695 to 1720 cm^{-1} , representative of oxidative ageing, is seen for all 6 spectra. It is not excluded as a possibility that such oxidative ageing occurred during subsequent handling of the CRMBs. CT16_190C_11h and _36h show the same 1539 cm^{-1} peak identified in Figure 4.33, which is most likely due to ZnO from the crumb rubber. A peak at 2357 cm^{-1} is revealed in the spectrum of CT16_190C_1h and, similarly to Figure 4.32, may be attributed to background atmospheric CO_2 .

Figure 4.35: FTIR spectra for melt-extracted binders blended using car tyre-derived crumb rubber at 190 °C; (a) S30 and (b) S16



The spectra for the extracted CB-derived crumb rubber binders blended at 165 °C are presented in Figure 4.36. The 3 main peaks associated with the base bitumen at 1373, 1454 and 1599 cm^{-1} can clearly be identified in Figure 4.36 also. In addition to these peaks, a peak at 722 cm^{-1} was found for all melt-extracted binders. This FTIR peak was observed for the CB-derived crumb rubber in Figure 4.30, and it was attributed to the presence of NR and SR.

Figure 4.36: FTIR spectra for melt-extracted binders blended using conveyor belt–derived crumb rubber at 165 °C; (a) S30 and (b) S16



The spectra for the melt-extracted CB-derived crumb rubber binders blended at 190 °C are presented in Figure 4.37. The main base bitumen peaks are identified in these samples at 1373, 1454 and 1599 cm⁻¹. As with previously discussed spectra, additional peaks identified at 722 cm⁻¹, which are attributed to NR and SR, were also found.

Figure 4.37: FTIR spectra for melt-extracted binders blended using conveyor belt–derived crumb rubber at 190 °C; (a) S30 and (b) S16

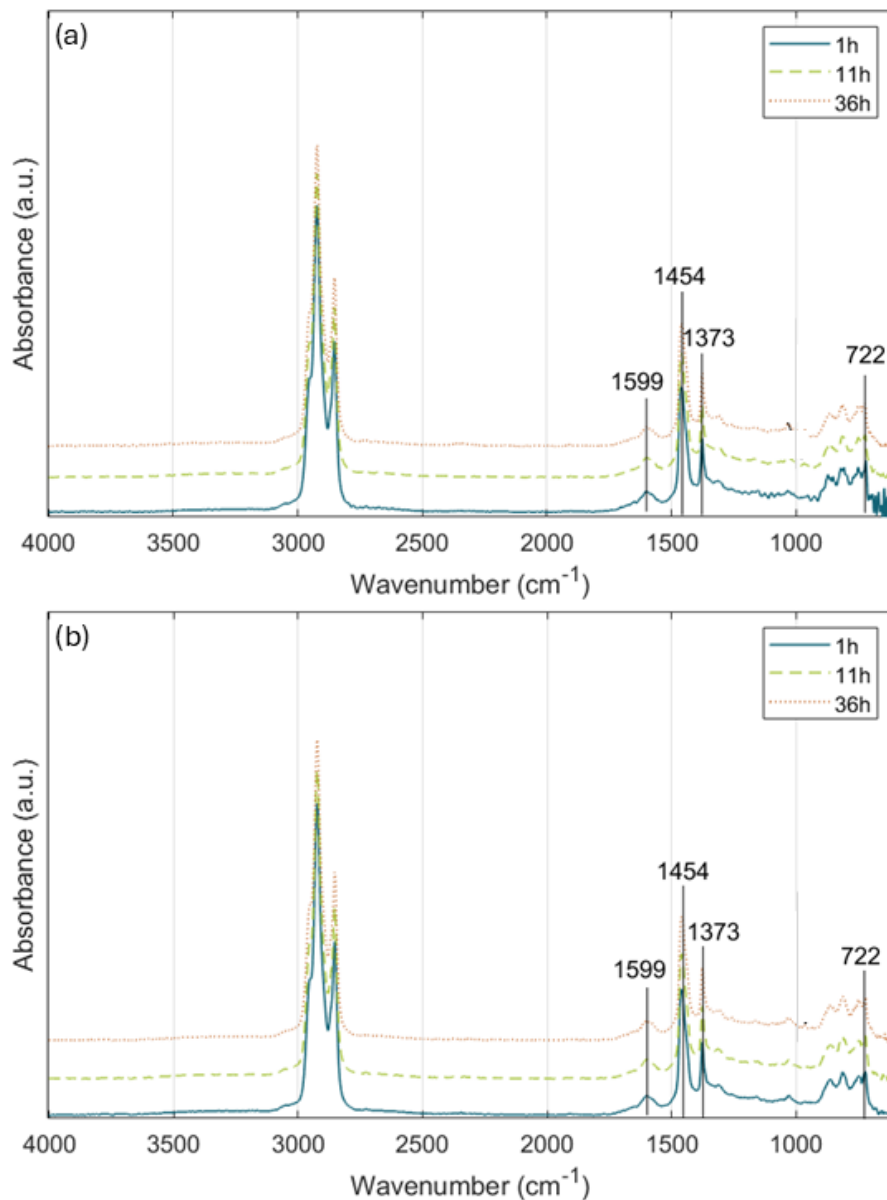


Figure 4.38 presents the FTIR spectra of melt-extracted MT-derived crumb rubber binders blended at 165 °C. The peaks at 1373, 1454 and 1599 cm^{-1} are characteristic to those of bitumen, as shown in Figure 4.27. Peaks around 722 cm^{-1} , attributed to the presence of NR and SR, were also identified for all S30 and S16 binders. The spectra for MT16_165C_11h also show a distinct peak at 1721 cm^{-1} . This peak can be attributed to the presence of a carbonyl group created as a result of oxidation reactions. Interestingly, however, the same peak was not identified for MT16_165C_36h, which was produced in the same tin as MT16_165C_11h. It may be suggested, therefore, that this oxidative reaction occurred at a different stage during sample preparation rather than during blending.

Figure 4.38: FTIR spectra for melt-extracted binders blended using mining tyre-derived crumb rubber at 165 °C; (a) S30 and (b) S16

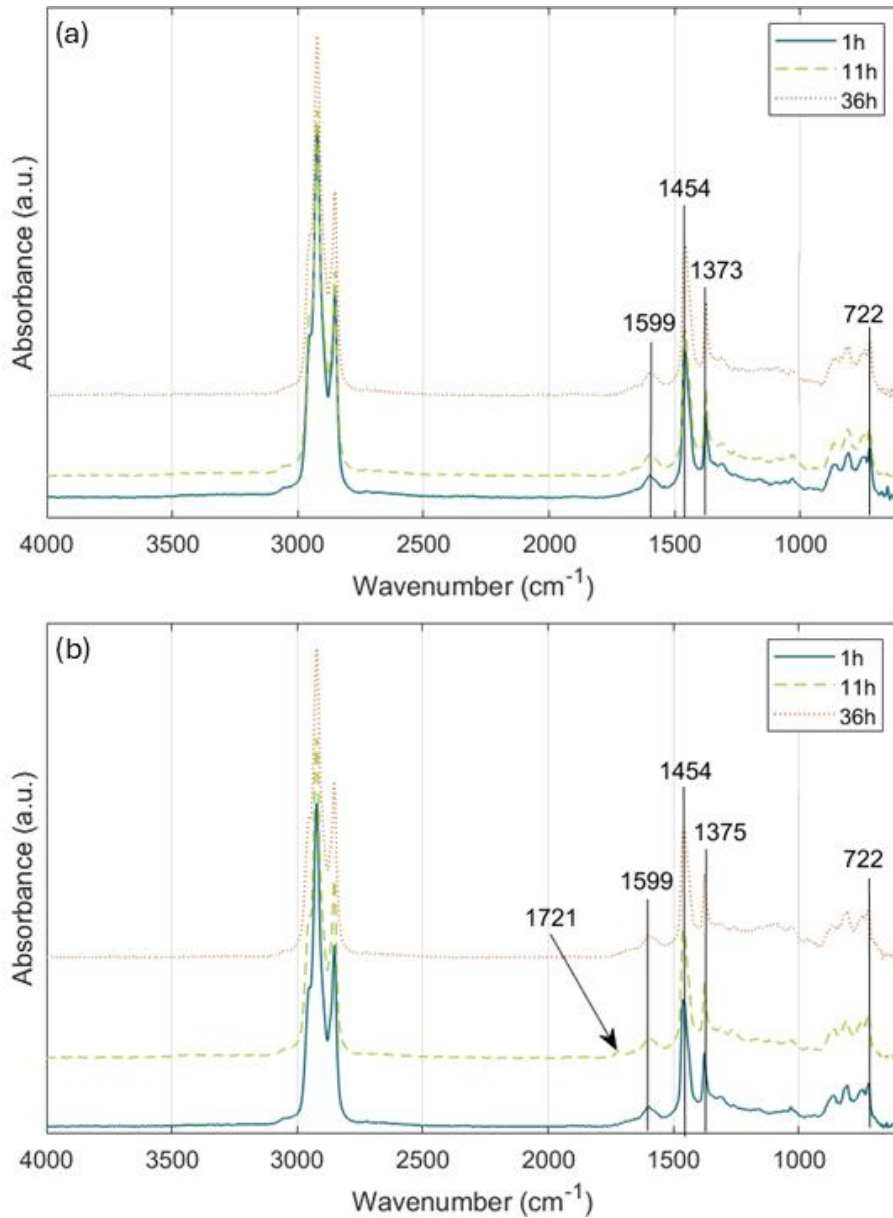
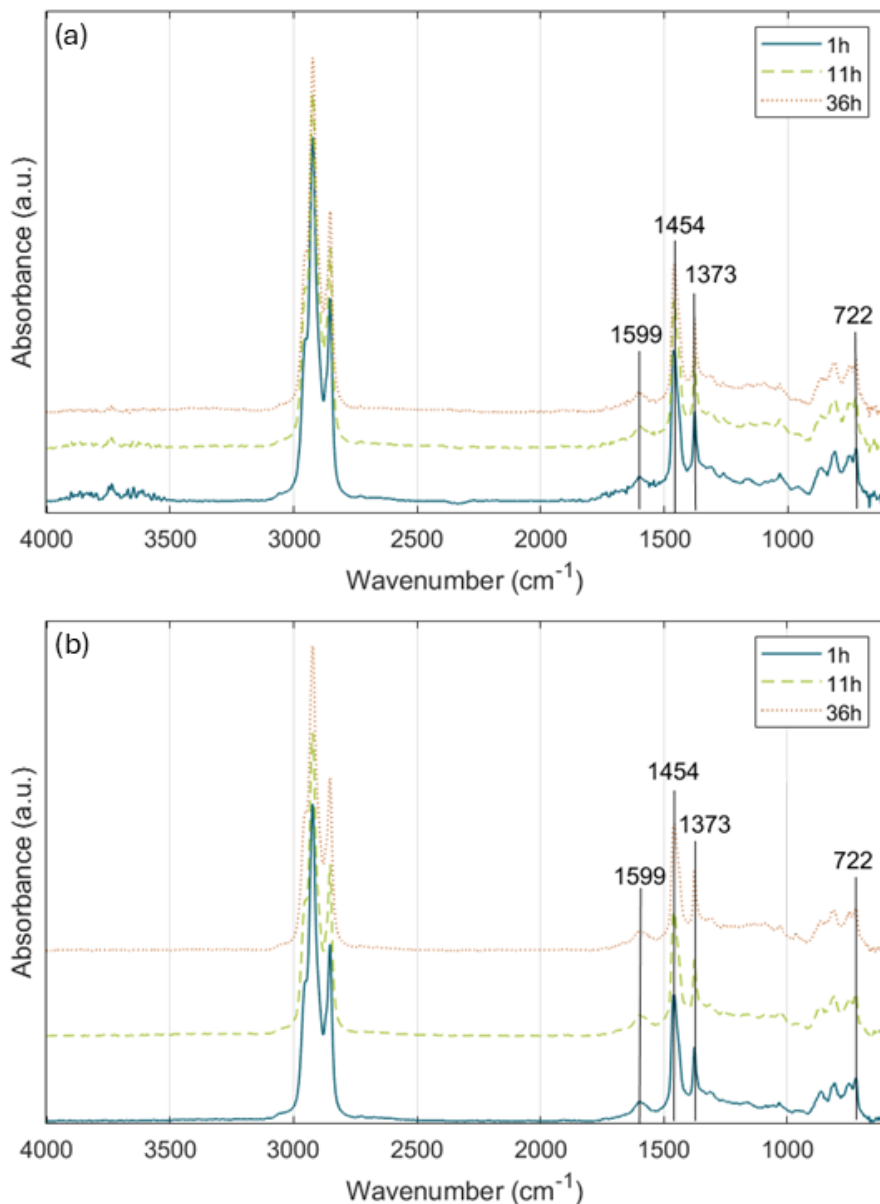


Figure 4.39 presents the spectra for the melt-extracts of MT-derived binders blended at 190 °C. Peaks were identified at 722, 1373, 1454 and 1599 cm⁻¹ as per Figure 4.36 to Figure 4.38.

Figure 4.39: FTIR spectra for melt-extracted binders blended using mining tyre-derived crumb rubber at 190 °C; (a) S30 and (b) S16



Overall, the spectra of the melt-extracted binders consistently presented peaks at 1373, 1454 and 1599 cm^{-1} attributed to the base bitumen. The spectra of the TR-derived CRMBs blended at 165 °C showed no additional peaks due to crumb rubber dissolution.

A peak at 1539 cm^{-1} attributed to the presence of ZnO from the crumb rubber was found in:

- TR30_190C_36h
- CT16_190C_1h
- CT16_190C_11h.

Lastly, peaks within the carbonyl region suggesting the oxidation of the melt-extracted binders were found for all CT-derived CRMBs as well as in the spectra of MT16_165C_11h.

Aside from the identification of additional peaks in the above listed spectra, the peaks between 706 and 922 cm^{-1} for CB- and MT-derived CRMB spectra were found to be amplified when compared to those of TR- and CT-derived CRMB spectra.

The most significant observation that can be drawn from the FTIR analysis is the lack of peaks associated with the source rubber in the melt-extracted binders. As noted earlier in this section, this could be a result of

the concentration of any dissolved portion of the rubber in the liquid phase. It is, therefore, concluded that FTIR is not an appropriate tool to distinguish between binders.

4.2.6 Optical Microscopy

Figure 4.40 shows optical microscope images for all as-received crumb rubber particles. All samples presented some particles with a relatively smooth surface and others with a more textured one. The particles also appear to mainly be of irregular shape. These observations are in line with expectations as the particles were mechanically ground at ambient temperature (Khalili et al. 2019).

Figure 4.40: 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

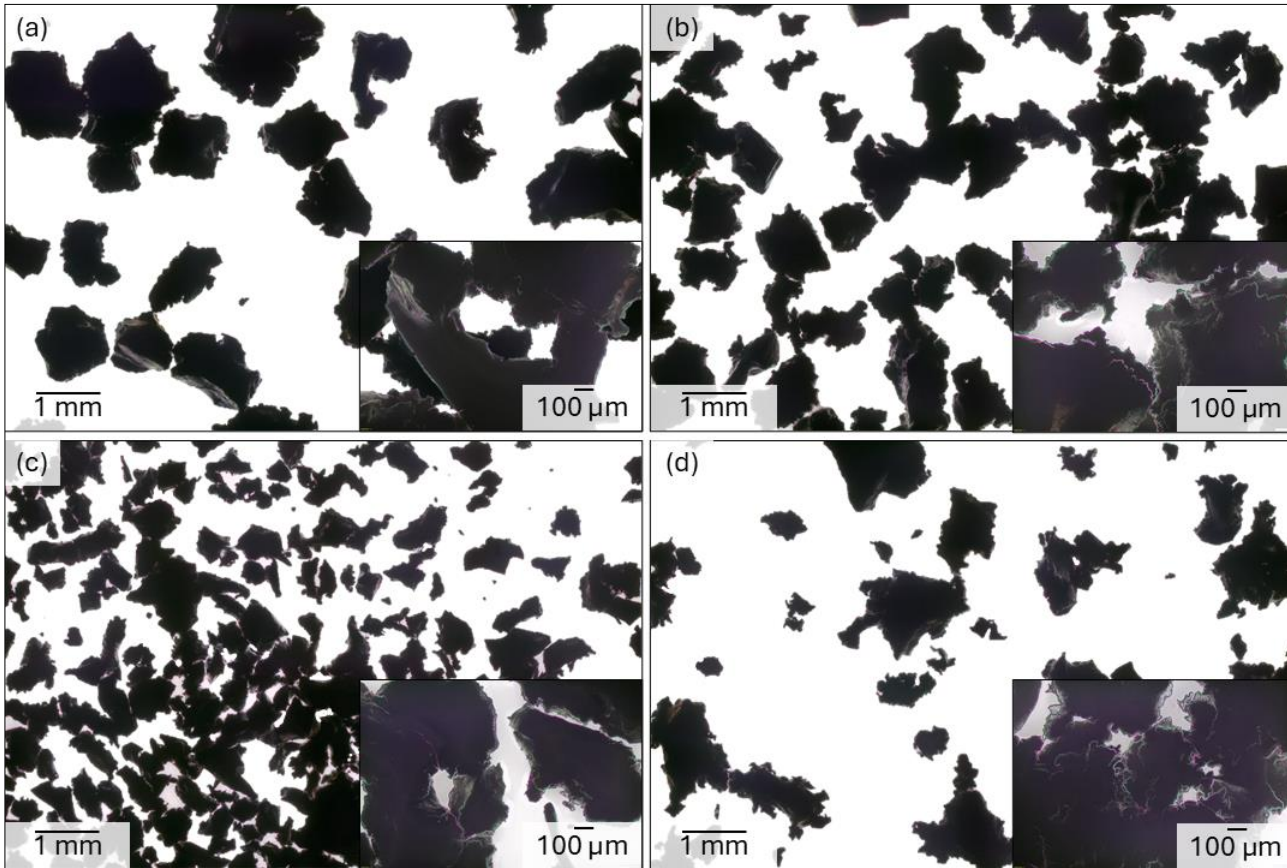


Table 4.3 to Table 4.10 present the optical microscope images of crumb rubber as extracted following digestion for 1, 11 and 36 hours according to the method described in Section 3.2.3. Overall, with an increase in blending time the particles were found to increasingly agglomerate. Some apparent agglomerates could be easily separated when being placed on the slides (meaning that the apparent agglomerates were not physically bonded), whereas others remained attached. From the images presented in Table 4.3 to Table 4.10, it is not always clear whether the apparent contact points are due to the bonding of 2 separate particles or the part separation of a single particle. However, agglomeration bridging (early-stage agglomeration) is in some cases evident. Some of these are highlighted with green in the optical microscope images presented in Table 4.3 to Table 4.10.

Some particle satellites could also be identified. Satellites are separate entities, in this case most likely identified due to the presence of smaller crumb rubber particles in the samples or the result of partially dissolved particles. Some identified satellites are marked with orange in Table 4.3 to Table 4.10.

Furthermore, in the case of TR-, CT- and MT-derived crumb rubber particles, textile fibres can be found, probably due to their presence in the tyre samples, as shown in Figure 2.1. Some are identified in Table 4.3, Table 4.6, Table 4.9 and Table 4.10.

The surface morphology of the different types of rubber was also found to vary with digestion. Extracted TR- and CB-derived particles appear to be smoother, whereas extracted CT- and MT-derived crumb rubber particles present a more textured surface. In all cases, particles appear to present a more elongated shape with increasing blending time.

The agglomeration of particles prevented any meaningful comparison between the 2 crumb rubber size gradations investigated in this research, to the extent that optical microscopy of S30 extracted crumb rubber was not conducted.

Table 4.3: Optical microscope images of extracted truck tyre-derived crumb rubber after digestion at 165 °C

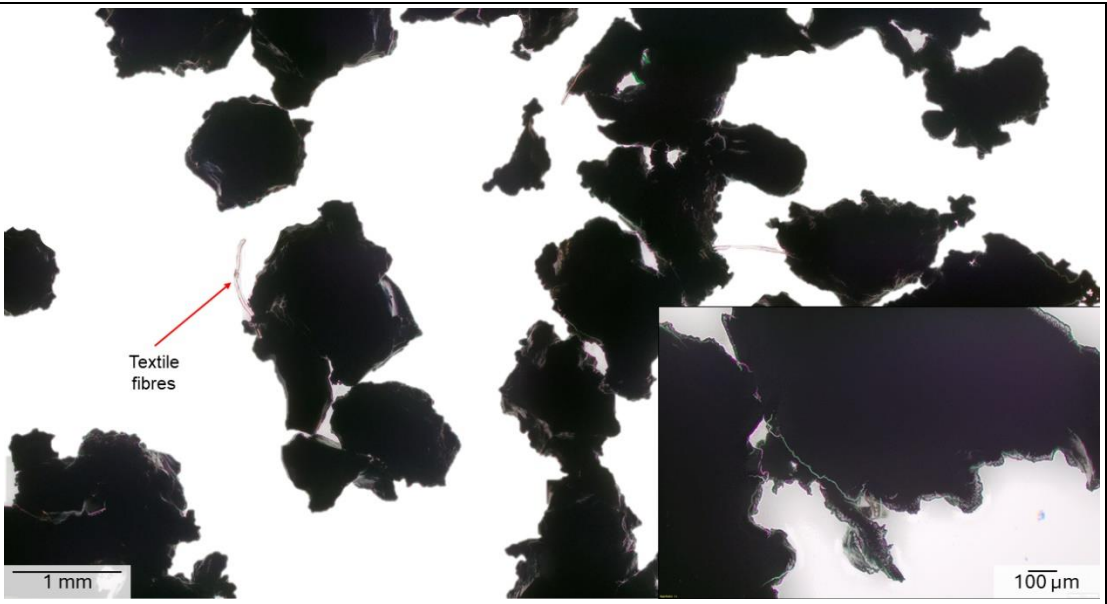
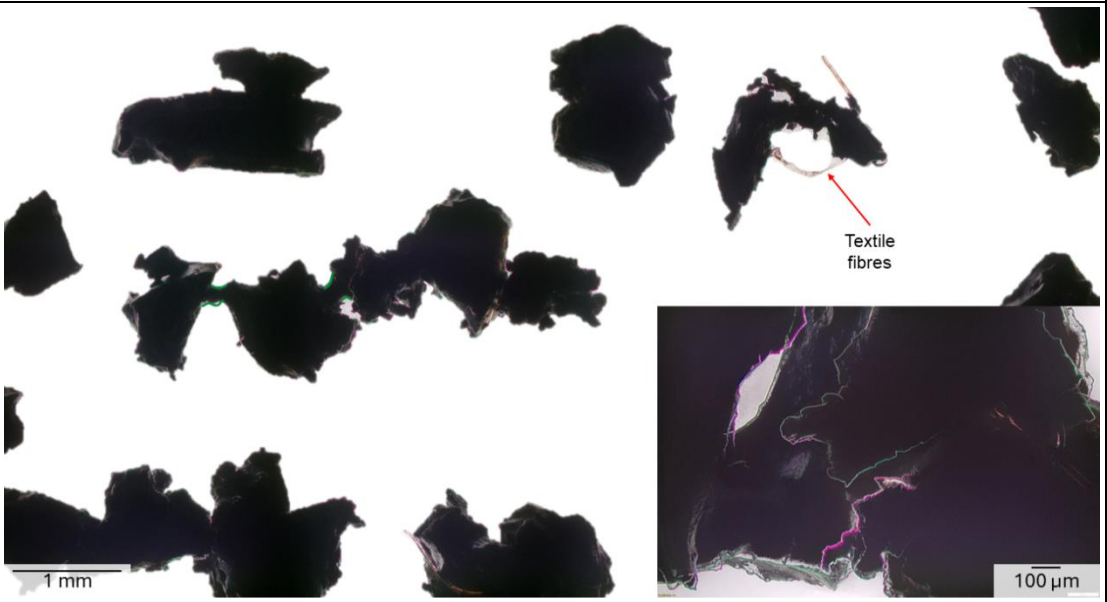
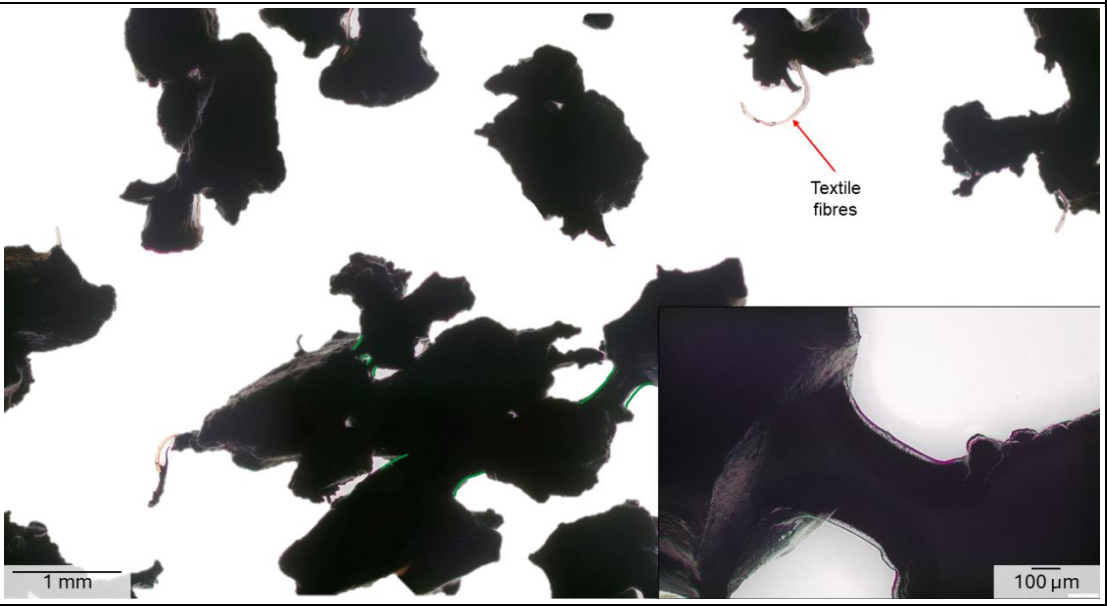
TR16_165C_1h	
TR16_165C_11h	
TR16_165C_36h	

Table 4.4: Optical microscope images of extracted truck tyre-derived crumb rubber after digestion at 190 °C

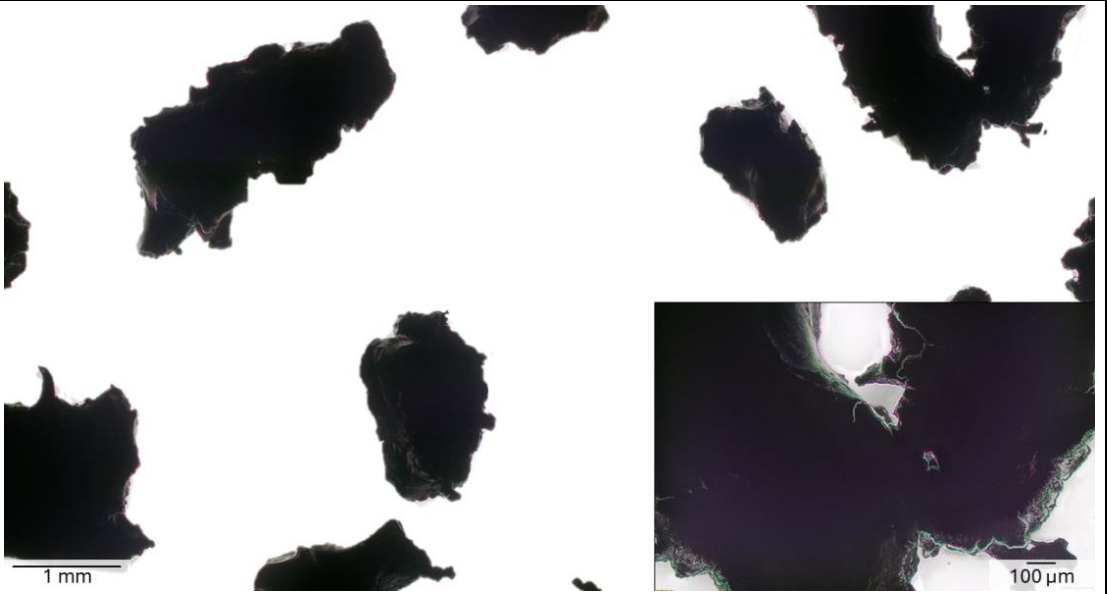
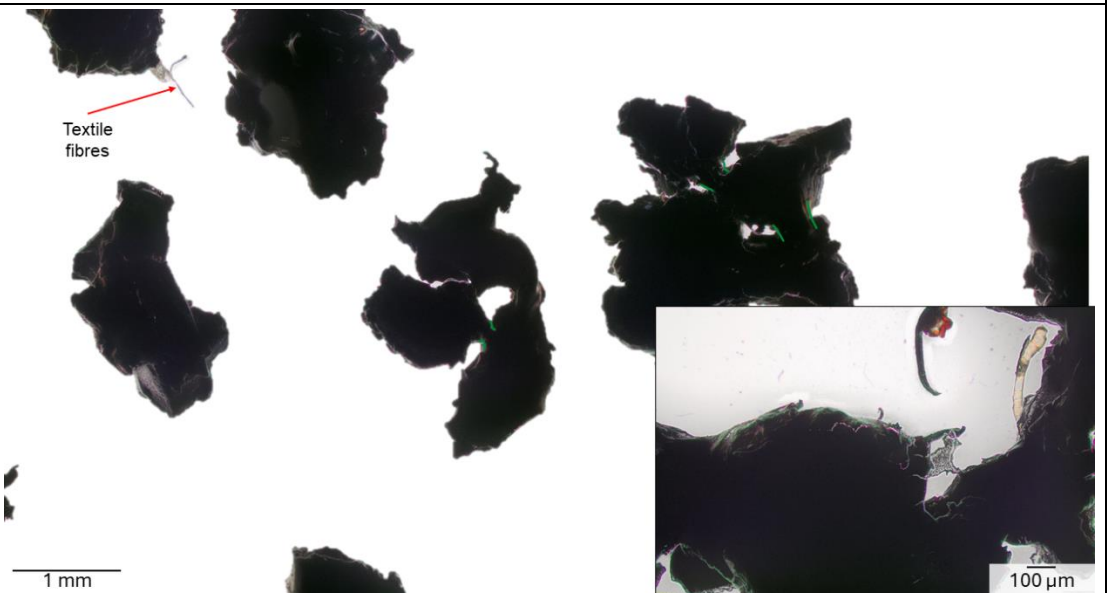
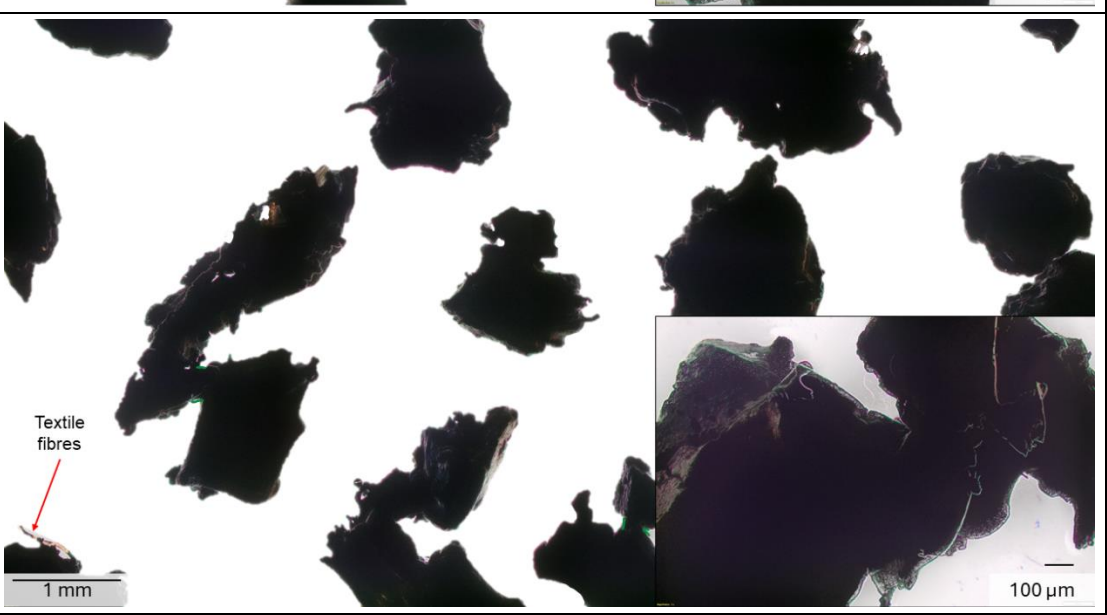
TR16_190C_1h	
TR16_190C_11h	
TR16_190C_36h	

Table 4.5: Optical microscope images of extracted car tyre–derived crumb rubber after digestion at 165 °C

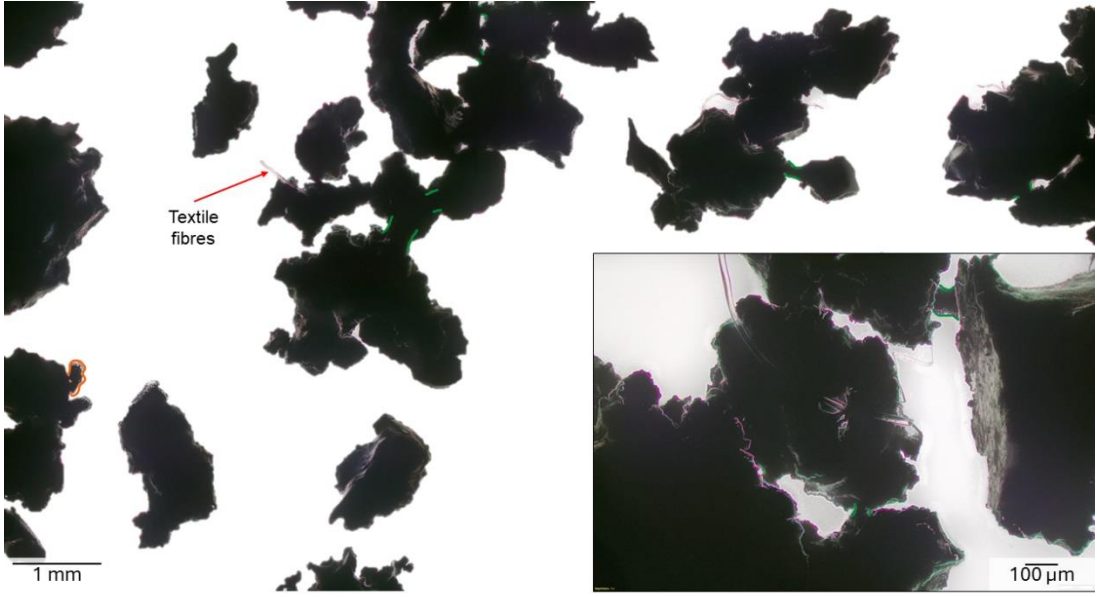
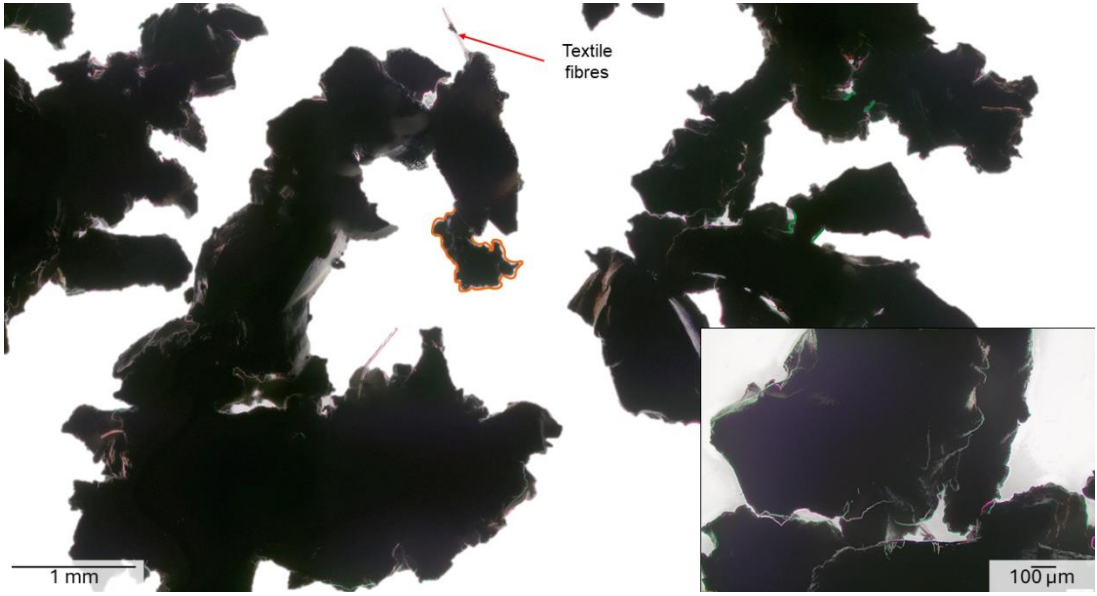
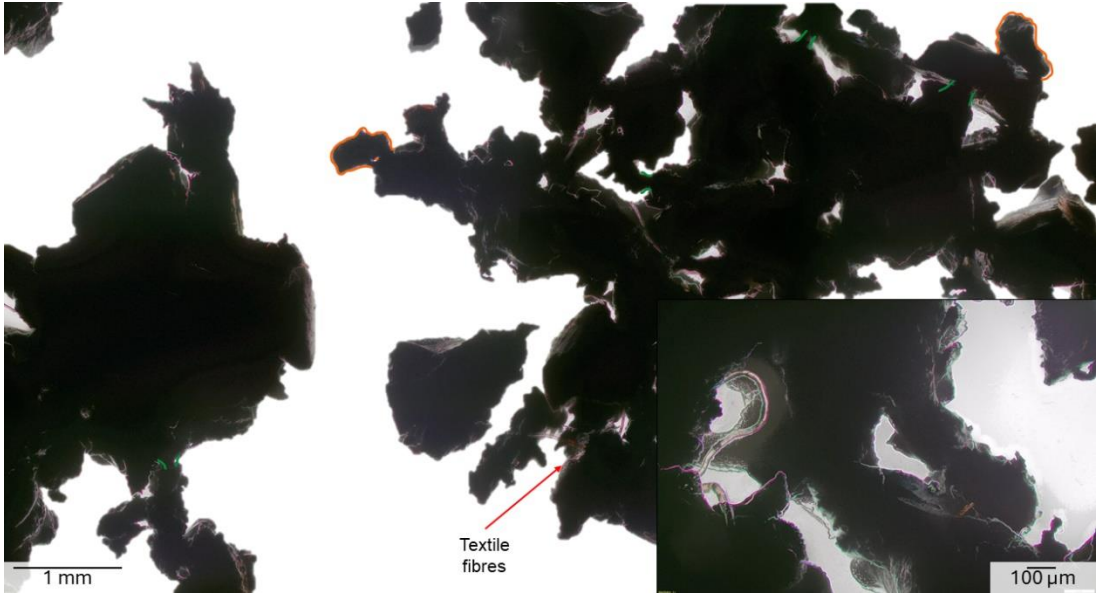
CT16_165C_1h	
CT16_165C_11h	
CT16_165C_36h	

Table 4.6: Optical microscope images of extracted car tyre–derived crumb rubber after digestion at 190 °C

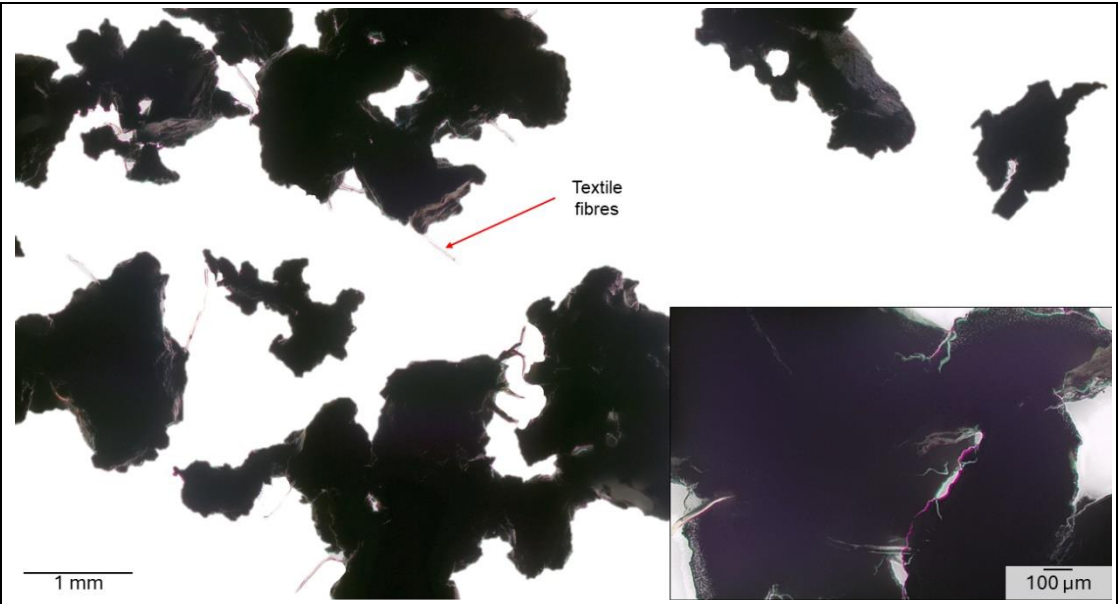
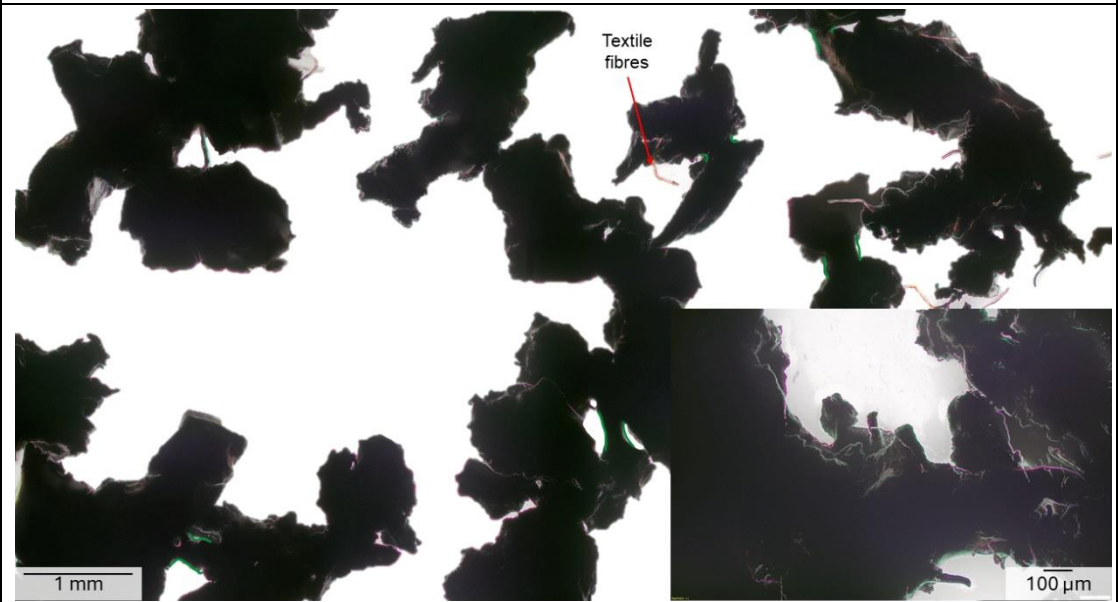
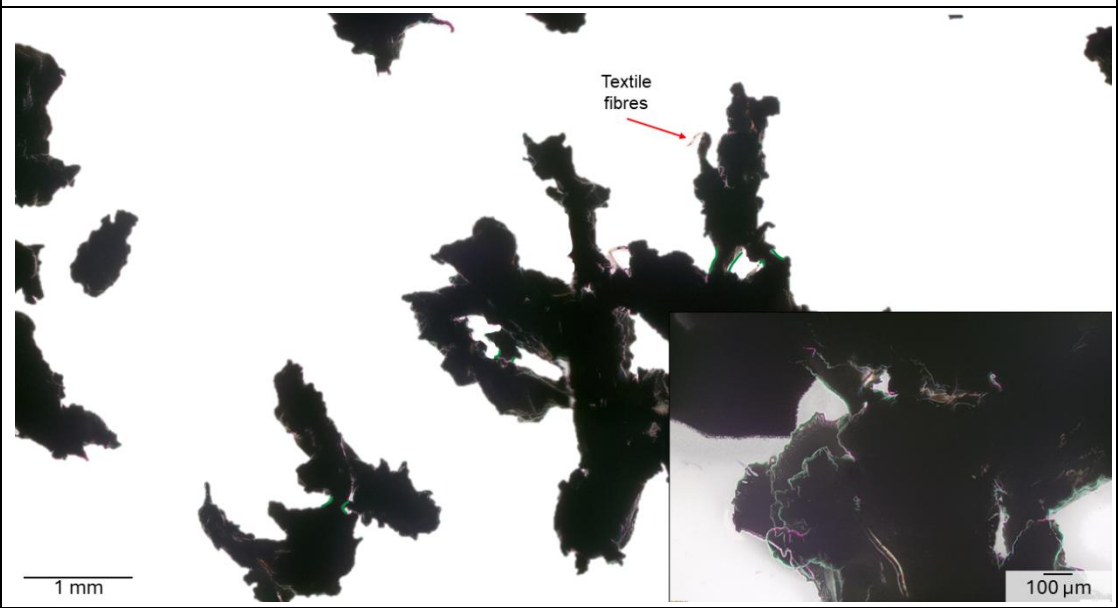
CT16_190C_1h	
CT16_190C_11h	
CT16_190C_36h	

Table 4.7: Optical microscope images of extracted conveyor belt-derived crumb rubber after digestion at 165 °C

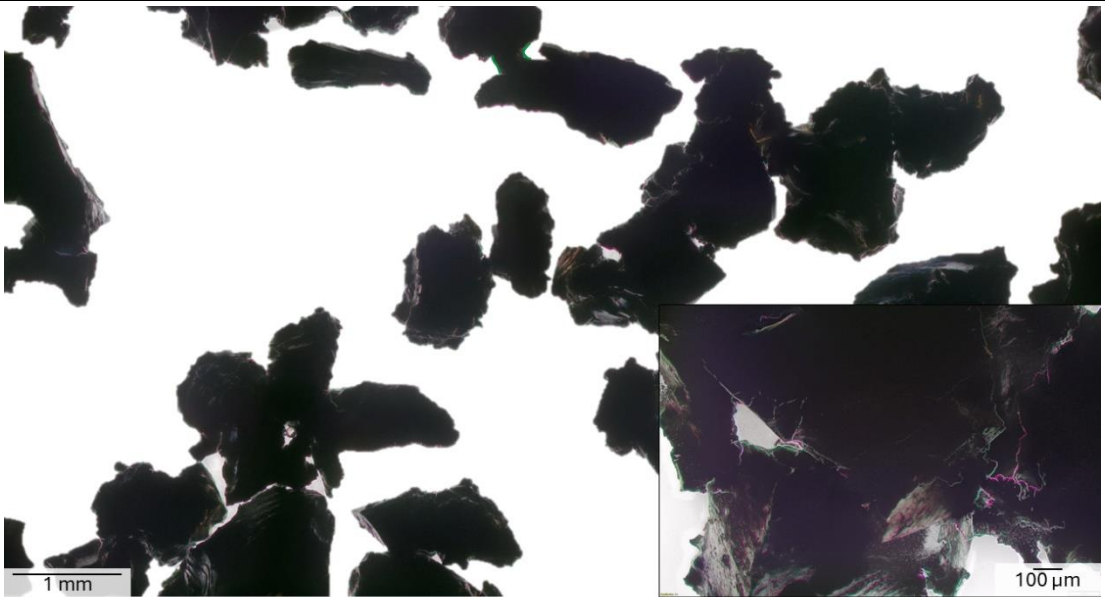
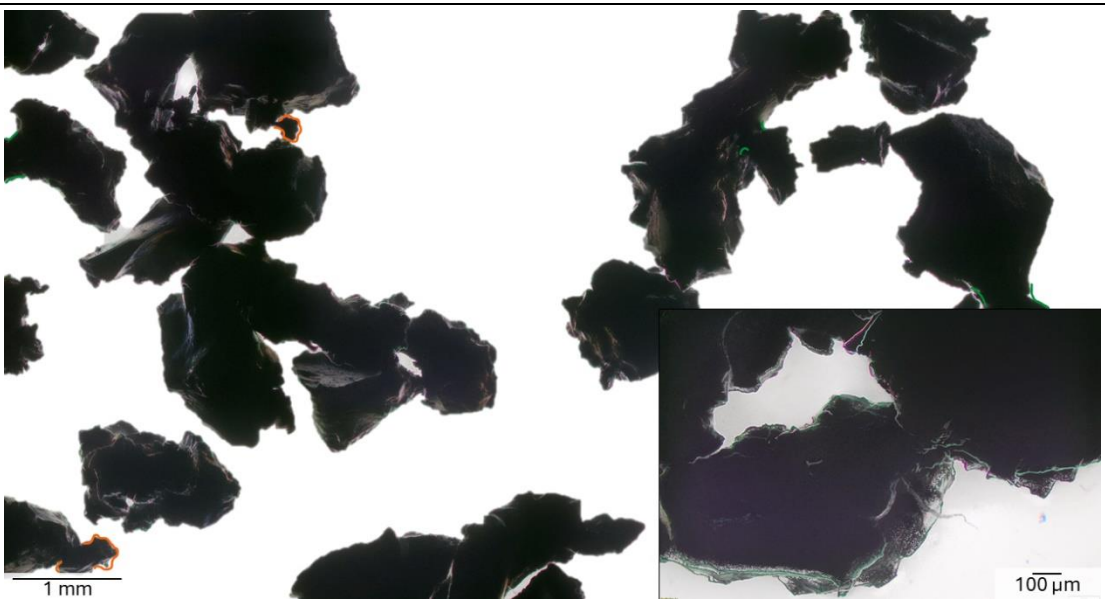
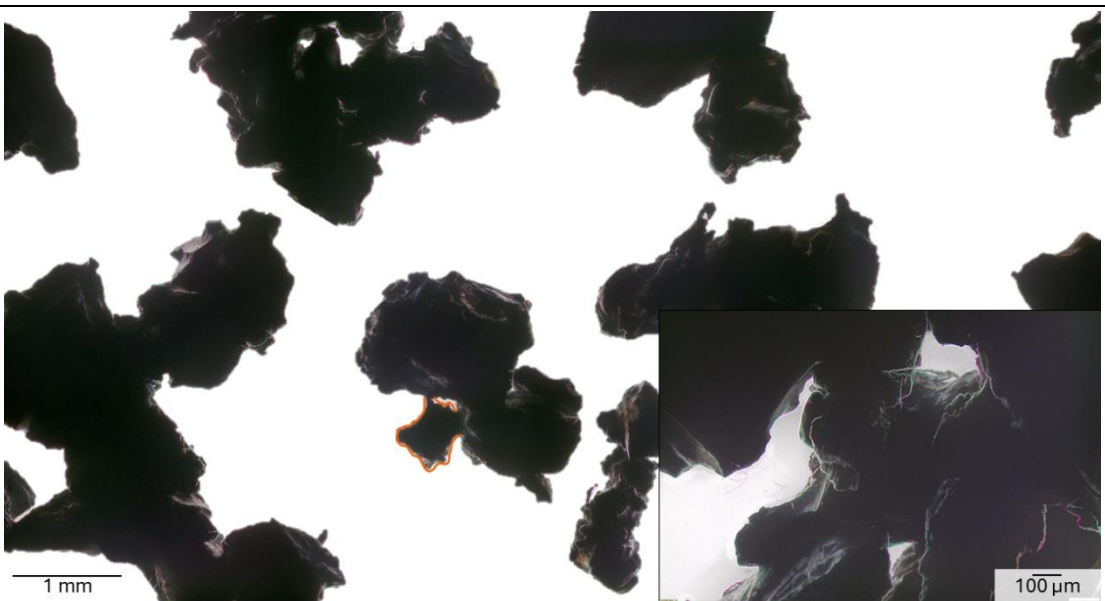
CB16_165C_1h	
CB16_165C_11h	
CB16_165C_36h	

Table 4.8: Optical microscope images of extracted conveyor belt-derived crumb rubber after digestion at 190 °C

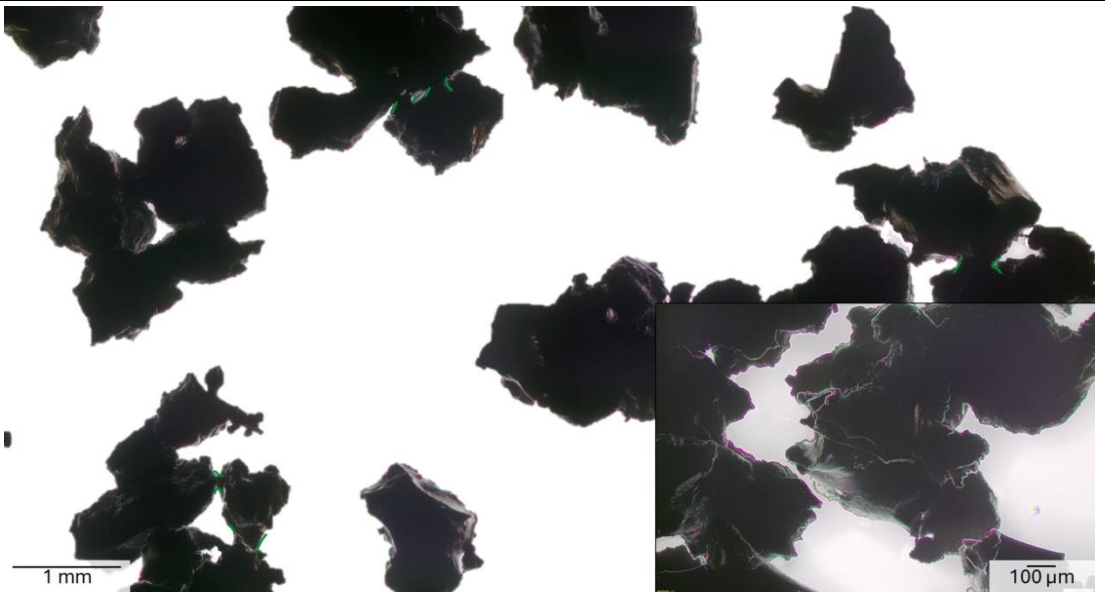
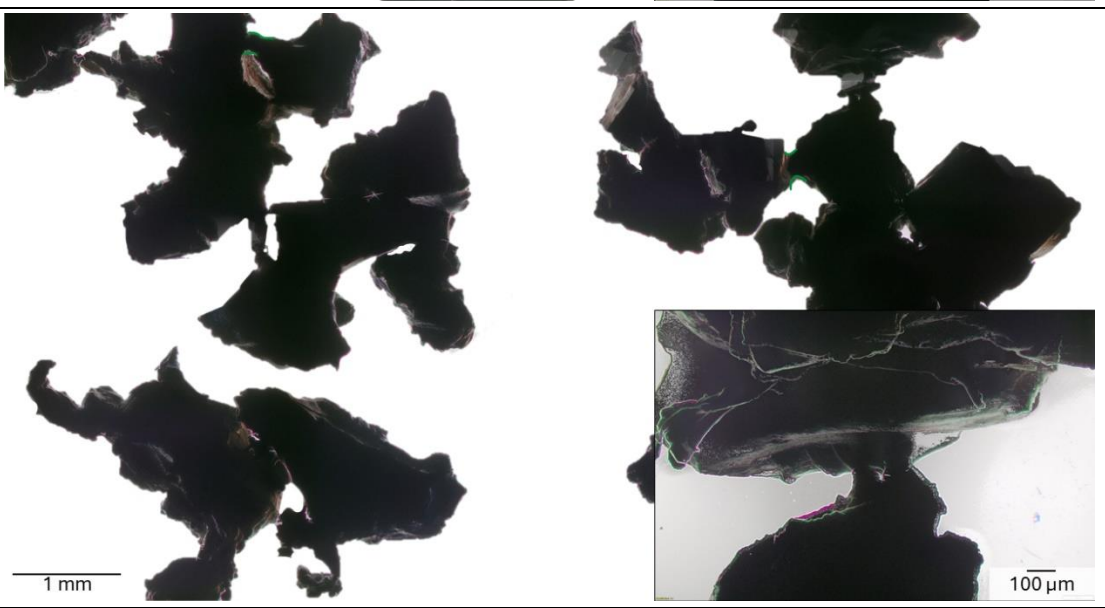
CB16_190C_1h	
CB16_190C_11h	
CB16_190C_36h	

Table 4.9: Optical microscope images of extracted mining tyre-derived crumb rubber after digestion at 165 °C

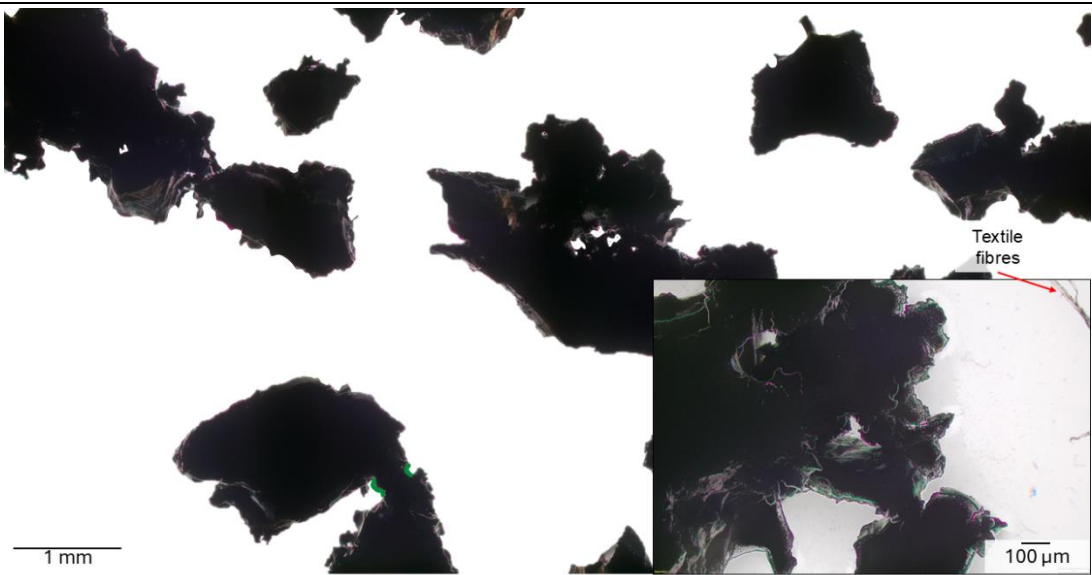
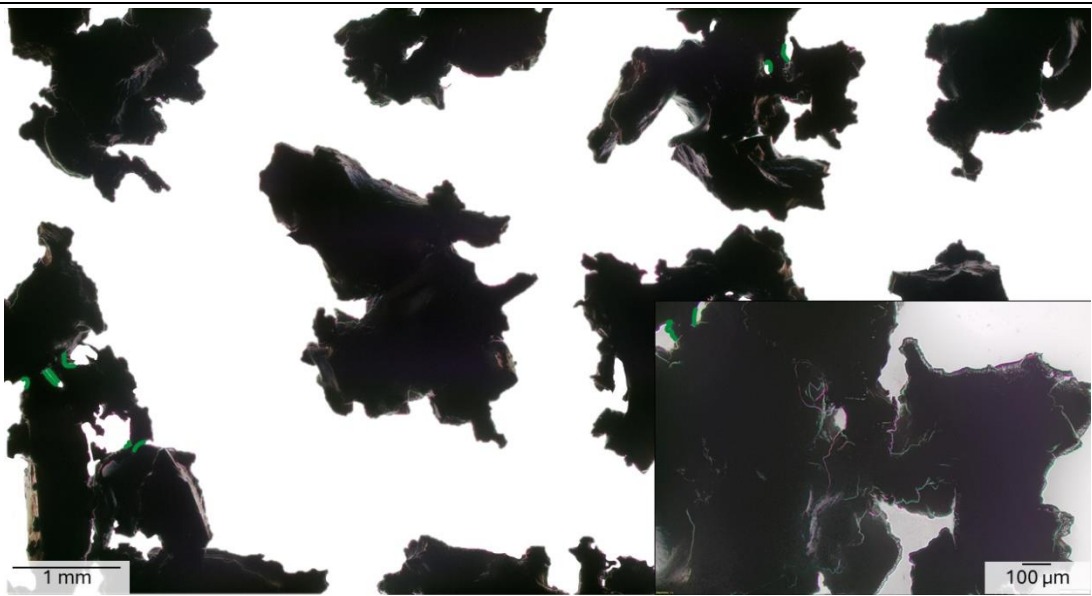
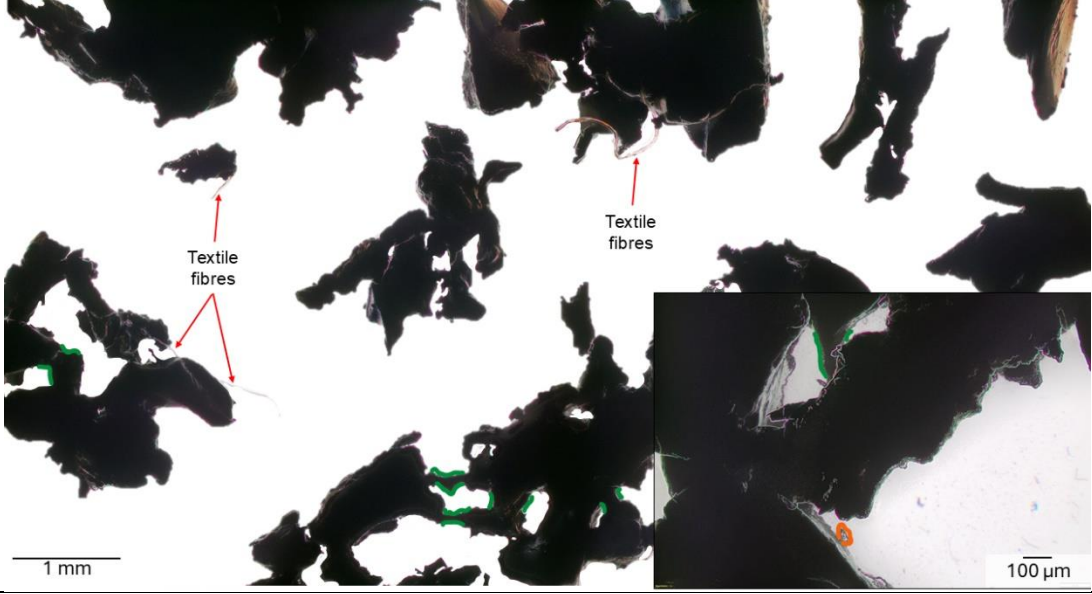
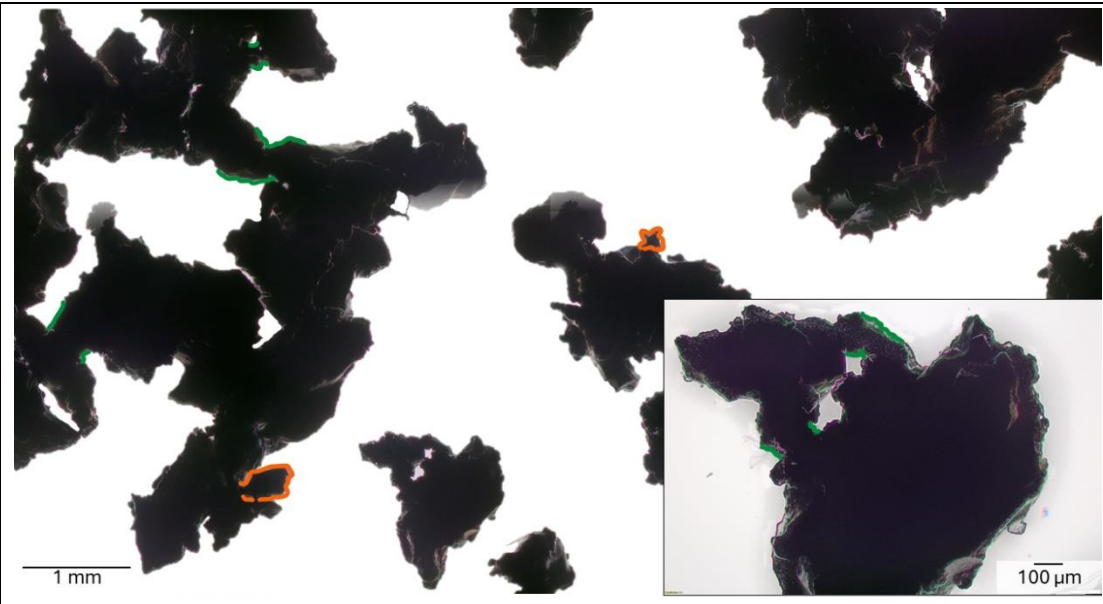
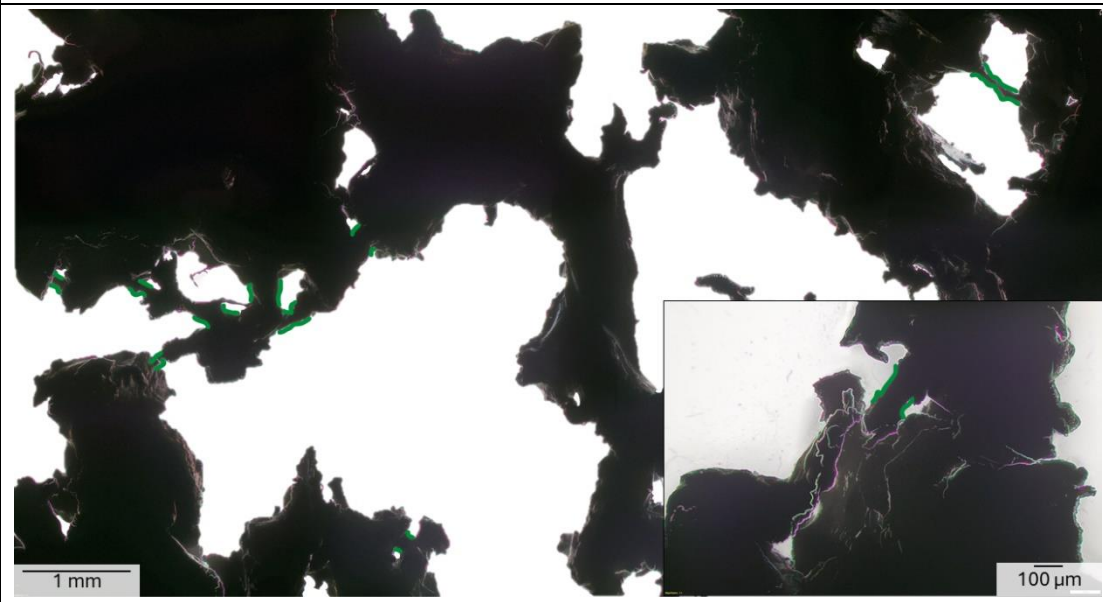
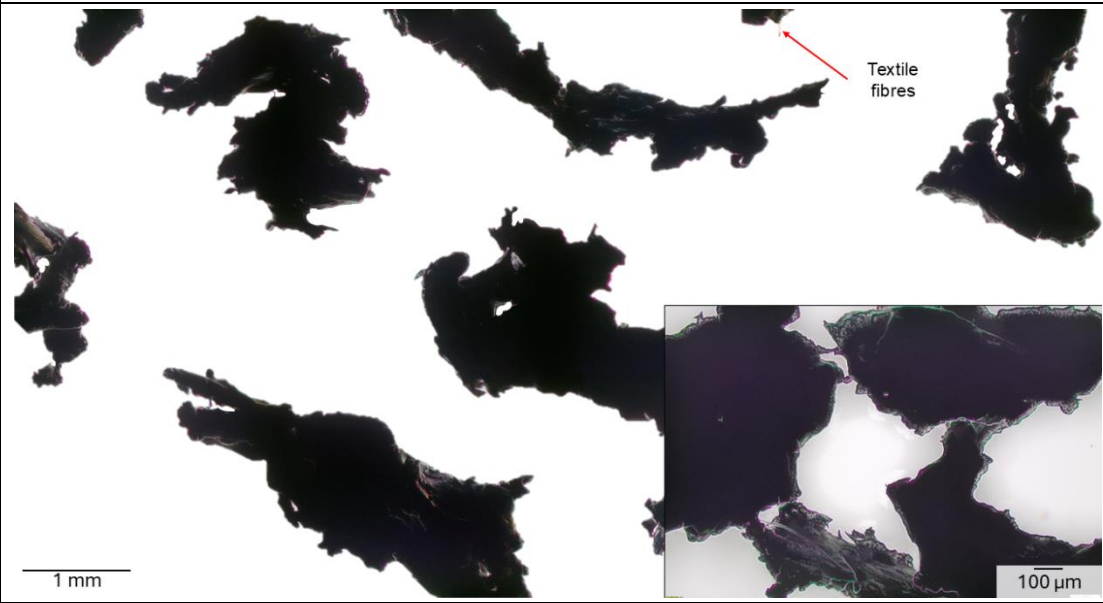
MT16_165C_1h	
MT16_165C_11h	
MT16_165C_36h	

Table 4.10: Optical microscope images of extracted mining tyre-derived crumb rubber after digestion at 190 °C

MT16_190C_1h	
MT16_190C_11h	
MT16_190C_36h	

4.2.7 Summary of Findings and Discussion

The different crumb rubbers investigated in this research were overall found to vary in their digestion behaviour as assessed using the methods described in Sections 3.3.3 to 3.3.7 and 3.3.9.

Swelling is a volume expansion process resulting from the absorption of the bitumen's light components. This mechanism typically stiffens the CRMBs due to both increased contact among the crumb rubber particles and the removal of the bitumen's light components from the liquid phase (Wang, Apostolidis et al. 2021). The swelling rate of the different types of rubber investigated in this research was found to vary; however, in all cases, swelling at 190 °C was more rapid when compared to that at 165 °C. At 165 °C, TR-derived rubber was found to swell the fastest followed by CB-, then MT- and, lastly, CT-derived rubber. At 190 °C, however, CB-derived rubber was found to swell more rapidly than TR-derived rubber.

The method described in Section 3.3.7 was found to not be sensitive enough to provide an accurate quantification for the dissolution of crumb rubber in bitumen. This was attributed to the various subsampling steps required to deliver the experimental program of this research (leading to minor but relevant sample rubber content inconsistencies) in combination with the comparatively small rubber mass variations expected due to dissolution. HPLC-GPC was found to be a more appropriate method to quantify the dissolution of rubber into bitumen. HPLC-GPC results revealed that MT-derived crumb rubber experienced an overall greater dissolution, followed by CT-derived, then by TR-derived and lastly by CB-derived crumb rubber.

TGA results were also challenging to use for the quantification of NR and SR dissolution in bitumen as they presented a notable variability within each sample group. This was attributed to the variability in EoL rubber composition (formulations proprietary to manufacturers) and variability in digestion stage of the crumb rubber particles analysed, accepting that within each CRMB swelling and dissolution of crumb rubber particles occur simultaneously due to particle size gradation (Wang, Apostolidis et al. 2021). TGA, however, was found to be a useful tool in understanding the variability among the as-received different rubber types used in this research and could provide insights relating to the suitability of various crumb rubber sources for use in bituminous binders as modifiers.

FTIR of the as-received crumb rubber particles revealed that the rubber types used in this research were chemically different according to their fingerprint regions. However, no significant evidence of crumb rubber presence in the liquid phase of the CRMBs could be identified using this method.

4.3 Crumb Rubber-Modified Binder Characteristics

This section presents the compressive limit at 70 °C results, which is a parameter used to determine the presence of undigested crumb rubber particles in CRMBs.

4.3.1 Compressive Limit at 70 °C

Table 4.11 presents the compressive limit at 70 °C results for all binder samples. Although this parameter is not reflective of a specific material property, it indicates the presence of crumb rubber particles in a bituminous binder. It measures the thickness of binders following exposure to an elevated temperature (70 °C) under extended loading. The compressive limit reported is the difference between the thickness of the sliding plates with the binder and their thickness without the presence of a specimen (ATM 132:2022).

According to Table 3.2, approximately 30% of S30 crumb rubber particles were between 0.6 and 1.18 mm, approximately 50% between 0.3 and 0.6 mm and approximately 15% between 0.15 and 0.3 mm. S16 primarily comprised particles between 0.6 and 1.18 mm (approximately 80%), while approximately 10% was between 1.18 and 2.36 mm. According to Table 4.11, the larger particles detected were smaller than the larger particles expected to be present in the sample in all cases.

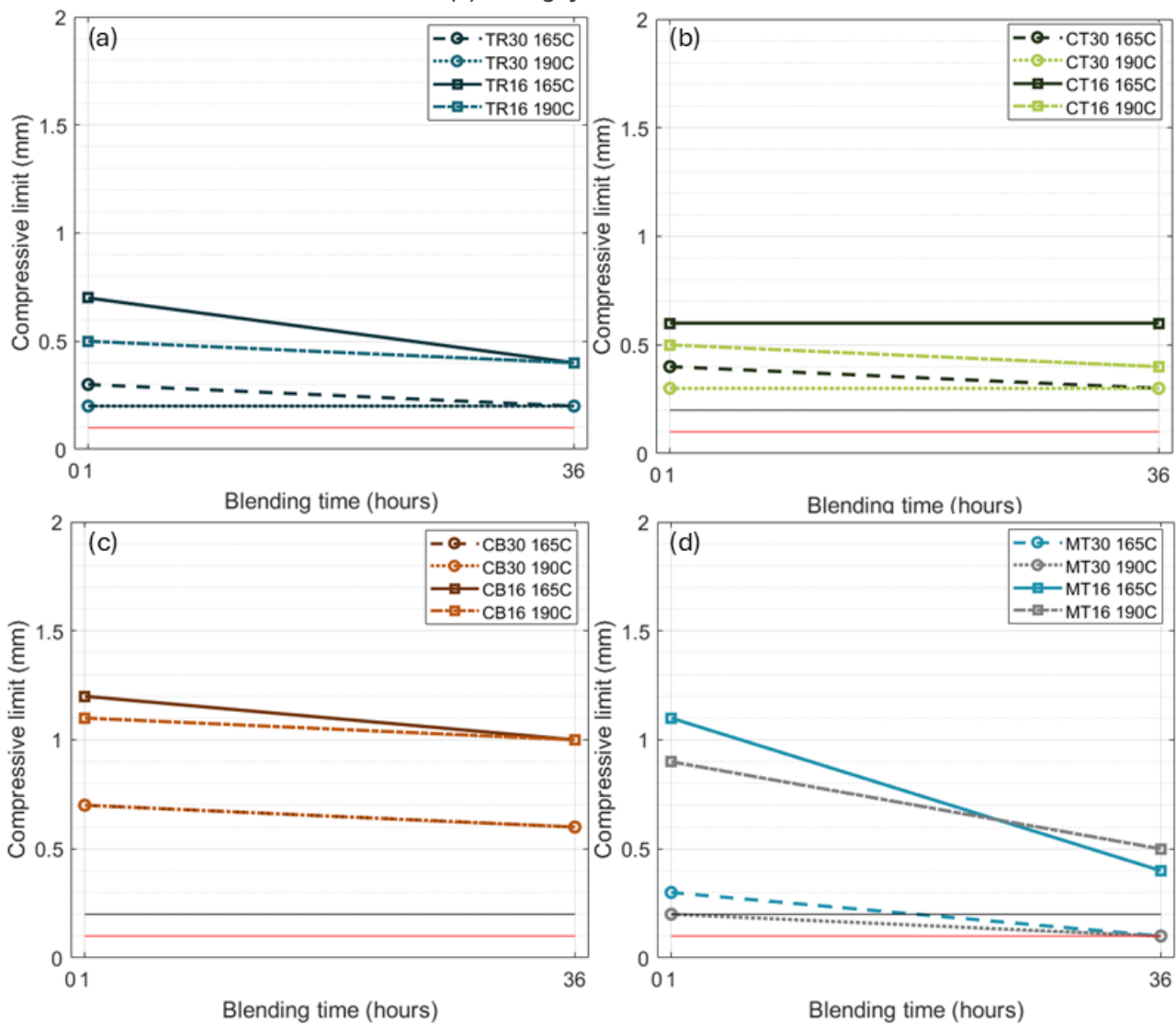
Table 4.11: Compressive limit at 70 °C results for all binders following 1 and 36 hours of blending; compressive limit in mm

Samples	Digestion time					
	1h	2h	4h	11h	24h	36h
TR30_165C_	0.3	–	–	–	–	0.2
TR30_190C_	0.2	–	–	–	–	0.2
TR16_165C_	0.7	–	–	–	–	0.4
TR16_190C_	0.5	–	–	–	–	0.4
CT30_165C_	0.4	–	–	–	–	0.3
CT30_190C_	0.3	–	–	–	–	0.3
CT16_165C_	0.6	–	–	–	–	0.6
CT16_190C_	0.5	–	–	–	–	0.4
CB30_165C_	0.7	–	–	–	–	0.6
CB30_190C_	0.7	–	–	–	–	0.6
CB16_165C_	1.2	–	–	–	–	1.0
CB16_190C_	1.1	–	–	–	–	1.0
MT30_165C_	0.3	–	–	–	–	0.1
MT30_190C_	0.2	–	–	–	–	0.1
MT16_165C_	1.1	–	–	–	–	0.4
MT16_190C_	0.9	–	–	–	–	0.5

Figure 4.41 graphically presents the compressive limit at 70 °C measurements of Table 4.11 for all CRMBs assessed in this research against the minimum requirements from MRWA Specification 511:2025 and ATS 3110:2023.

TR-, CT- and CB-derived binders were found to meet the minimum requirements of both MRWA Specification 511:2025 and ATS 3110:2023. However, the compressive limit at 70 °C of MT30_165C_36h and MT30_190C_36h was measured to be 0.1 mm, which is below the minimum requirements of MRWA Specification 511:2025. In all cases, binders blended with S16 particles were found to have a greater compressive limit at 70 °C, as anticipated based on the crumb rubber particle gradation introduced.

Figure 4.41: Blending time versus compressive limit at 70 °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 minimum requirement according to Austroads ATS 3110:2023 and black line denotes minimum requirement according to MRWA Specification 511:2025.

Table 4.12 presents the ANOVA results for compressive limit at 70 °C. According to these results, all investigated parameters, except for blending temperature, were found to have a statistically significant effect on the compressive limit at 70 °C results. However, provided that the particles were of different gradation when introduced in the binder, the significance of crumb rubber size may be disregarded in this instance as the samples were different by design rather than as a result of the blending process.

Table 4.12: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on compressive limit at 70 °C

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	1.24750	0.41583	26.07	0.000
Size	1	1.05125	1.05125	65.91	0.000
Temperature	1	0.03125	0.03125	1.96	0.174
Time	1	0.21125	0.21125	13.24	0.001
Error	25	0.39875	0.01595		
Total	31	2.94000			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

Figure 4.42 presents the main effect plots for the compressive limit at 70 °C results. It shows that CB-derived CRMBs resulted at an increased compressive limit at 70 °C. In addition, binders prepared with S16 crumb rubber were found to have a greater compressive limit at 70 °C. This is not surprising as, according to Table 3.2, S16 crumb rubber has a greater concentration of larger size particles to begin with. An increase in blending time was also found to result in a decrease in compressive limit at 70 °C.

Figure 4.42: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on compressive limit at 70 °C

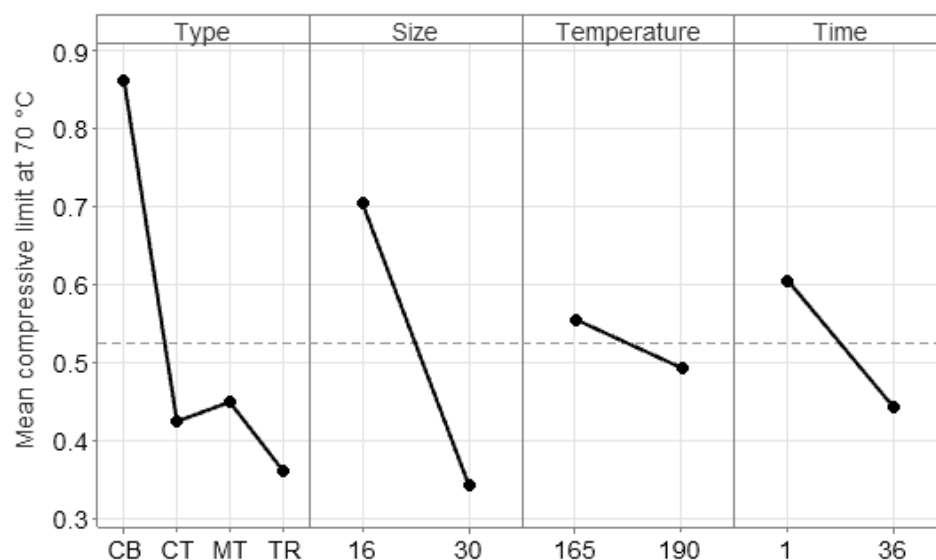
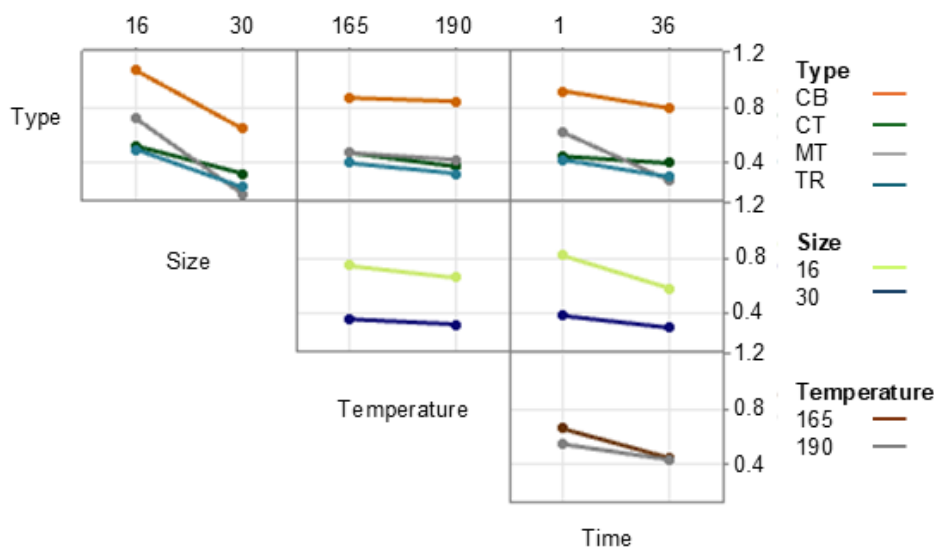


Figure 4.43 presents the interaction plots among the different parameters investigated and their impacts on the compressive limit at 70 °C results. Although the investigated parameters were found to impact the results to a different extent, no notable interactions between any of the investigated parameters can be observed.

Figure 4.43: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for compressive limit at 70 °C

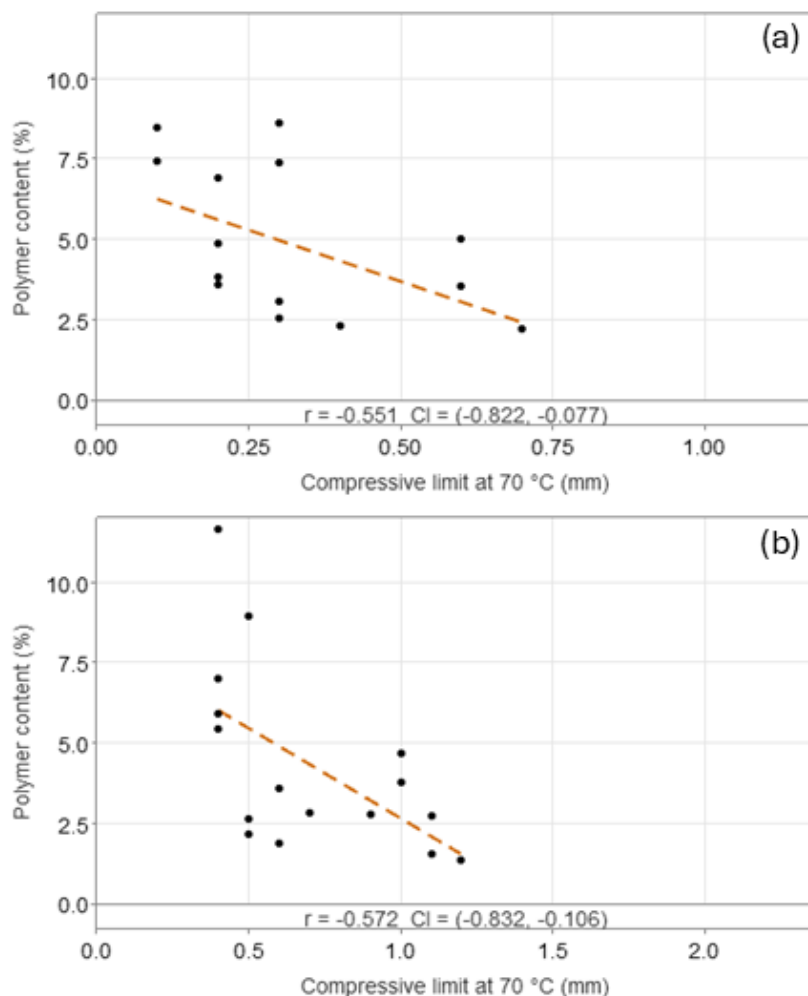


4.3.2 Summary of Findings and Discussion

The compressive limit at 70 °C results revealed an overall decrease in particle size with increasing blending time and, to a smaller degree, with increasing temperature. These observations are in line with expectations. Importantly, TR-, CT- and CB-derived CRMBs all met the limits specified in MRWA Specification 511:2025 and ATS 3110:2023.

The matrix plots for S30 and S16 compressive limit at 70 °C against polymer content are presented in Figure 4.44. The measured decrease in compressive limit at 70 °C was found to have a moderate correlation ($0.40 \leq r \leq 0.69$) with polymer dissolution as measured by HPLC-GPC. Although it is recognised that a single data point for each condition, as acquired in this research, may not be sufficient to ensure the robustness of this correlation, it may still be useful to communicate that compressive limit at 70 °C, a method already used for the characterisation of CRMBs in MRWA Specification 511, can provide some insights regarding the comparative dissolution of different rubber types in the binder.

Figure 4.44: Correlation between compressive limit at 70 °C and polymer content as measured by HPLC-GPC for (a) S30 and (b) S16



As mentioned, compressive limit at 70 °C results provide insights regarding the presence of crumb rubber particles in the binder and an indication of the largest particle sizes present. These could be used to inform asphalt mix design, predominantly the necessity for air voids to allow for crumb rubber particles. They could further inform asphalt compactability as the presence of undigested elastic crumb rubber particles is known to lead to the expansion of specimens immediately after compaction (Grobler 2020).

The comparatively larger CB-derived crumb rubber particles as well as MT16-derived crumb rubber particles at early digestion stages (1 hour of blending) means that their suitability for use in existing mixes (including OGA and GGA) needs to be further investigated prior to adoption as business-as-usual.

4.4 Handling Properties of Crumb Rubber-Modified Binders

This section presents the results for viscosity at 165 and 175 °C and loss on heating, describing the handling parameters of CRMBs. The viscosity at 165 and 175 °C is a measure of the ability of the binder to be pumped during production, whereas the loss on heating parameter indicates whether the binder is safe to handle at elevated temperatures typical for binder and asphalt production.

4.4.1 Viscosity at 165 and 175 °C

Table 4.13 presents the viscosity at 165 °C results for all binders as well as those for unmodified C170 as-received and following blending. It is worth noting that the current version of the rotational viscosity test method does not set the shear rate used during a test (which is controlled by the rotational speed (in rpm) of

the test spindle). The equipment used in the rotational viscosity test can only measure torque within a limited range, which is then converted into viscosity results. This torque range is denoted as between 0 and 100% torque on the instrument. As a consequence of the limited torque range of the instrument, samples of greater viscosity may need to be tested at lower spindle speeds (i.e. lower shear rates) than samples of comparatively lower viscosity. This may affect the results obtained for non-Newtonian fluids such as CRMBs. Therefore, the viscosity results along with the rpm used for testing of all samples are listed in Appendix C.1 for reference.

In addition, as expressed in Section 3.2.1, for each crumb rubber type and size blended at each temperature, 2 blends were prepared, one which was subsampled following 1 and 2 hours of blending and removed from the oven after 4 hours and one that was subsampled at 11 and 24 hours of blending and taken out of the oven after 36 hours. As it may be understood, this effectively changed the headspace in the blending containers, potentially allowing the binder to oxidise even though a CO₂ blanket was applied. Such oxidation can affect the viscosity results. The possible oxidation of the binders for blending times of 1, 4, 11 and 36 hours and any potential effects this had on the binder properties is separately discussed in Section 5.1.

From Table 4.13, the viscosity of the C170 appears to only be slightly affected by the blending time with the only notable change being observed after 36 hours of blending at 165 °C and after 24 hours of blending at 190 °C. The viscosity of the CRMBs does not present any consistent trends with time across the different crumb rubber types. As expected, the viscosity at 165 °C of the CRMBs is greater than that of the C170. Bahia and Davies (1994) explained that this behaviour is common due to the introduction of crumb rubber particles, which remain in a solid state under these temperatures, as well as due to their swelling during blending, as discussed in Section 2.2.

Table 4.13: Viscosity at 165 °C test results; results in Pa·s

Samples	Digestion time						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_	–	1.67	1.68	2.35	1.78	1.86	2.33
TR30_190C_	–	4.17	3.80	5.28	2.88	2.64	2.04
TR16_165C_	–	1.24	1.26	1.73	1.44	1.46	1.64
TR16_190C_	–	2.85	1.60	2.43	2.31	1.62	1.15
CT30_165C_	–	2.35	2.24	2.50	1.78	2.65	2.83
CT30_190C_	–	3.75	2.87	2.88	3.56	2.10	1.85
CT16_165C_	–	1.16	1.53	1.38	1.28	1.35	1.74
CT16_190C_	–	1.64	1.35	1.86	2.53	1.43	1.73
CB30_165C_	–	2.28	2.42	2.31	1.80	2.37	2.68
CB30_190C_	–	2.95	2.85	2.92	3.07	3.02	3.88
CB16_165C_	–	0.75	0.82	1.11	2.79	1.56	2.87
CB16_190C_	–	0.87	1.27	1.32	1.50	4.48	2.29
MT30_165C_	–	2.78	4.67	17.58	12.35	13.88	0.74
MT30_190C_	–	18.26	14.47	13.71	11.62	2.95	1.27
MT16_165C_	–	0.95	2.11	8.53	5.57	15.85	1.19
MT16_190C_	–	7.32	14.89	14.28	12.19	3.12	1.46
C170	0.11	–	–	–	–	–	–
C170_165C_	–	0.12	–	–	–	–	0.19
C170_190C_	–	0.13	0.13	0.14	0.13	0.18	0.46

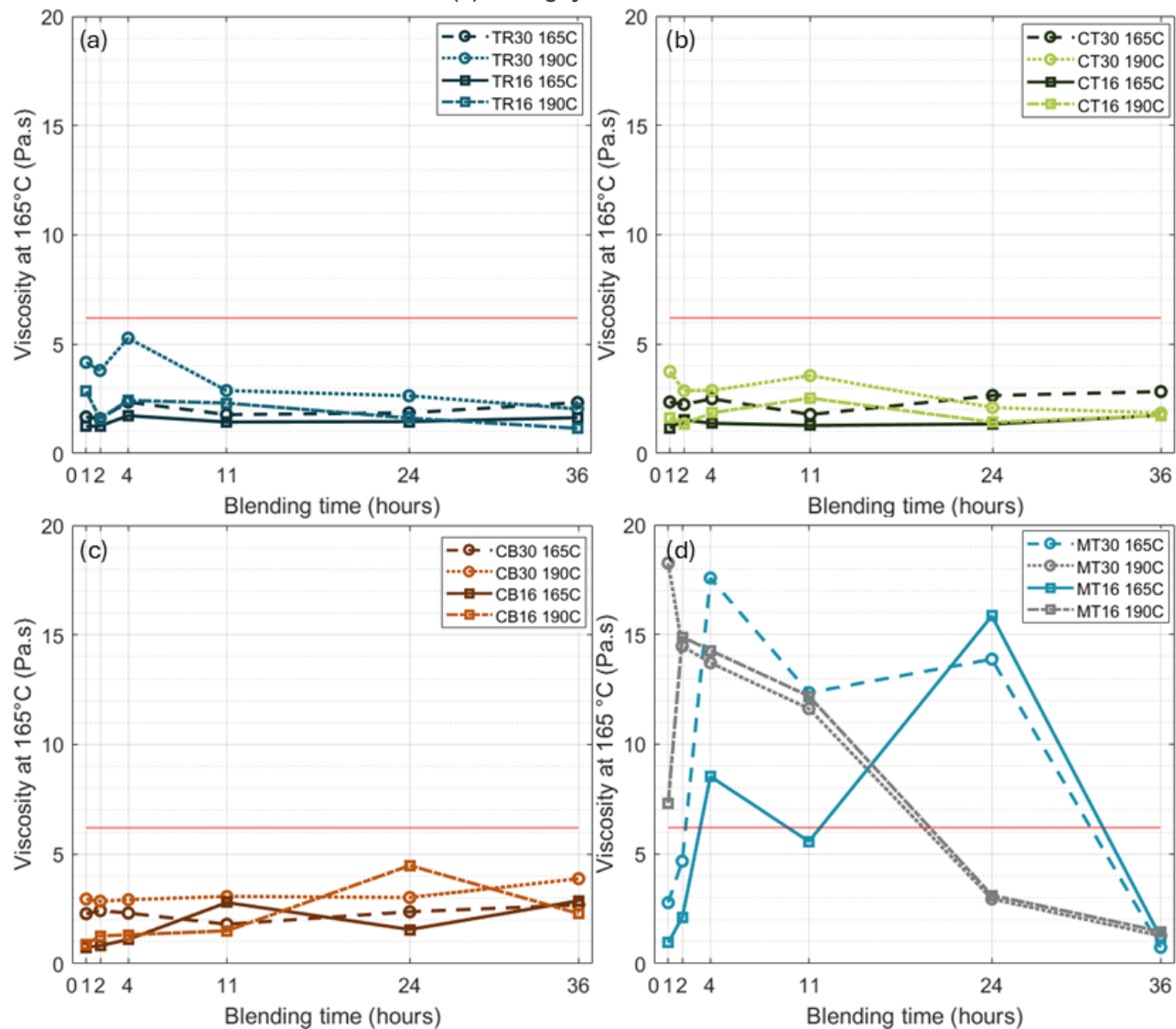
Figure 4.45 graphically presents the viscosity at 165 °C results of Table 4.13 along with the Austroads ATS 3110:2023 allowable upper limit.

The viscosity at 165 °C of TR-, CT- and CB-derived CRMBs was found to be below the maximum limit set by Austroads ATS 3110:2023; however, the viscosity at 165 °C of some MT-derived CRMBs was notably above

this limit. Specifically, the viscosity at 165 °C of MT-derived CRMBs blended at 190 °C was only found to be below 6.2 Pa.s following 24 and 36 hours of blending. The conformance of MT-derived CRMBs blended at 165 °C varied with blending time, whereby MT30_165C blended for 1, 2 and 36 hours and MT16_165C blended for 1, 2, 11 and 36 hours were found to have viscosity at 165 °C below 6.2 Pa.s.

The viscosity at 165 °C for MT-derived CRMBs presented a variance of 17.52 Pa.s depending on the selected parameters, while that for TR-, CT- and CB-derived CRMBs was notably narrower at 4.13, 2.59 and 3.73 Pa.s, respectively.

Figure 4.45: Blending time versus viscosity at 165 °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 maximum requirement.

Table 4.14 presents the ANOVA results for viscosity at 165 °C. Table 4.14 shows that all investigated parameters had a statistically significant impact on the viscosity at 165 °C results.

Table 4.14: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on viscosity at 165 °C

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	2,428.70	809.568	91.78	0.000
Size	1	108.06	108.057	12.25	0.001
Temperature	1	55.21	55.214	6.26	0.013
Time	5	269.35	53.870	6.11	0.000
Error	286	2,522.80	8.821		
Lack of fit	85	2,487.39	29.263	166.07	0.000
Pure error	201	35.42	0.176		
Total	296	5,358.26			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

From the main effects plots of Figure 4.46, it is shown that the viscosity at 165 °C for TR-, CT- and CB-derived CRMBs was overall comparable, while that of MT-derived CRMBs was overall notably greater. An increase in blending temperature from 165 to 190 °C and a change in crumb rubber particle gradation from S16 to S30 were found to result in an increase in viscosity at 165 °C. Lastly, viscosity at 165 °C as a result of blending time was found to fluctuate, but overall, the greatest viscosities may be expected following blending for 4 hours, whereas the lowest viscosities may be expected following blending for 36 hours.

Figure 4.46: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on viscosity at 165 °C

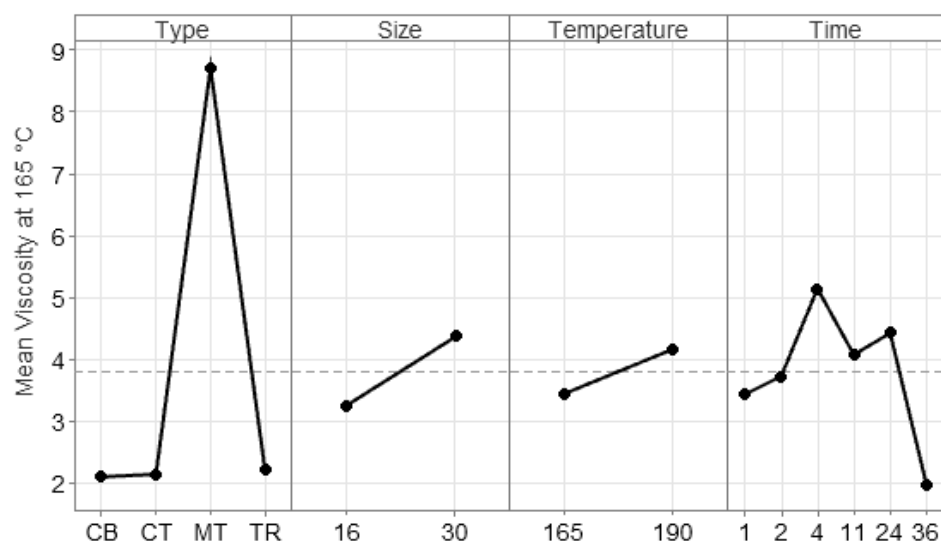


Figure 4.47 reveals only a few interactions among the investigated parameters. Those may be observed between blending time and all other parameters. Between blending time and crumb rubber type, the most pronounced interactions are between MT-derived CRMBs and all others blended between 24 and 36 hours, where the viscosity at 165 °C of MT-derived CRMBs decreases significantly.

Interactions between blending time and crumb rubber size can also be observed, predominantly for blending between 11 and 24 hours, where viscosity at 165 °C for CRMBs produced with S16 crumb rubber particles increases, whereas viscosity at 165 °C for CRMBs produced with S30 crumb rubber particles decreases.

Lastly, the viscosity at 165 °C for CRMBs blended at 165 °C was found to increase for blending between 11 and 24 hours, whereas the viscosity at 165 °C of CRMBs blended at 190 °C was found to decrease.

Figure 4.47: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for viscosity at 165 °C

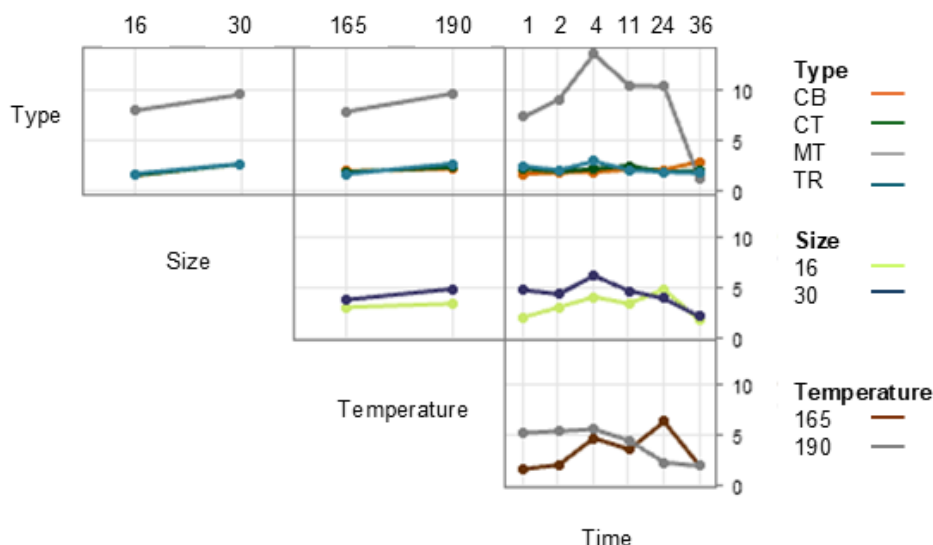


Table 4.15 shows the viscosity at 175 °C results for all binders assessed. In agreement with observations of the assessment of the viscosity at 165 °C, the viscosity at 175 °C results also fluctuate with blending time, maintaining interacting trends. As expected, due to the presence of crumb rubber particles with a greater melting temperature to bitumen (Bahia & Davies 1994), the viscosity at 175 °C for all CRMBs was greater than that of the C170.

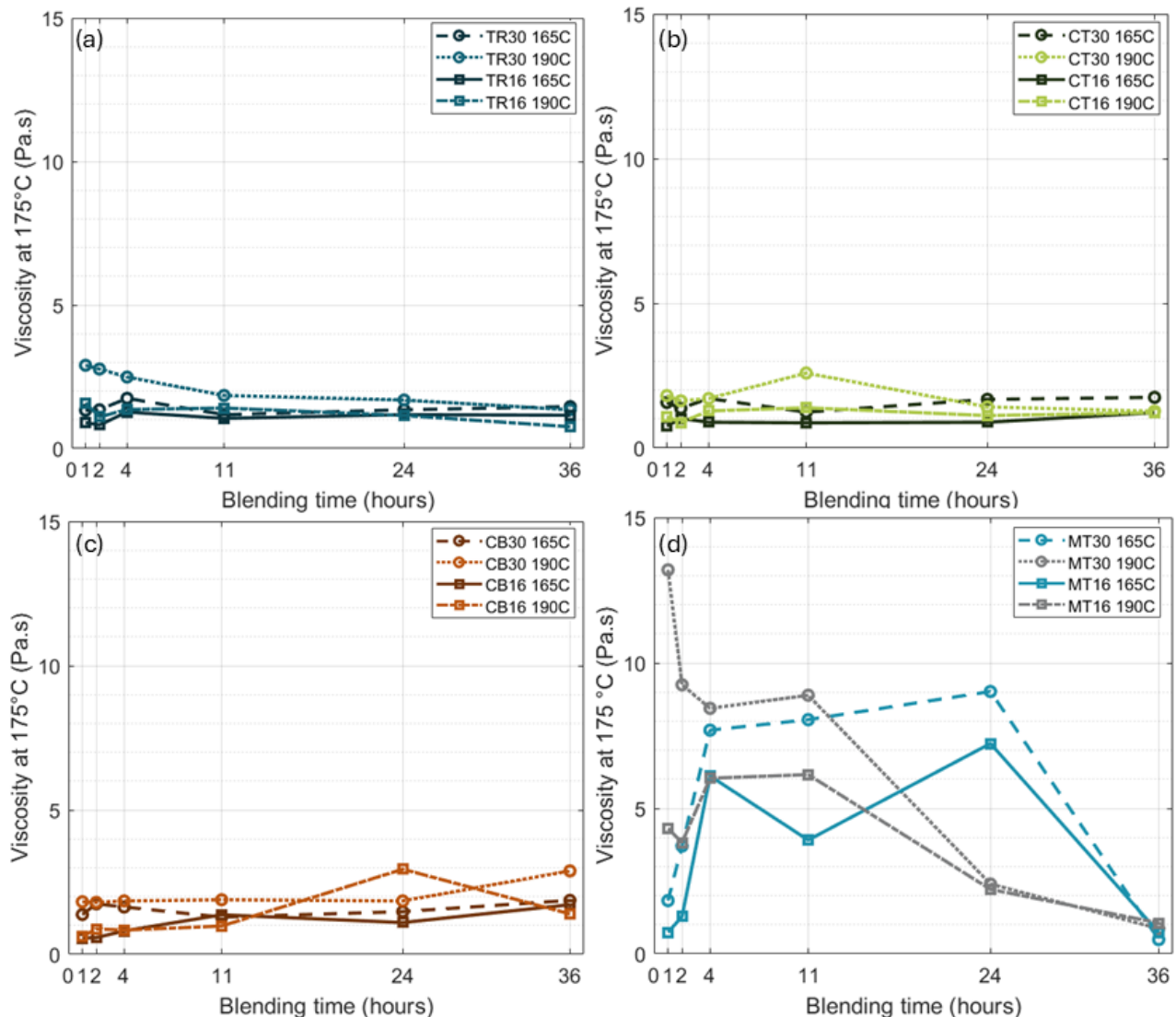
Table 4.15: Viscosity at 175 °C test results; results in Pa·s

Samples	Digestion time						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_	–	1.33	1.35	1.75	1.18	1.35	1.46
TR30_190C_	–	2.90	2.77	2.49	1.85	1.69	1.35
TR16_165C_	–	0.90	0.82	1.27	1.05	1.18	1.16
TR16_190C_	–	1.59	1.10	1.36	1.41	1.15	0.76
CT30_165C_	–	1.57	1.37	1.71	1.24	1.68	1.75
CT30_190C_	–	1.81	1.63	1.71	2.59	1.42	1.27
CT16_165C_	–	0.76	1.02	0.89	0.87	0.89	1.22
CT16_190C_	–	1.08	0.88	1.28	1.39	1.12	1.22
CB30_165C_	–	1.38	1.75	1.64	1.29	1.48	1.87
CB30_190C_	–	1.82	1.80	1.85	1.89	1.85	2.89
CB16_165C_	–	0.56	0.58	0.81	1.37	1.10	1.72
CB16_190C_	–	0.62	0.88	0.84	0.98	2.95	1.40
MT30_165C_	–	1.83	3.71	7.69	8.05	9.02	0.49
MT30_190C_	–	13.20	9.25	8.44	8.89	2.40	0.87
MT16_165C_	–	0.74	1.28	6.11	3.91	7.23	0.73
MT16_190C_	–	4.31	3.82	6.04	6.16	2.22	1.06
C170	0.08	–	–	–	–	–	–
C170_165C_	–	0.09	–	–	–	–	0.13
C170_190C_	–	0.10	0.10	0.10	0.10	0.13	0.29

Figure 4.48 graphically presents the viscosity at 175 °C for all CRMBs assessed in this research. There are currently no upper or lower limits specified for viscosity at 175 °C as measured following ATM 111, with MRWA Specification 511:2025 requiring for the value to be reported.

The viscosity at 175 °C for MT-derived CRMBs presented a variance of 12.71 Pa.s depending on the selected parameters, whereas that for TR-, CT- and CB-derived CRMBs was notably narrower at 2.14, 1.83 and 2.39 Pa.s, respectively.

Figure 4.48: Blending time versus viscosity at 175 °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



The ANOVA results of Table 4.16 show that all investigated parameters significantly affected the viscosity at 175 °C results, as was also found for viscosity at 165 °C.

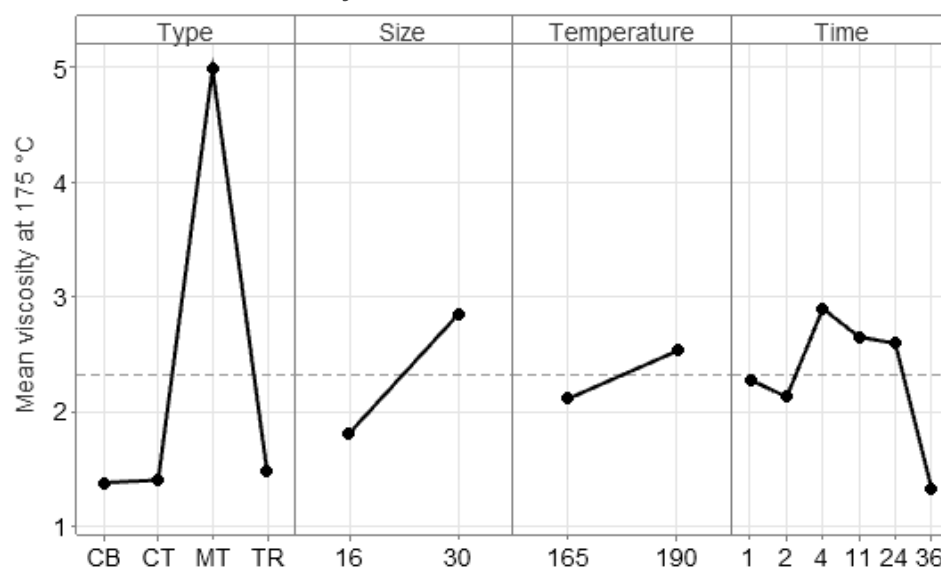
Table 4.16: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on viscosity at 175 °C

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	729.71	243.236	91.05	0.000
Size	1	89.53	89.531	33.51	0.000
Temperature	1	17.66	17.659	6.61	0.011
Time	5	75.50	15.101	5.65	0.000
Error	286	764.07	2.672		
Lack of fit	85	751.81	8.845	145.00	0.000
Pure error	201	12.26	0.061		
Total	296	1,662.84			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

The main effect plots of Figure 4.49 reveal that the results of TR-, CT- and CB-derived CRMBs were comparable, whereas those of MT-derived binders were notably greater overall. In addition, an increase in crumb rubber particle size gradation and a decrease in blending temperature resulted in a decrease in the measured viscosity at 175 °C of the binders. When considering blending time, the greatest viscosity at 175 °C was measured following 4 hours of blending and the lowest following 36 hours of blending. These findings agree with those for viscosity at 165 °C.

Figure 4.49: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on viscosity at 175 °C



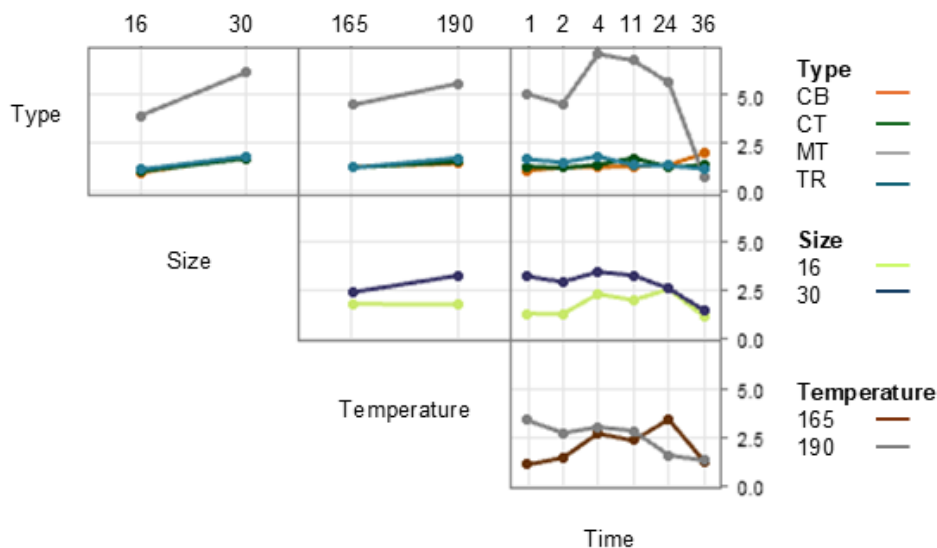
Very few interactions between the investigated parameters can be observed in Figure 4.50. The crumb rubber type was only found to interact with blending time and, most notably, between 24 and 36 hours of blending where the viscosity at 175 °C of TR- and CT-derived CRMBs was found to remain relatively constant, that of CB-derived CRMBs was found to slightly increase, and that of MT-derived CRMBs was found to notably decrease.

There was also some interaction between crumb rubber size gradation and blending temperature, where the viscosity at 175 °C of CRMBs produced with S16 particles remained unchanged irrespective of blending temperature and that of CRMBs produced with S30 CRMBs was found to increase with an increase in temperature. Crumb rubber particle gradation also showed some interactions with blending time between 11 and 24 hours of blending where the viscosity at 175 °C of CRMBs produced with S30 crumb rubber particles was found to decrease and that of CRMBs produced with S30 CRMBs was found to slightly increase.

Lastly, the viscosity at 175 °C presented some interaction between blending temperature and time between 2 and 4 hours of blending and between 11 and 24 hours of blending. In both cases, the viscosity at 175 °C of

CRMBs produced at 165 °C was found to increase, whereas that of CRMBs produced at 190 °C was found to decrease.

Figure 4.50: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for viscosity at 175 °C



Overall, the trends in the viscosity at 165 °C results were found to be consistent with those in the viscosity at 175 °C results. Furthermore, viscosity increases occurred for CRMBs with increasing blending temperature and decreasing particle size. This is in line with expectations.

The insights from the ANOVA were also found to be consistent between the 2 test temperatures. This is not surprising as the same specimen was tested under both temperature conditions for each of the samples. In addition, the ATM 111-2022 test method recommends that the torque reading during viscosity testing should be between 50 and 100% of the maximum torque achievable by the rotational viscometer in use. This torque reading is typically achieved following an optimisation process. The process, however, allows for testing to be undertaken using different rotational speeds. This does not impact the viscosity readings where the sample tested behaves as a Newtonian fluid. This torque reading optimisation process, however, would affect viscosity results for non-Newtonian fluids where the viscosity of the material is dependent on the shear rate, such as the CRMBs tested in this research. The speed of the spindle during the viscosity measurements for all tests conducted in this research, as recorded in Appendix C.1, varied between 5 and 100 rpm. In all cases, though, the rotational speed of the spindle, when the viscosity at 165 °C was recorded, was equal or lower than that of when the viscosity at 175 °C was recorded. This also contributed to this consistency in the ANOVA observations.

Austrroads ATS 3110:2023 sets an upper limit for viscosity at 165 °C, and this was surpassed by the results of some of the MT-derived CRMBs. According to Austrroads (2021b), this limit was determined based on existing limits for viscosity at 175 °C according to Equation 3:

$$\text{Viscosity at 165 °C} = 1.545 \times \text{Viscosity at 175 °C}$$

3

None of the results of this research follow Equation 3. If a 5% error on 1.545 is allowed, then 30 results comply with Equation 3, while the rest are either above (24 results) or below (42 results) this range. This could be related to the previously discussed variability in the rotational speed of the spindle during the viscosity measurements, which was not always proportional.

When compared to the other types of rubber, MT-derived CRMBs presented not only the greatest viscosities between 1 and 24 hours of blending but also the greatest parameter dependency. Testing was repeated for those apparent outliers. The results are presented in Appendix C.5. Unlike repeat testing for CB16_190C_11h presented in Appendix C.4, repeat testing for MT-derived CRMBs did not consistently improve confidence in the findings, with results either being comparable to those presented in this section or

significantly different. In all cases, they demonstrate a dependence to the speed of the spindle (shear rate dependency), as expected. Table C.6 shows that where the results from the repeated testing were recorded to be comparable to those of the first test, so was the speed of the spindle during the measurement. However, a decrease in the spindle speed during the measurement resulted in a proportional increase in the measured viscosity and vice versa.

The key takeaway from these results is that TR-, CT- and CB-derived CRMBs met the limits set by Austroads ATS 3110:2023, exhibiting little difference in their viscosity results. The MT-derived CRMBs yielded inconsistent and, often, non-complying behaviour.

4.4.2 Loss on Heating

Loss on heating (or mass change) after RTFO treatment is a measure of volatile loss during heating. This property is used to quickly estimate the potential of worker exposure to potentially harmful volatiles when polymer modifiers are incorporated in bituminous binders (ATM 103:2022). The temperature of the oven during RTFO treatment was set to 165 ± 0.5 °C, according to ATM 103:2022.

Table 4.17 presents the mass loss on heating results for all binders investigated following blending for 1 and 36 hours. The greatest loss on heating was measured for MT16_190C_1h and the lowest for TR16_190C_36h and CT30_190C_36h at 0.82 and 0.02%, respectively.

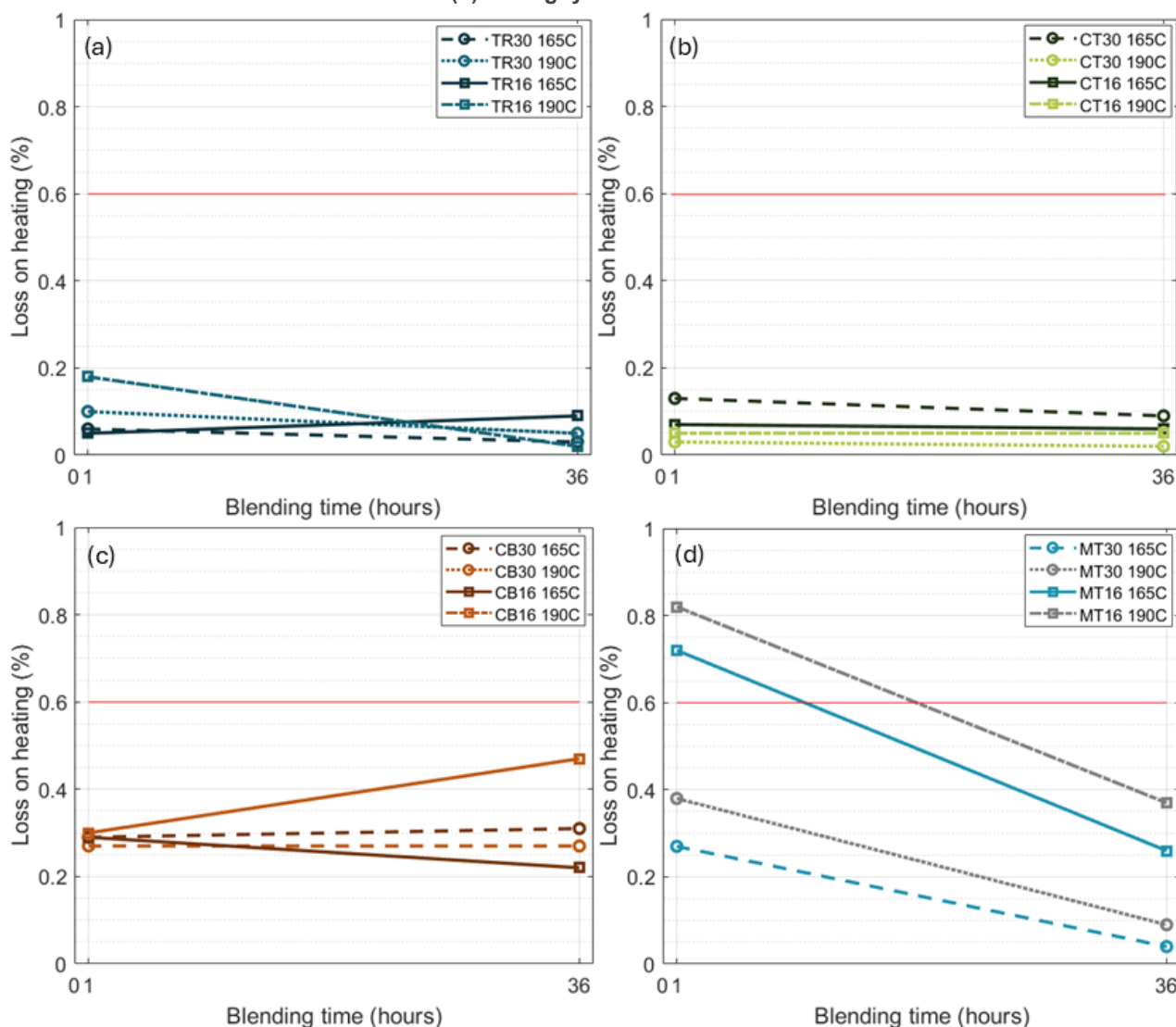
MRWA Specification 511:2025 has a requirement for a maximum loss on heating value of 0.6% for polymer modified binders (Table 511.5), but there are no limits explicitly specified for CRMBs in Table 511.6. Austroads ATS 3110:2023 specifies a maximum loss on heating value of 0.6%.

Table 4.17: Loss on heating results for all binders; loss on heating in %

Samples	Digestion time					
	1h	2h	4h	11h	24h	36h
TR30_165C_	0.06	–	–	–	–	0.03
TR30_190C_	0.10	–	–	–	–	0.05
TR16_165C_	0.05	–	–	–	–	0.09
TR16_190C_	0.18	–	–	–	–	0.02
CT30_165C_	0.13	–	–	–	–	0.09
CT30_190C_	0.03	–	–	–	–	0.02
CT16_165C_	0.07	–	–	–	–	0.06
CT16_190C_	0.05	–	–	–	–	0.05
CB30_165C_	0.29	–	–	–	–	0.31
CB30_190C_	0.27	–	–	–	–	0.27
CB16_165C_	0.29	–	–	–	–	0.22
CB16_190C_	0.30	–	–	–	–	0.47
MT30_165C_	0.27	–	–	–	–	0.04
MT30_190C_	0.38	–	–	–	–	0.09
MT16_165C_	0.72	–	–	–	–	0.26
MT16_190C_	0.82	–	–	–	–	0.37

Figure 4.51 graphically presents the results of Table 4.17. 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 4.51: 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.

Table 4.18 presents the ANOVA for loss on heating, revealing that all factors had a statistically significant impact on the results except from temperature.

Table 4.18: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on loss on heating

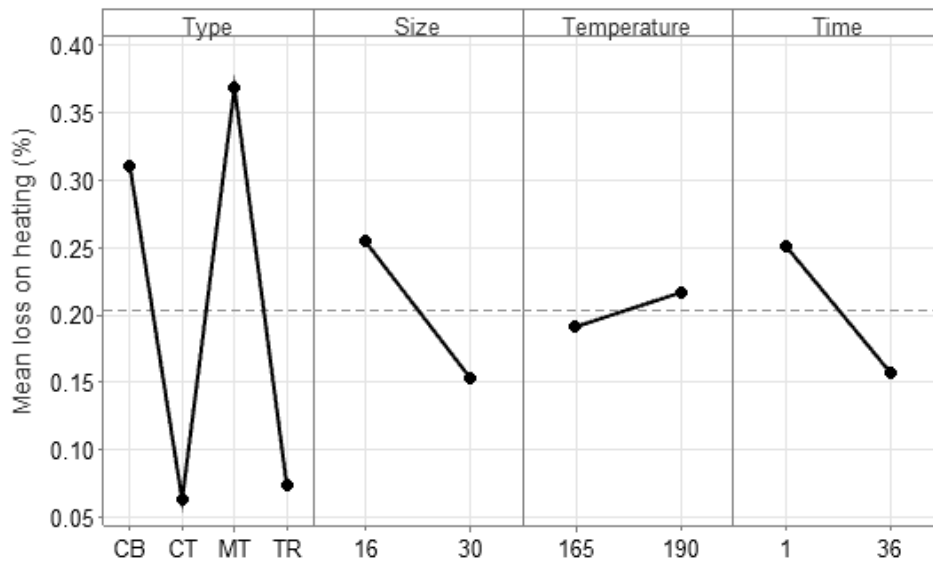
Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	1.21244	0.40415	18.22	0.000
Size	1	0.16423	0.16423	7.40	0.009
Temperature	1	0.01046	0.01046	0.47	0.495
Time	1	0.14213	0.14213	6.41	0.014
Error	57	1.26418	0.02218		
Lack of fit	25	0.86084	0.03443	2.73	0.004
Pure error	32	0.40334	0.01260		
Total	63	2.79344			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

The main effects plots of Figure 4.52 reveal that binders produced with CB- and MT-derived crumb rubber resulted in comparatively greater loss on heating values when compared to TR- and CT-derived CRMBs. In

addition, the loss on heating of CRMBs generally decreased for S30 crumb rubber particles and when the blending temperature was increased from 1 to 36 hours.

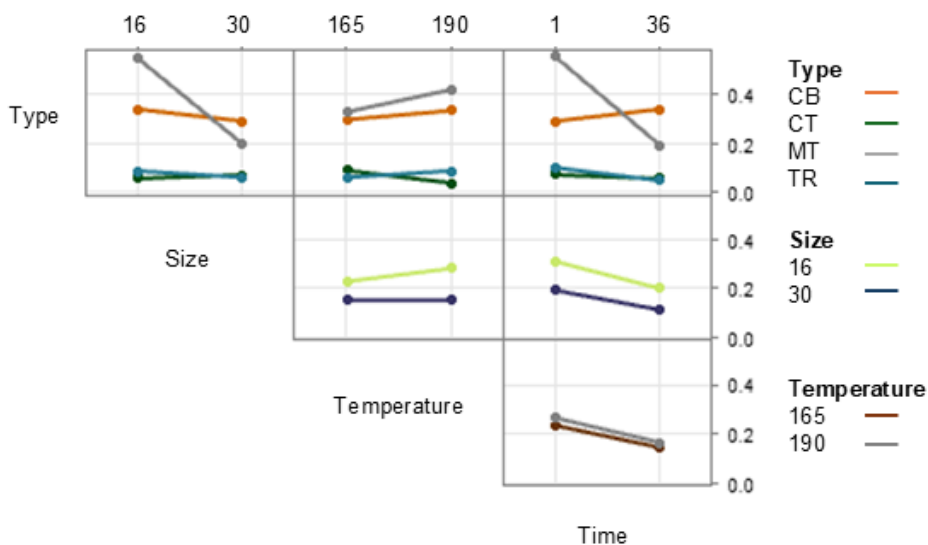
Figure 4.52: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on loss on heating



The interaction among the parameters investigated are graphically presented in Figure 4.53. Crumb rubber type was found to interact with all other parameters. Considering the crumb rubber type and size, loss on heating decreased when S30 particles were used instead of S16, except for CT-derived CRMBs. An increase in blending temperature from 165 to 190 °C was also found to result in an increase in loss on heating for all CRMBs except for those produced using CT-derived crumb rubber. Increasing the blending temperature from 1 to 36 hours was found to result in a decreased loss on heating for all CRMBs except for those produced with CB-derived crumb rubber.

No interactions were identified between crumb rubber size and blending temperature and crumb rubber size and blending time or between blending temperature and time.

Figure 4.53: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for loss on heating



These findings demonstrate that each of the investigated parameters impacted the loss on heating results for each crumb rubber type differently. TR- and MT-derived crumb rubber binders were similarly impacted by the changes in blending time, blending temperature and particle size gradation. However, CT-derived CRMBs

reacted differently to changes in crumb rubber size gradation and blending temperature, and CB-derived CRMBs behaved differently under prolonged blending time from 1 to 36 hours.

For S16 MT-derived CRMBs, extending the blending time from 1 to 36 hours meant that the binders could meet the Austroads ATS 3110:2023 upper limit requirements. A similarly impactful change could also be achieved by changing the crumb rubber size gradation from S16 to S30. For the rest of the crumb rubber types, the effects of crumb rubber size gradation and blending parameters were not found to notably increase the potential exposure of workers to volatiles during asphalt manufacture and construction.

4.4.3 Summary of Findings and Discussion

The viscosity results presented in Section 4.4.1 reflect the flowability of the produced CRMBs. This property is an indication of sufficiently low viscosity to facilitate asphalt mixing activities. All CRMBs were found to meet the upper limit requirements set by Austroads ATS 3110:2023 for viscosity at 165 °C, except for MT-derived CRMBs. Discussions in Section 4.4.1 and Appendix C.5 explain the dependency of these results on the speed of the spindle when the viscosity at 165 and 175 °C is recorded. The spindle speeds for MT-derived CRMBs were comparatively lower than those of TR-, CT- and CB-derived CRMBs according to Table C.1, Table C.2, Table C.3 and Table C.4. Therefore, even though the results of Section 4.4.1 suggest that the viscosity of MT-derived CRMBs would be too high for successful asphalt manufacture activities, Table C.7 suggests that measurements may have been comparable to those of TR-, CT- and CB-derived CRMBs had they been obtained at the same spindle speeds. It is, however, recognised that more work is required to confirm this.

Loss on heating results relate to worker safety during asphalt mixing and compaction as they are a measure of volatile loss at elevated temperatures. Volatile loss was found to be below that permitted by Austroads ATS 3110:2023 limits, except from that of MT16_165C_1h and MT16_190C_1h.

These findings overall suggest that the handling of MT-derived CRMBs may be expected to be different when compared to the CRMBs produced with different rubber types. Handling of some MT-derived CRMBs in the laboratory was indeed different, affected by a composite morphology with a more prominent solid phase when compared to the other CRMBs of this research. Images of the blended samples in Appendix B may provide a visual indication.

The loss on heating results showed that TR-, CT- and CB-derived CRMBs were within the requirements of MRWA Specification 511:2025. MT-derived CRMBs showed inconsistent results that were not in all cases within the MRWA Specification 511:2025 limits. CB-derived CRMBs consistently showed greater loss on heating when compared to TR- and CT-derived CRMBs but remained within the specified limits.

4.5 Elasticity

The elasticity of binders has been thought to impact the fatigue life (Dantas-Neto et al. 2006a) and overall durability of the resultant asphalt by improving resistance to reflective and fatigue cracking (Denneman et al. 2015). In Australia, the torsional recovery and resilience at 25 °C tests are used to measure the elasticity of modified binders. The torsional recovery at 25 °C and resilience at 25 °C results for all CRMBs investigated in this research are presented in Sections 4.5.1 and 4.5.2, respectively.

4.5.1 Torsional Recovery at 25 °C

The torsional recovery at 25 °C was measured in accordance with ATM 122:2022 and is typically used to measure the elasticity of modified binders whereby the greater the recovery the more elastic the binder.

Table 4.19 presents the average results for all CRMBs examined in this research. The greatest torsional recovery at 25 °C was measured for MT30_165C_11h at 59% and the lowest at 28% for both CB16_165C_1h and MT16_165C_24h.

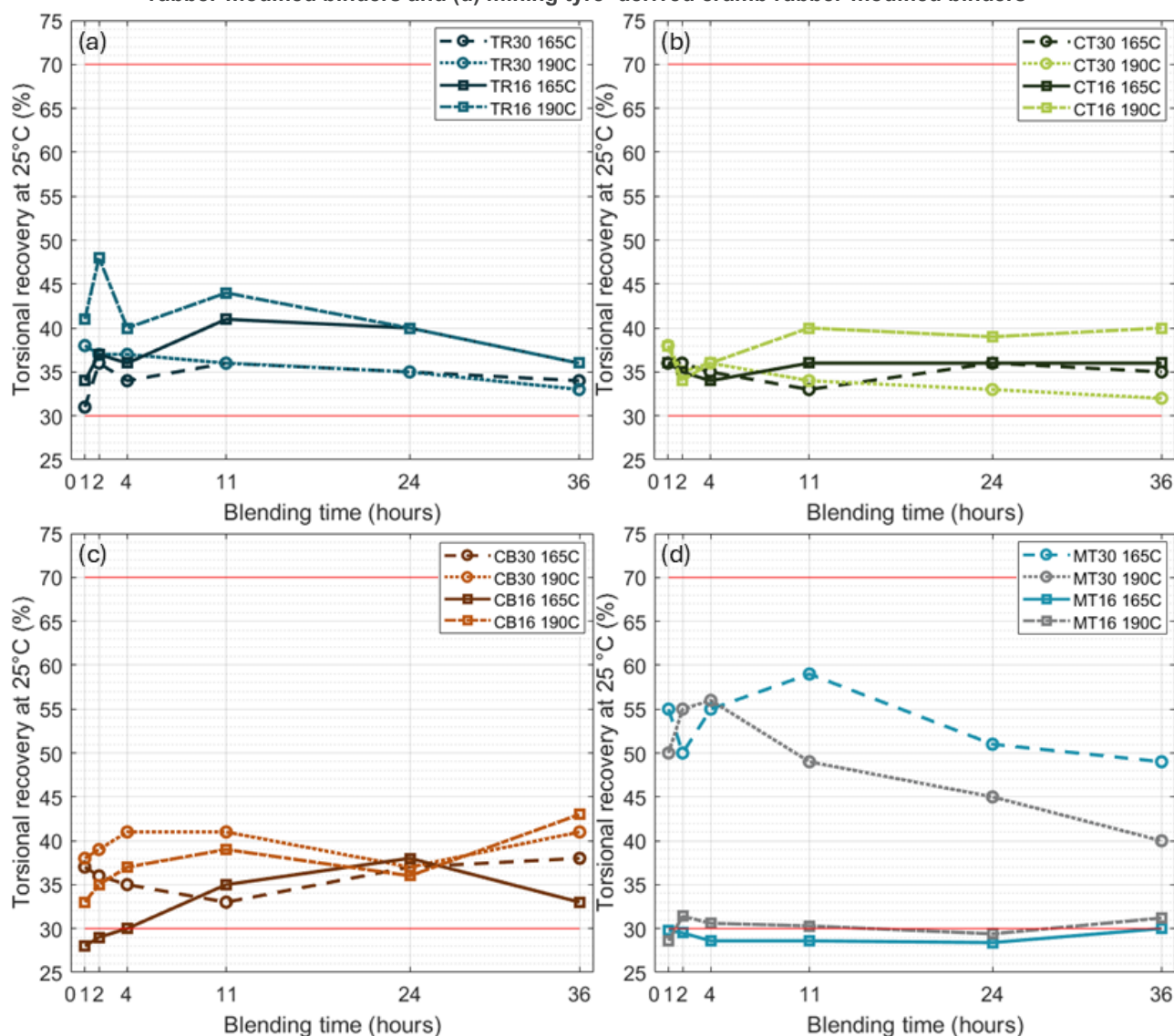
Table 4.19: Torsional recovery at 25 °C results for all binders. Results in %

Samples	Digestion time					
	1h	2h	4h	11h	24h	36h
TR30_165C_	31	36	34	36	35	34
TR30_190C_	38	37	37	36	35	33
TR16_165C_	34	37	36	41	40	36
TR16_190C_	41	38	40	44	40	36
CT30_165C_	36	36	35	33	36	35
CT30_190C_	38	35	36	34	33	32
CT16_165C_	36	35	34	36	36	36
CT16_190C_	38	34	36	40	39	40
CB30_165C_	37	36	35	33	37	38
CB30_190C_	38	39	41	41	37	41
CB16_165C_	28	29	30	35	38	33
CB16_190C_	33	35	37	39	36	43
MT30_165C_	55	50	55	59	51	49
MT30_190C_	50	55	56	49	45	40
MT16_165C_	30	30	29	29	28	30
MT16_190C_	29	31	31	30	29	31

Austroads ATS 3110:2023 provides an acceptable range for the torsional recovery at 25 °C of CRMBs for asphalt applications between 30 and 70%. Figure 4.54 graphically presents the torsional recovery at 25 °C results for all examined binders against the ATS 3110:2023-specified limits.

CRMBs produced with TR-derived crumb rubber binders were found to be within the limits specified by ATS 3110:2023. The results were found to cover 32.5% of the allowable range. Similarly, CRMBs produced with CT-derived crumb rubber were found to be within those specified limits with their results covering only 20% of this allowable range. From Figure 4.54 (c), the torsional recovery at 25 °C of CB16_165C_1h and CB16_165C_2h was below that specified limit with the rest of the CB-derived CRMBs meeting the requirements of ATS 3110:2023. The range in the results, however, is still only 37.5% of the allowable range. Lastly, Figure 4.54 (d) reveals that the torsional recovery at 25 °C of MT16_165C_4h, MT16_165C_11h, MT16_165C_24h, MT16_190C_1h and MT16_190C_24h was below 30%. Notably, the torsional recovery at 25 °C results were found to vary depending on the selected parameters more distinctly between CRMBs produced with S30 and S16 crumb rubber particles. That variation was found to expand to 77.5% of the allowable range.

Figure 4.54: Blending time versus torsional recovery 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: Red lines denote Austroads ATS 3110:2023 limits.

From the ANOVA results of Table 4.20 it is evident that only the crumb rubber type and size were found to have a statistically significant effect on the results of the torsional recovery at 25 °C test.

Table 4.20: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on torsional recovery at 25 °C

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	650.27	216.76	6.35	0.000
Size	1	1,087.28	1,087.28	31.85	0.000
Temperature	1	93.10	93.10	2.73	0.100
Time	5	52.55	10.51	0.31	0.908
Error	181	6,178.19	34.13		
Lack of fit	85	6,112.20	71.91	104.60	0.000
Pure error	96	66.00	0.69		
Total	191	8,061.39			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

The main effect plots of Figure 4.55 show that CRMBs produced with MT-derived crumb rubber had a comparatively greater mean torsional recovery at 25 °C, as were those produced with S30 crumb rubber particles. Figure 4.55 also shows that increasing the blending temperature from 165 to 190 °C resulted in CRMBs with greater torsional recovery at 25 °C, and the torsional recovery at 25 °C for binders blended for 11 hours was also greater; however, these effects were not found to be statistically significant.

Figure 4.55: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on torsional recovery at 25 °C

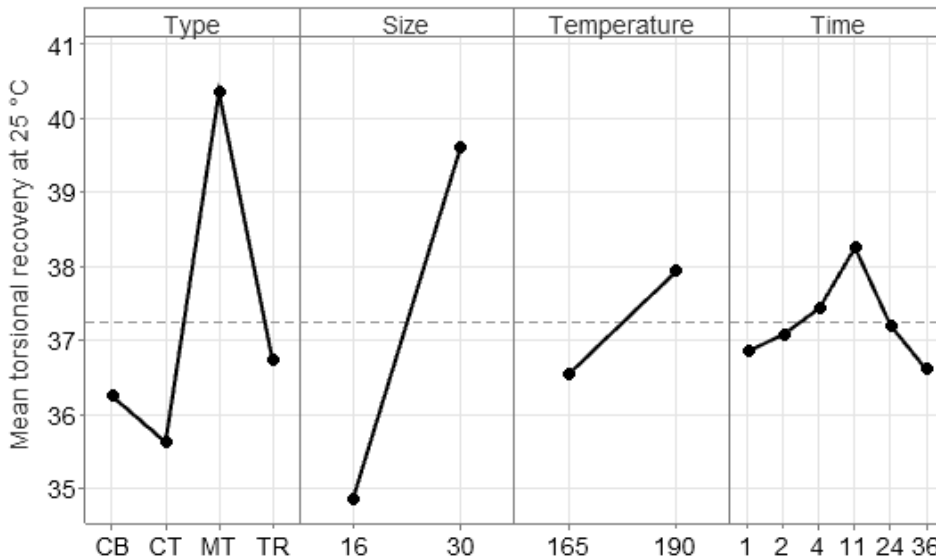
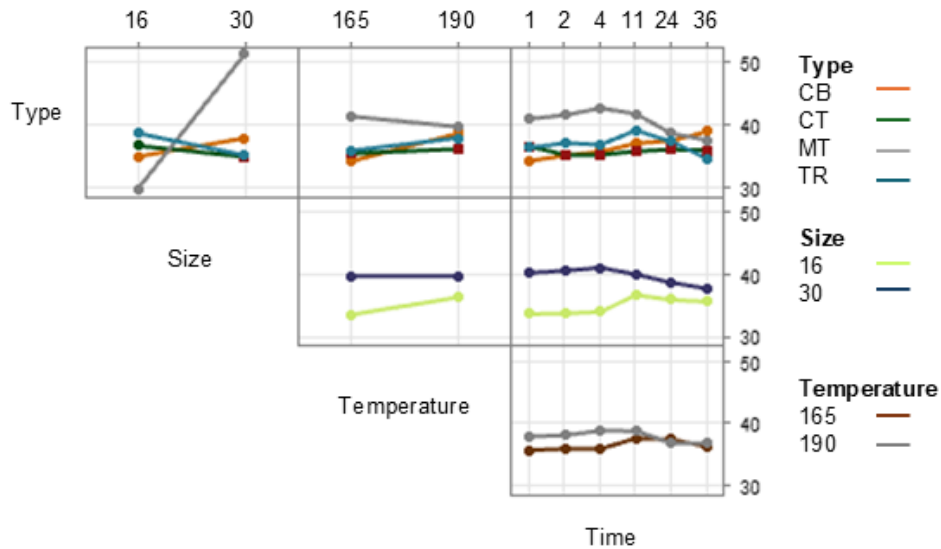


Figure 4.56 presents the interaction plots for the torsional recovery at 25 °C results. Crumb rubber type was found to interact with all other parameters investigated. Changing the crumb rubber particle gradation from S16 to S30 was found to result in an increase in torsional recovery at 25 °C for MT- and CB-derived CRMBs but a decrease for TR- and CT-derived CRMBs. Increasing the blending temperature from 165 to 190 °C was found to result in an increase in torsional recovery at 25 °C for all binders but those produced with MT-derived CRMBs, for which a decrease in torsional recovery at 25 °C was observed. Crumb rubber type was also found to have various interactions with blending time, with no 2 of the CRMBs found to follow the same trends as blending time increased.

Crumb rubber size was also found to interact with blending temperature, where the torsional viscosity at 25 °C of CRMBs produced with S30 crumb rubber particles remained constant, whereas that of CRMBs produced with S16 crumb rubber particles was found to increase with an increase in blending temperature from 165 to 190 °C. Crumb rubber size was found to interact with blending time between 4 and 11 hours, where the torsional recovery at 25 °C for CRMBs produced with S30 crumb rubber particles was found to slightly decrease, whereas that of CRMBs produced with S16 crumb rubber particles was found to increase.

The torsional recovery at 25 °C results were also found to have interactions between blending temperature and blending time between 4 and 11 hours. The torsional recovery at 25 °C of CRMBs produced at 165 °C was found to increase with that increase in blending duration, whereas that of binders produced at 190 °C remained largely unchanged.

Figure 4.56: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for torsional recovery at 25 °C



Nevertheless, MT-derived CRMBs whose torsional recovery at 25 °C was not within the Austroads ATS 3110:2023-specified limits were limited to results slightly below the lower specification limit, noting that these results did not increase with blending time. All TR-, CT- and CB-derived CRMBs, except CB16_165C_1h and CB16_165C_2h, were within the Austroads ATS 3110:2023 specified limits.

4.5.2 Resilience at 25 °C

Resilience, as measured following ASTM D5329-20, quantifies a binder's ability to recover after the short application of a compressive load (Yao et al. 2022). It is, therefore, an indication of the binder's elastic properties in a similar way to torsional recovery at 25 °C test.

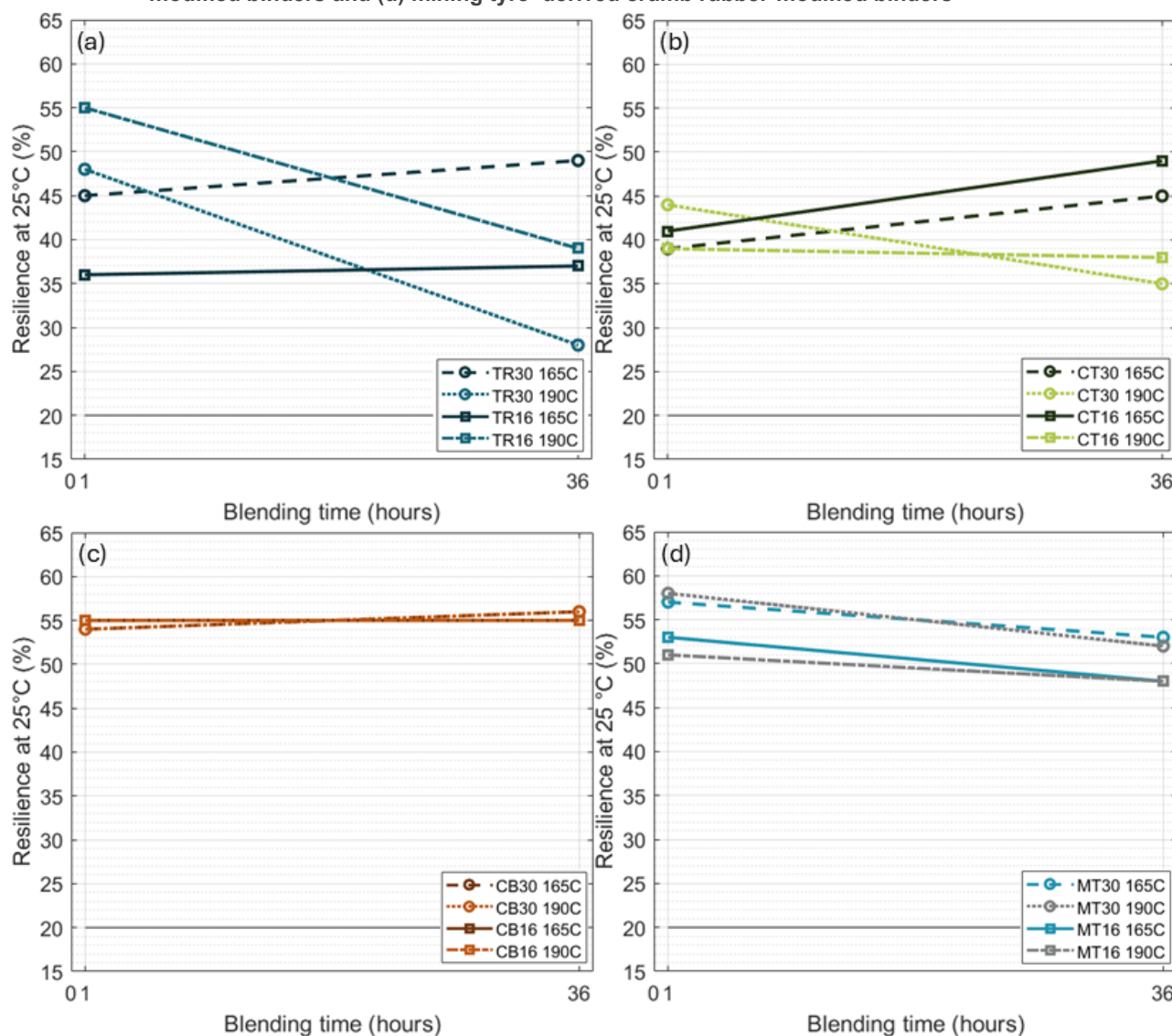
Table 4.21 presents the results for all CRMBs investigated. The lowest resilience at 25 °C was found for TR30_190C_36h and the greatest for MT30_190C_1h at 28 and 58%, respectively.

Table 4.21: Resilience at 25 °C results following 1 and 36 hours of blending for the C170 and all binders; MRWA Specification 511:2025 (Table 511.6) specifies a minimum requirement of 20%; results in %

Samples	Digestion time					
	1h	2h	4h	11h	24h	36h
TR30_165C_	45	–	–	–	–	39
TR30_190C_	48	–	–	–	–	28
TR16_165C_	36	–	–	–	–	37
TR16_190C_	55	–	–	–	–	39
CT30_165C_	39	–	–	–	–	45
CT30_190C_	44	–	–	–	–	35
CT16_165C_	41	–	–	–	–	49
CT16_190C_	39	–	–	–	–	38
CB30_165C_	54	–	–	–	–	56
CB30_190C_	54	–	–	–	–	56
CB16_165C_	55	–	–	–	–	55
CB16_190C_	55	–	–	–	–	55
MT30_165C_	57	–	–	–	–	53
MT30_190C_	58	–	–	–	–	52
MT16_165C_	53	–	–	–	–	48
MT16_190C_	51	–	–	–	–	48

Figure 4.57 graphically presents the resilience at 25 °C results of Table 4.21. All binders were found to meet the minimum requirement of 20% as per MRWA Specification 511:2025.

Figure 4.57: 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.

The ANOVA results of Table 4.22 show that crumb rubber type and blending time had a statistically significant impact on the resilience at 25 °C results.

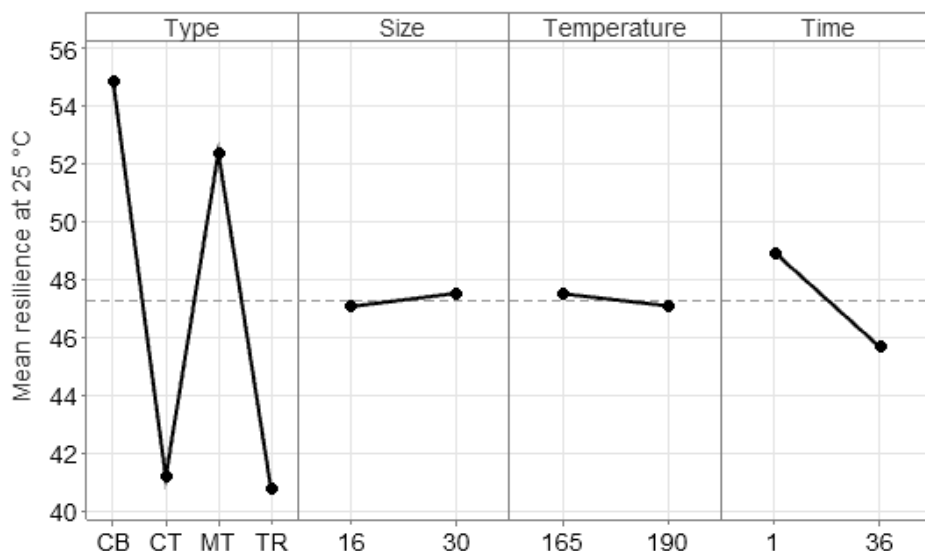
Table 4.22: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on resilience at 25 °C

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	2,609.71	869.903	39.31	0.000
Size	1	3.38	3.376	0.15	0.698
Temperature	1	2.85	2.848	0.13	0.721
Time	1	170.63	170.629	7.71	0.007
Error	57	1,261.52	22.132		
Lack of fit	25	1,256.81	50.272	341.19	0.000
Pure error	32	4.71	0.147		
Total	63	4,048.09			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

The main effects plots are presented in Figure 4.58. They show that binders produced with CB- and MT-derived crumb rubber exhibited greater resilience when compared to binders produced using TR- and CT-derived crumb rubber. Further, it is shown that resilience at 25 °C typically decreased with an increase in blending time from 1 to 36 hours.

Figure 4.58: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on resilience at 25 °C

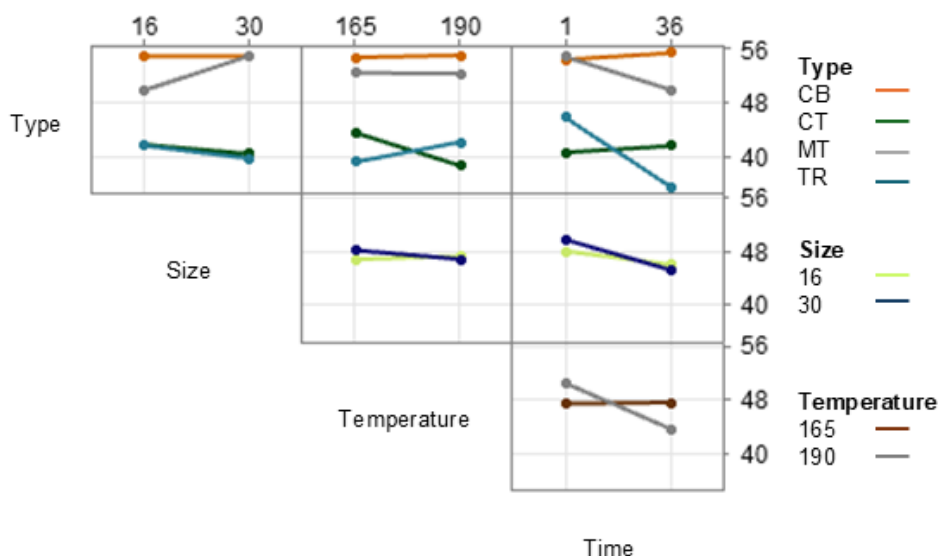


The interaction plots among the parameters investigated are shown in Figure 4.59. Considering the interactions between crumb rubber type and size, the resilience at 25 °C for TR- and CT-derived CRMBs was found to decrease with a change in crumb rubber size from S16 to S30, whereas the opposite was true for MT-derived CRMBs. CB-derived CRMBs were found to be relatively unaffected by the change in crumb rubber size gradation. The resilience at 25 °C of CB- and MT-derived CRMBs was found to be relatively unaffected by a change in blending temperature as well. However, an interaction between the resilience at 25 °C of TR- and CT-derived CRMBs and blending temperature can be seen, where that of TR-derived CRMBs was found to increase with an increase in blending temperature from 165 to 190 °C, whereas that of CT-derived CRMBs decreased. Interactions between crumb rubber type and blending time were also identified. With an increase in blending time from 1 to 36 hours, the resilience at 25 °C of CT- and CB-derived CRMBs was found to increase, whereas that of TR- and MT-derived CRMBs was found to decrease.

Crumb rubber size gradation and blending temperature were also found to marginally interact. The resilience at 25 °C of binders manufactured with S16 crumb rubber particles was found to slightly increase, whereas that of binders manufactured with S30 crumb rubber particles was found to slightly decrease with an increase in temperature from 165 to 190 °C. Crumb rubber size gradation and blending time were not found to interact. Blending time was, however, found to interact with blending temperature, whereby an increase in blending time from 1 to 36 hours resulted in a decrease in the resilience at 25 °C for binders blended at 190 °C, whereas a marginal increase in resilience at 25 °C was observed for binders blended at 165 °C.

These findings demonstrate that, when all else is kept equal, CB- and MT-derived binders may be expected to result in comparatively greater resilience at 25 °C. The resilience at 25 °C of MT-derived CRMBs, however, was found to be more susceptible to changes in crumb rubber particle gradation and blending time than that of CB-derived CRMBs.

Figure 4.59: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for resilience at 25 °C



4.5.3 Summary of Findings and Discussion

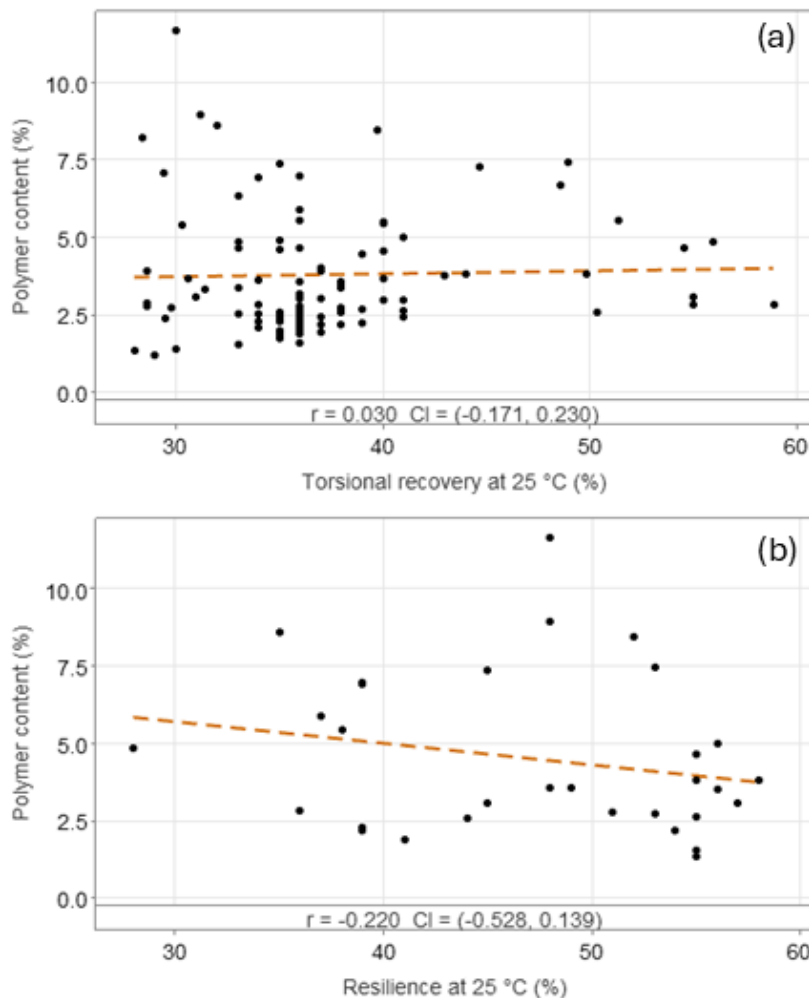
According to the ANOVA results of Table 4.20 and Table 4.22, crumb rubber type was found to affect the torsional recovery at 25 °C and resilience at 25 °C results, respectively. Even though both torsional recovery at 25 °C and resilience at 25 °C are a measure of the elastic properties of the binders, the results of Sections 4.5.1 and 4.5.2 convey a different story regarding the elasticity of the examined binders in this research. The torsional recovery at 25 °C results in Section 4.5.1 suggest that sample MT30_190C_11h is the most elastic, whereas the resilience at 25 °C test results of Section 4.5.2 suggest that this is the case for CB30_165C_1h binders. However, it is worth noting that the input parameters in the ANOVA were different between the 2 methods, as all binders were tested following the torsional recovery at 25 °C, whereas only those blended for 1 and 36 hours were tested for resilience at 25 °C, which would have affected these findings to some degree. Considering the as-received test results, though, this observation remains. For example, some notable differences can be observed for sample CB16_165C_1h, which according to the torsional recovery at 25 °C test, exhibited the lowest elasticity with a result of 28%, but its elasticity according to the resilience at 25 °C test was among the greatest at 55% (resilience at 25 °C results ranged between 28 and 58%). Similarly, sample TR30_190C_36h exhibited the lowest elasticity according to the resilience at 25 °C result at 28%, whereas it was approximately in the middle of the range according to the torsional recovery at 25 °C result at 36% (torsional recovery at 25 °C results ranged between 28 and 59%).

These discrepancies could be attributed to the different working mechanisms of the 2 tests. It is suspected that torsional recovery at 25 °C results predominantly reflect the elasticity of the liquid phase, whereas the resilience at 25 °C is more representative of the behaviour of the composite (undigested rubber particles and binder liquid phase). Austroads (2021b) also noted that CRMBs whose resilience at 25 °C is measured to be above 50% cannot effectively be characterised using the torsional recovery at 25 °C test. This was the case for all CB- and all MT-derived CRMBs except from MT16_165C_36h and MT16_190C_36h.

Oliver (1981) explained that the elastic properties of CRMBs are a result of the digestion process. Elasticity is a result of crumb rubber dissolution (Billiter et al. 1997; Ghavibazoo & Abdelrahman 2013; Ghavibazoo et al. 2013; Lo Presti 2013); however, the dissolved rubber only positively contributes to the elasticity of the binder before it starts to notably degrade (Huang et al. 2017). This is because at that stage, the molecular weight of the dissolved rubber is decreased (Daly et al. 2019). Figure 4.60 presents the correlation between the elasticity results of Section 4.5 and the dissolved polymer content as measured by HPLC-GPC. As per Sections 3.3.5 and 4.2.3, only samples blended for 1, 11 and 36 hours were analysed through HPLC-GPC. For the correlation of Figure 4.60 (a), the data points for samples blended for 2, 4 and 24 hours were calculated using the equations of Figure 4.14. The torsional recovery at 25 °C results show a negligible correlation to the polymer content in the liquid phase of the binder, and the results of resilience at 25 °C

show a weak correlation. This either suggests that, contrary to literature findings, the increase in polymer content in the binder's liquid phase cannot be correlated to the elasticity of the binder or that the tests used to measure binder elasticity in this research cannot effectively reflect the properties of the produced CRMBs.

Figure 4.60: Correlation between (a) torsional recovery at 25 °C and (b) resilience at 25 °C with polymer content as measured by HPLC-GPC



Overall, an increased elasticity of the binder is a desired property potentially impacting fatigue life. The torsional recovery at 25 °C results of this research suggest that the resultant asphalt mix would have a comparable performance irrespective of the crumb rubber type used, except in the case of MT-derived CRMBs, which not only presented greater results when compared to the rest of the CRMBs but also the greatest dependence on crumb rubber size. Importantly, all CRMBs assessed were within the limits set in MRWA Specification 511:2025.

4.6 Rutting Resistance

Rutting in asphalt is the result of repeated loading from traffic, especially at elevated pavement temperatures, causing the accumulation of viscous and plastic deformation. The rutting performance is influenced by a variety of factors including aggregate characteristics, mix design as well as the properties and content of the selected binder (Pan et al. 2023). The softening point (Lo Presti 2013) and consistency 6% at 60 °C (Austroads 2019) results for binders have been thought to impact the rutting performance of asphalt. These are presented in Sections 4.6.1 and 4.6.2, respectively.

4.6.1 Softening Point

Table 4.23 shows the softening point results of all binders assessed, including the blended unmodified bitumen. Softening point is an empirical method used to assess the flowability of modified binders at elevated temperatures (Grobler 2020). For the unmodified bitumen, any increase in softening point is likely the result in oxidation, whereas for CRMBs it is a result of both potential oxidation and the digestion process, whereby the absorption of the bitumen light components increases the stiffness of the liquid phase.

From Table 4.23, the greatest softening point was measured for sample MT30_165C_4h and the lowest for sample CT16_190C_24h at 76.2 and 60.0 °C, respectively. The softening point of the unmodified C170 was measured at 49.0 °C, which means that the incorporation of crumb rubber, irrespective of type, size or blending parameters, is expected to contribute to a decrease in binder flowability.

The results of the blended unmodified C170 suggest that some bitumen oxidation is likely during the blending process described in Section 3.2.1. It is, however, not possible to distinguish between the contribution of each of these mechanisms to the softening point increase in the CRMBs, especially as CRMBs are affected differently by thermo-oxidative phenomena when compared to unmodified bitumen (Borinelli et al. 2024). It is, therefore, noted that the results presented in this section reflect both mechanisms.

Table 4.23: Softening point results for all samples assessed; results in °C

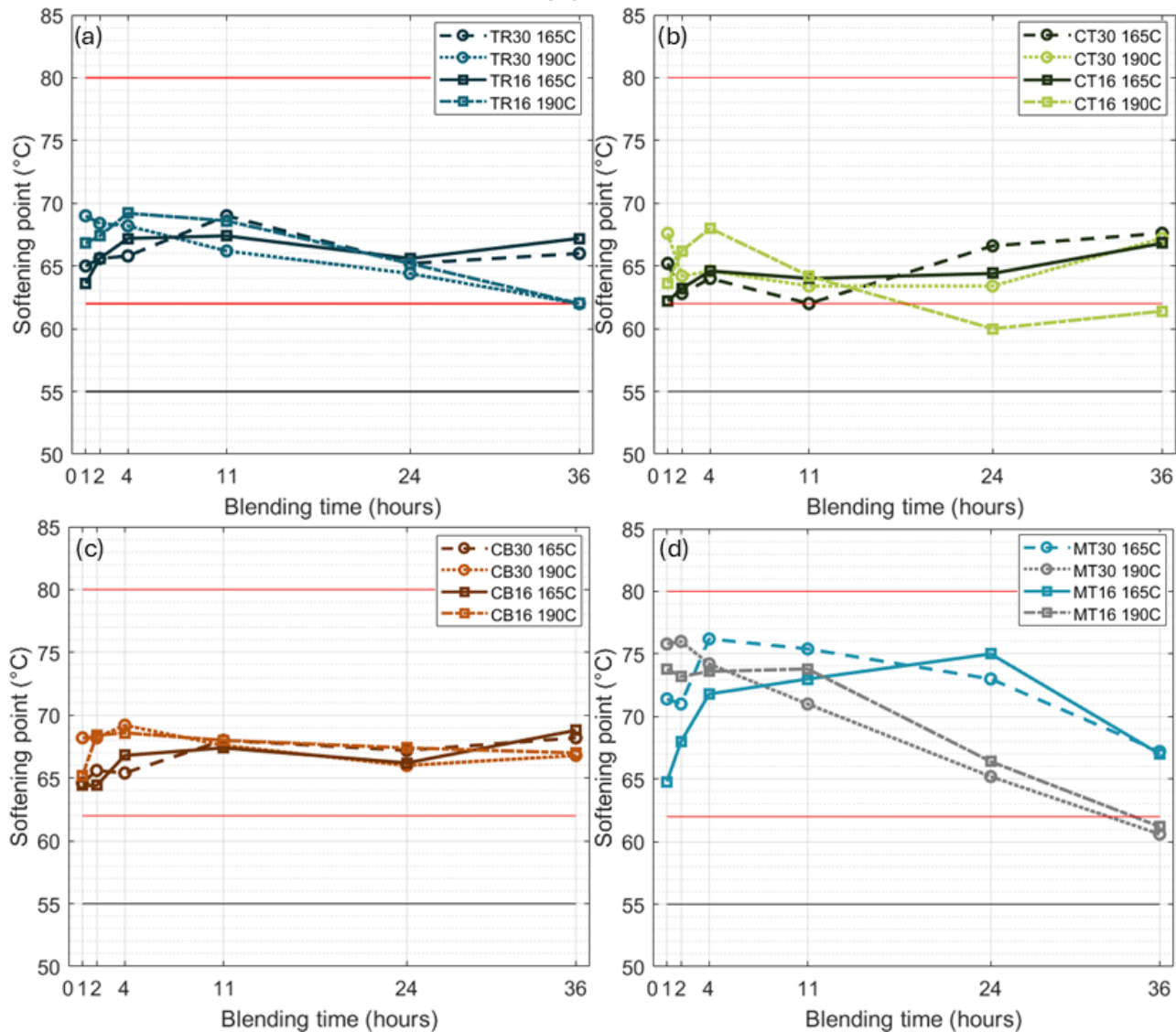
Samples	Digestion time						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_	–	65.0	65.6	65.8	69.0	65.2	66.0
TR30_190C_	–	69.0	68.4	68.2	66.2	64.4	62.0
TR16_165C_	–	63.6	65.6	67.2	67.4	65.6	67.2
TR16_190C_	–	66.8	67.4	69.2	68.6	65.2	62.0
CT30_165C_	–	65.2	62.8	64.0	62.0	66.6	67.6
CT30_190C_	–	67.6	64.2	64.6	63.4	63.4	67.2
CT16_165C_	–	62.2	63.2	64.6	64.0	64.4	66.8
CT16_190C_	–	63.6	66.2	68.0	64.2	60.0	61.4
CB30_165C_	–	64.6	65.6	65.4	68.0	67.2	68.2
CB30_190C_	–	68.2	68.2	69.2	67.6	66.0	66.8
CB16_165C_	–	64.4	64.4	66.8	67.4	66.2	68.8
CB16_190C_	–	65.2	68.4	68.6	68.0	67.4	67.0
MT30_165C_	–	71.4	71.0	76.2	75.4	73.0	67.2
MT30_190C_	–	75.8	76.0	74.2	71.0	65.2	60.6
MT16_165C_	–	64.8	68.0	71.8	73.0	75.0	67.0
MT16_190C_	–	73.8	73.2	73.6	73.8	66.4	61.2
C170	49.0	–	–	–	–	–	–
C170_165C_	–	48.2	–	–	–	–	58.6
C170_190C_	–	49.0	49.8	51.6	49.2	55.6	74.2

Figure 4.61 graphically presents the results of Table 4.23 against the limits specified by MRWA Specification 511:2025 and Austroads ATS 3110:2023.

The TR-derived CRMBs were found to meet both specification requirements. In fact, the variability of the TR-derived CRMB binder performance was approximately 39% of the Austroads ATS 3110:2023 range irrespective of crumb rubber gradation and processing parameters. All CT-derived CRMBs were found to have a softening point above the minimum requirement of MRWA Specification 511:2025; however, CT16_190C_24h and CT16_190C_36h were below the required minimum limit of Austroads ATS 3110:2023. The variability of the results of all CT-derived CRMBs were 44% of the Austroads ATS 3110:2023 specified range. The CB-derived CRMBs were found to meet both MRWA

Specification 511:2025 and Austroads ATS 3110:2023 requirements. The variability in the softening point results of CB-derived CRMBs was approximately 27% of the Austroads ATS 3110:2023 specified range. All MT-derived CRMBs were found to meet the requirements of MRWA Specification 511:2025; however, MT-derived CRMBs blended at 190 °C for 36 hours, irrespective of crumb rubber size gradation, were found to have a softening point below the minimum specified by Austroads ATS 3110:2023. The range of softening point results for MT-derived CRMBs was found to be 87% of the Austroads ATS 3110:2023 specified range.

Figure 4.61: 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 lines denote Austroads ATS 3110:2023 limits and black line denotes minimum requirement according to MRWA Specification 511:2025.

Table 4.24 presents the ANOVA results on all investigated parameters and all binders. From these results, it is evident that only the crumb rubber type and blending time have a statistically significant impact on the softening point results.

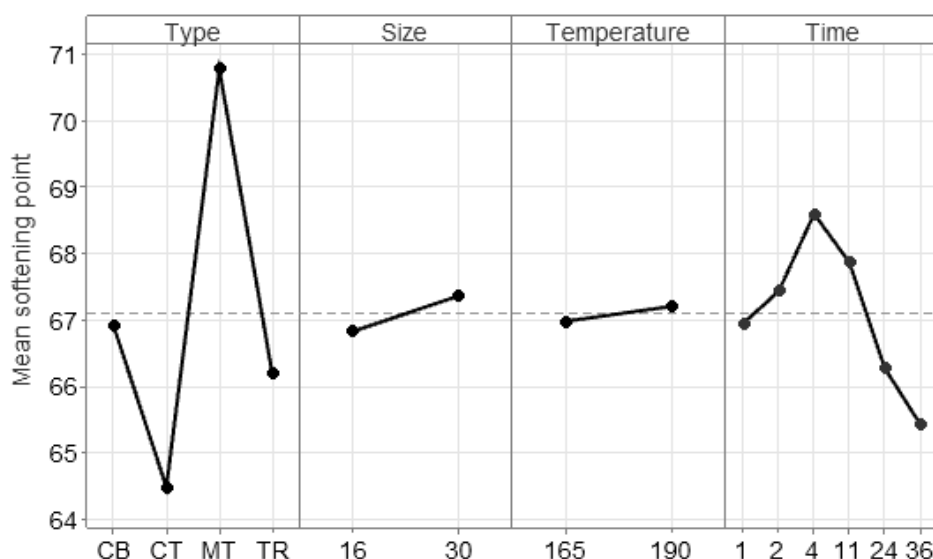
Table 4.24: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on softening point

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	1,022.41	340.804	49.85	0.000
Size	1	13.95	13.954	2.04	0.155
Temperature	1	2.44	2.444	0.36	0.551
Time	5	205.78	41.155	6.02	0.000
Error	181	1,237.49	6.837		
Lack of fit	85	1,234.43	14.523	456.68	0.000
Pure error	96	3.05	0.032		
Total	191	2,482.07			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

The main effect plots of Figure 4.62 reveal that CRMBs produced with MT-derived crumb rubber will have greater softening points on average. It is also shown that softening point typically would increase with blending time up to 4 hours and start decreasing beyond that.

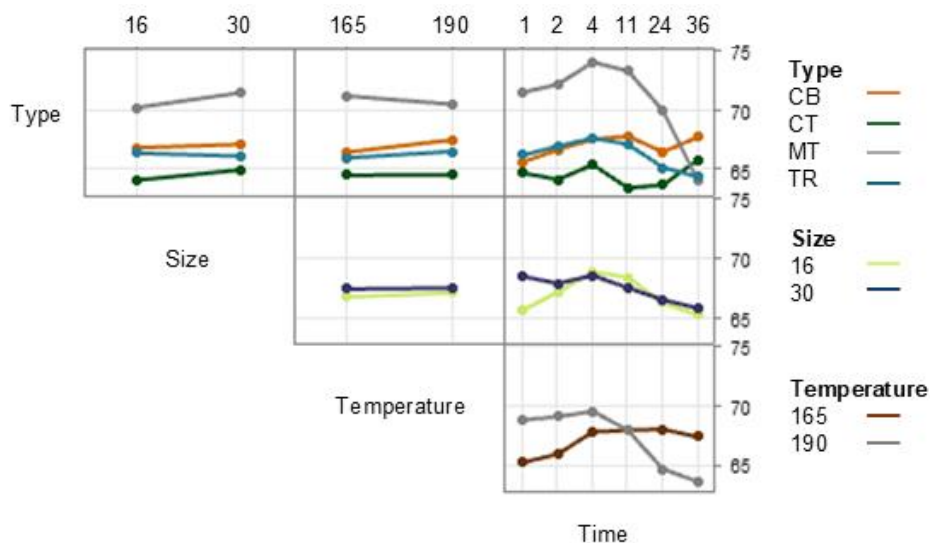
Figure 4.62: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on softening point



Some interactions between the various parameters investigated can be observed in Figure 4.63. The softening point of all crumb rubber types was found to slightly decrease when S16 particles were used, except for TR-derived CRMBs for which the opposite was true. The impact of blending temperature can also be seen to vary depending on the type of crumb rubber, whereby the softening point of CB- and TR-derived CRMBs increased with an increase in blending temperature, that of CT-derived CRMBs remained largely unaffected by the blending temperature and that of MT-derived CRMBs was found to decrease with an increase in blending temperature. The softening point of CB- and CT-derived CRMBs was found to slightly fluctuate with blending time, where in both cases the greatest mean softening point was found to be achieved following 36 hours of blending. On the other hand, the softening point of TR- and MT-derived CRMBs was found to increase with up to 4 hours of blending and decrease beyond that.

Interactions can also be observed between blending temperature and blending time. The softening point of CRMBs was found to increase with blending time up to 4 hours irrespective of blending temperature. However, beyond that, the softening point of CRMBs blended at 165 °C was found to remain relatively constant, whereas it notably decreased for binders blended at 190 °C.

Figure 4.63: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for softening point



Overall, MT-derived CRMBs were found to exhibit greater softening points when compared to the other crumb rubber types. However, the softening point of MT-derived CRMBs was also found to be more susceptible to blending time, ultimately resulting in an approximately 10 °C variation between the greatest and lowest softening point results.

All CRMBs tested met the minimum softening point of MRWA Specification 511:2025 and, in general, exhibited softening point results within the Austroads ATS 3110:2023-specified range. This was except for 2 MT-derived and 1 CT-derived CRMBs, all blended at 190 °C for 24 hours. These CRMBs showed softening point results just below the lower limit of Austroads ATS 3110:2023.

4.6.2 Consistency 6% at 60 °C

The consistency 6% at 60 °C test was developed to measure the shear deformation of modified binders at elevated temperatures. A binder's resistance to shear deformation has been found to be a comparatively good ranking measure for the rutting resistance of binders, whereby a greater consistency 6% at 60 °C value indicates a greater resistance to rutting (Austroads 2021a).

Table 4.25 presents the results for consistency 6% at 60 °C for the CRMBs blended for 1, 2, 4, 11 and 36 hours. The greatest consistency 6% at 60 °C was measured for CB16_165C_36h at 5,944 Pa.s and the lowest was measured for MT30_190C_36h at 828 Pa.s.

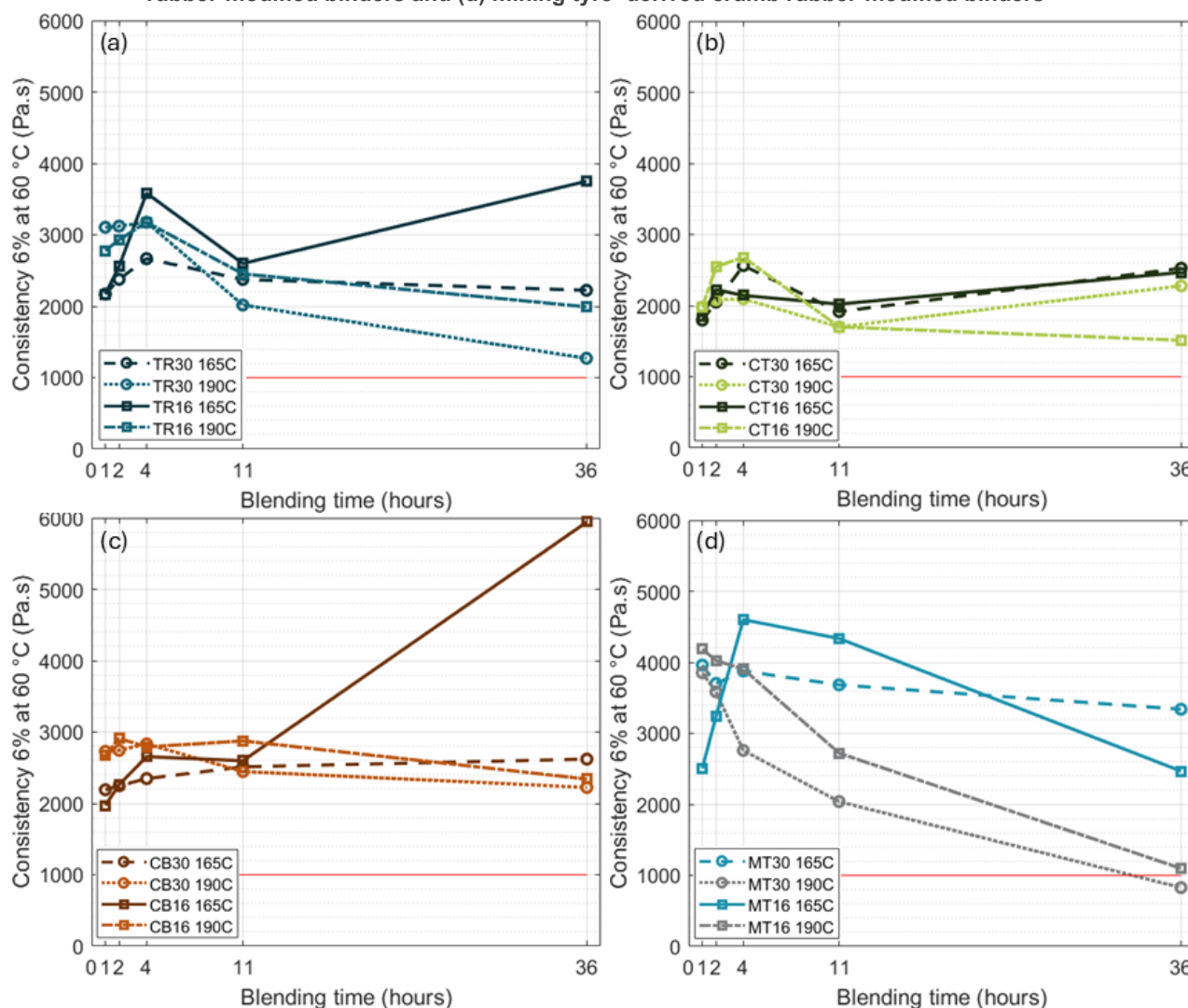
Table 4.25: Results for consistency 6% at 60 °C for all binders; results in Pa.s

Samples	Digestion time					
	1h	2h	4h	11h	24h	36h
TR30_165C_	2,170	2,376	2,666	2,375	–	2,223
TR30_190C_	3,105	3,121	3,176	2,017	–	1,271
TR16_165C_	2,170	2,556	3,583	2,598	–	3,750
TR16_190C_	2,779	2,929	3,176	2,453	–	1,991
CT30_165C_	1,795	2,049	2,561	1,915	–	2,526
CT30_190C_	1,977	2,081	2,095	1,700	–	2,278
CT16_165C_	1,850	2,225	2,142	2,021	–	2,467
CT16_190C_	1,977	2,546	2,680	1,700	–	1,512
CB30_165C_	2,191	2,264	2,347	2,512	–	2,620
CB30_190C_	2,729	2,743	2,833	2,446	–	2,223
CB16_165C_	1,971	2,262	2,655	2,597	–	5,944
CB16_190C_	2,670	2,910	2,791	2,875	–	2,341
MT30_165C_	3,961	3,702	3,878	3,682	–	3,339
MT30_190C_	3,853	3,584	2,759	2,039	–	828
MT16_165C_	2,505	3,238	4,601	4,335	–	2,468
MT16_190C_	4,185	4,025	3,905	2,721	–	1,098

Figure 4.64 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 TR-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. CT-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. CB-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. This result was recognised as an outlier, and the test was repeated with comparable findings. The details of this assessment are presented in Appendix C.3, concluding that the result was true to the behaviour of the binder. MT-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 MT-derived CRMBs was presented as a notable variation depending on the investigated parameters of 3,773 Pa.s.

Figure 4.64: 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.

The ANOVA results of Table 4.26 show that all investigated parameters except blending time significantly affected the consistency 6% at 60 °C results.

Table 4.26: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on consistency 6% at 60 °C

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	26,953,680	8,984,560	14.41	0.000
Size	1	3,872,725	3,872,725	6.21	0.014
Temperature	1	3,809,157	3,809,157	6.11	0.015
Time	4	4,557,391	1,139,348	1.83	0.126
Error	152	94,767,278	623,469		
Lack of fit	70	94,484,544	1,349,779	391.47	0.000
Pure error	82	282,735	3,448		
Total	161	134,109,251			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

Figure 4.65 presents the main effect plots for the consistency 6% at 60 °C results. It is shown that the greatest mean consistency 6% at 60 °C results were achieved by MT-derived CRMBs, followed by CB-derived CRMBs, TR-derived CRMBs and then CT-derived CRMBs. It is also shown that a change in the

crumb rubber gradation from S16 to S30 and blending temperature from 165 to 190 °C resulted in a decrease in the mean consistency 6% at 60 °C results. Lastly, even though the effects of blending time were not found to be significant, it is shown that blending for 4 hours generally resulted in the greatest and blending for 11 hours resulted in the lowest mean consistency 6% at 60 °C.

Figure 4.65: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on consistency 6% at 60 °C

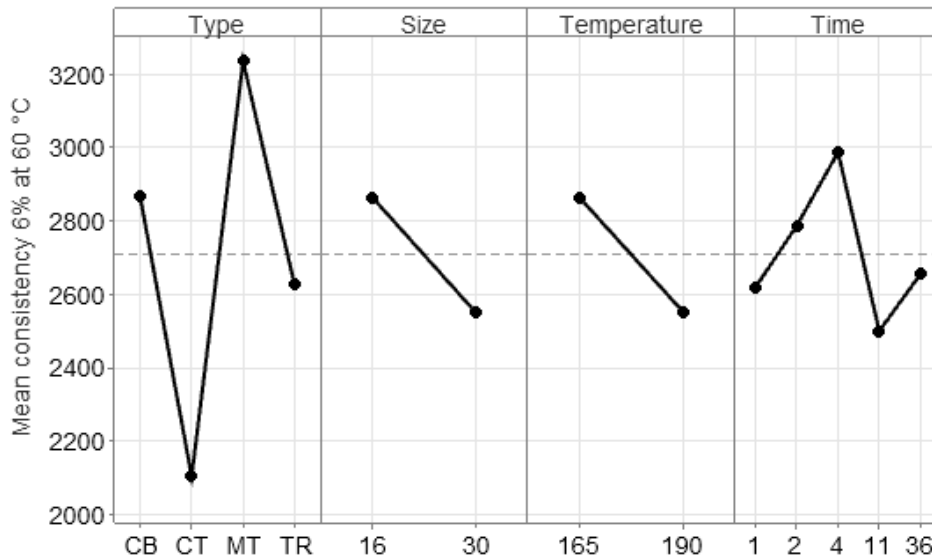
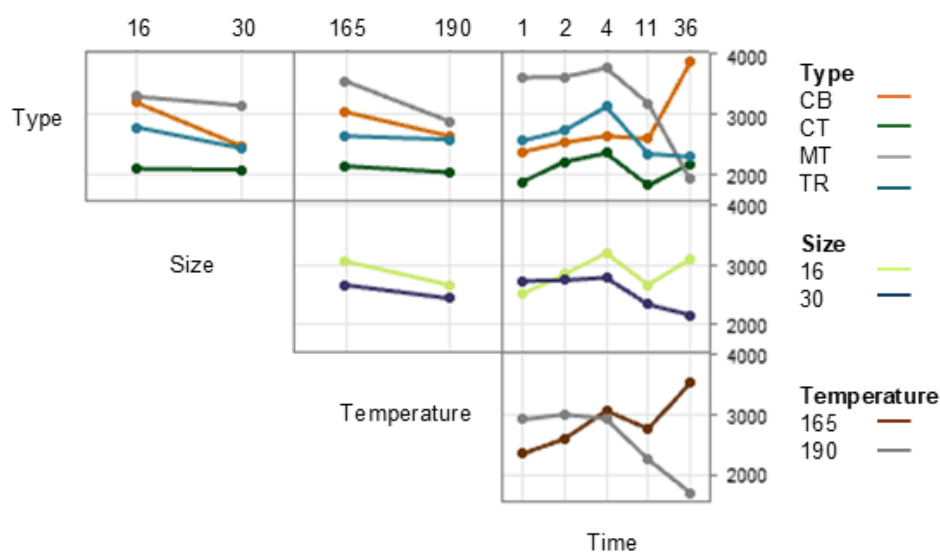


Figure 4.66 presents the interaction plots between the investigated parameters for the consistency 6% at 60 °C results. As expected, interactions were primarily observed between blending time and all other parameters. Between crumb rubber type and blending time, interactions can be seen between 11 and 36 hours of blending. During that time, the consistency 6% at 60 °C for CRMBs produced with TR-, CT- and, most notably, CB-derived crumb rubber was found to increase, whereas that of MT-derived crumb rubber was found to decrease. Interactions between blending time and crumb rubber size gradation were also observed during 11 and 36 hours of blending. During that time, the consistency 6% at 60 °C of CRMBs produced with S16 crumb rubber particles increased, whereas that of CRMBs produced with S30 crumb rubber particles decreased. Blending temperature and time also presented interactions when blending was undertaken for 11 and 36 hours. The consistency 6% at 60 °C was found to increase during that time when binders were blended at 165 °C and decrease when binders were blended at 190 °C.

Figure 4.66: Interaction plots of crumb rubber type, crumb rubber size, blending temperature and blending time for consistency 6% at 60 °C



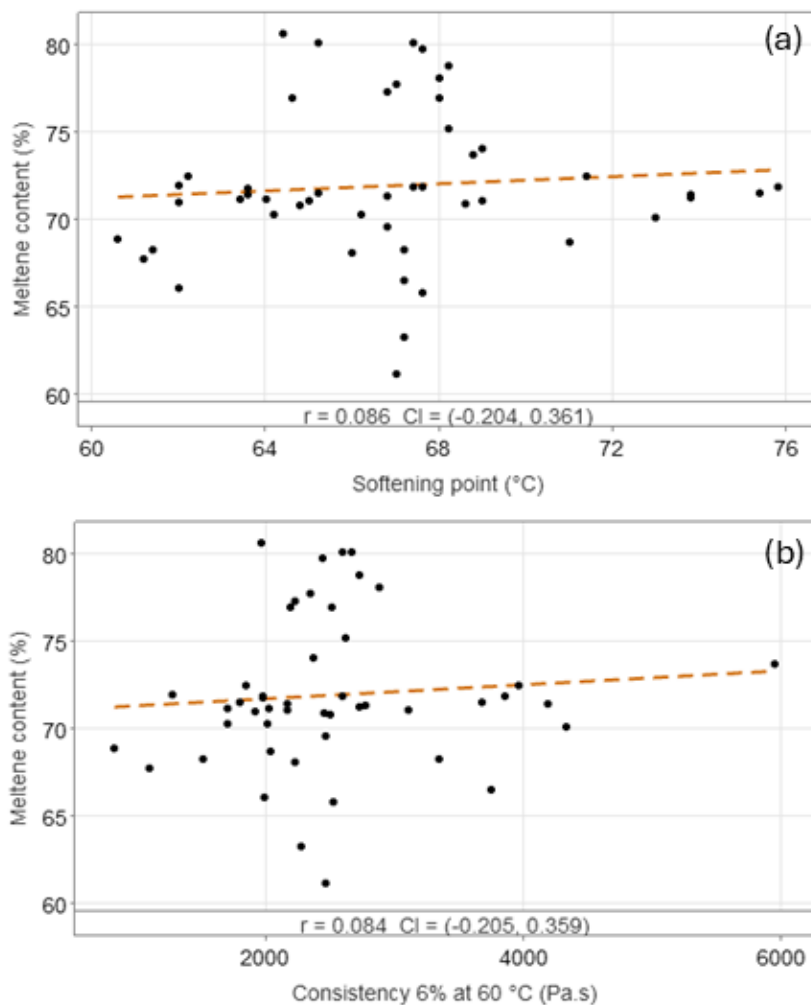
The Austroads ATS 3110:2023 minimum consistency 6% at 60 °C limit of 1,000 Pa.s was met by all CRMBs, with the only exception of MT30_190C_36h.

4.6.3 Summary of Findings and Discussion

Softening point and consistency 6% at 60 °C are both used as measures of rutting resistance for the resultant asphalt mixes. Both tests indicate that MT-derived CRMBs may result in asphalt mixes with greater rutting resistance when compared to asphalt made using TR-, CT- or CB-derived CRMBs. Irrespective of the test used, however, it was found that the properties of MT-derived CRMBs diminished when blending was extended to 36 hours, which was not the case for the rest of the CRMBs. Notably, repeat testing of CB16_165C_36h showed that its consistency 6% at 60 °C was significantly greater than all others.

Grobler (2020) expressed that softening point increases as the crumb rubber particles transition to becoming gel-like as they absorb the bitumen's light components. An increased softening point, therefore, is a measure of increased stiffness (Airey et al. 2003). Consistency 6% is also a measure of stiffness, more directly, as it is calculated to be the stress at 0.06 strain over the actual strain rate during testing (AGPT-T121:2014). In this research, the concentration of light components in the liquid phase of the binder were quantified using HPLC-GPC, as presented in Section 4.2.3. Figure 4.67 shows, however, that the results from both tests had a negligible correlation with the maltene content ($|r| < 0.09$).

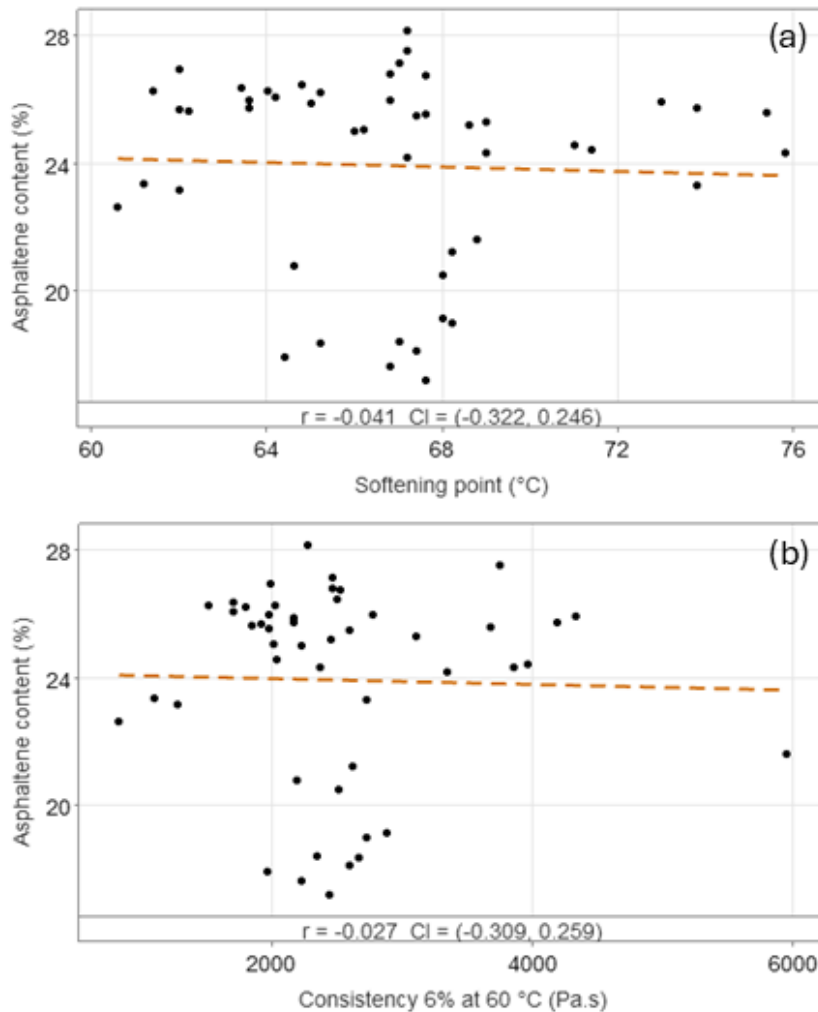
Figure 4.67: Correlation between (a) softening point and (b) consistency 6% at 60 °C with maltene content as measured by HPLC-GPC



An increase in the stiffness of the binders could also be the result of oxidation. The viscosity at 60, 165 and 175 °C results, as well as those of softening point for blended unmodified C170, suggest that some oxidation of the binder can be expected during the sample preparation method described in Section 3.2.1. Oxidation

typically results in an increase in the concentration of apparent asphaltenes (Primerano et al. 2024). Figure 4.68 shows the correlation between asphaltene content and stiffness was also negligible ($|r| < 0.09$).

Figure 4.68: Correlation between (a) softening point and (b) consistency 6% at 60 °C with asphaltene content as measured by HPLC-GPC



It may, therefore, be suggested that the stiffness of the asphalt mix is impacted by the combination of crumb rubber swelling and oxidation of the liquid phase, with not one single mechanism having a more prominent impact on the results.

4.7 Fatigue Life

The fatigue life of a pavement is defined by its ability to resist cracking under repeated loading. It is one of the most common failure mechanisms in asphalt pavements, especially at low temperature environments (Sudarsanan & Kim 2022). Section 4.7.1 describes the expected resistance to cracking of the CRMBs investigated in this research as measured through the stress ratio at 10 °C test.

4.7.1 Stress Ratio at 10 °C

The stress ratio at 10 °C test is used as a quality control test to rank the low temperature cracking performance of modified binders in Australia. It has been found to have some correlation with asphalt fatigue life when only the binder is varied within an asphalt mix (i.e. assuming all other aspects of the mix remain the same), in which case the ability of the asphalt mix to resist failure under repeat loading would be dependent on the ability of the binder to resist cracking (Austroads 2017). The greater the stress ratio at 10 °C result, the greater the expected fatigue life of the resultant asphalt.

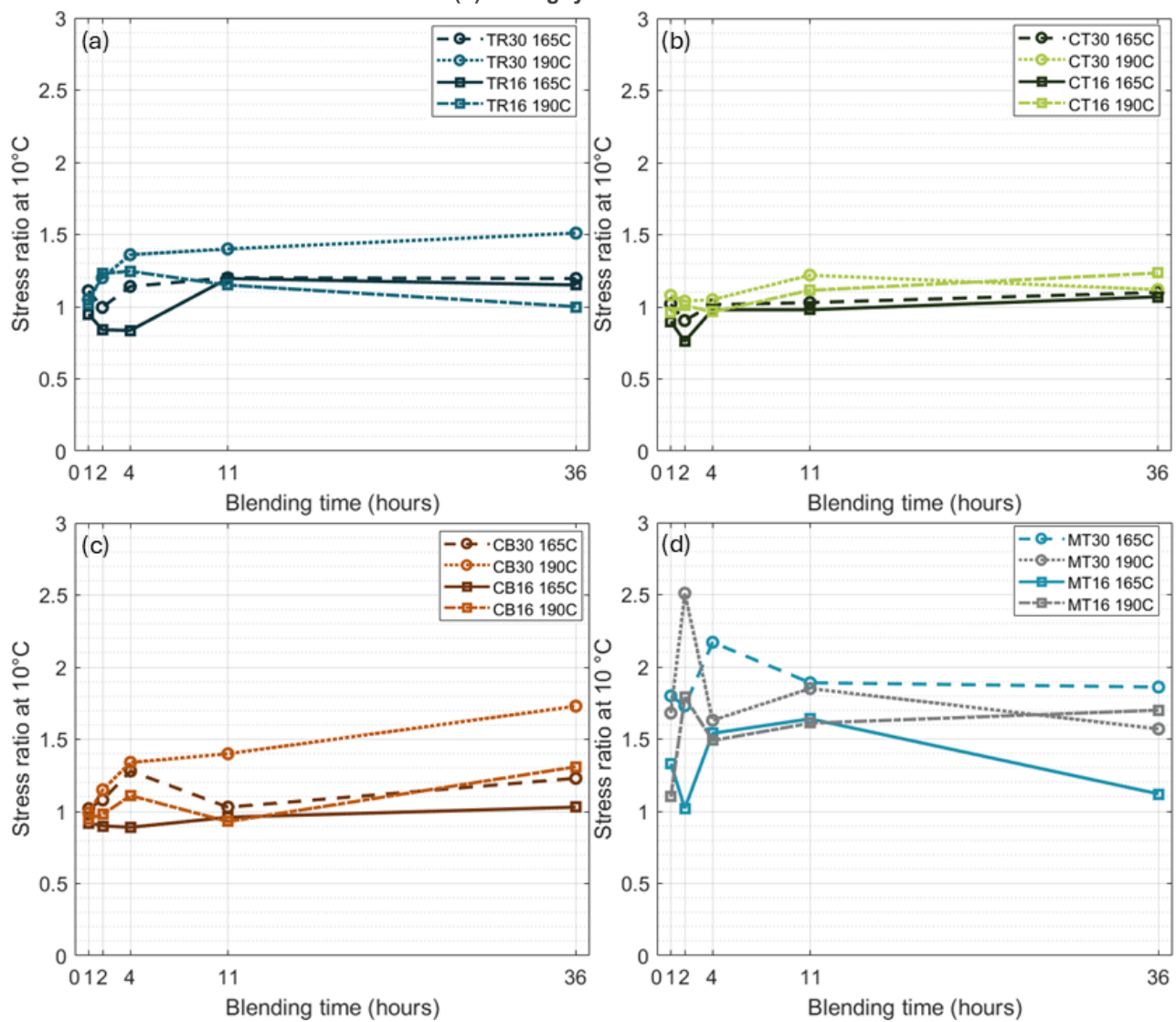
Table 4.27 presents the stress ratio at 10 °C results for the investigated CRMBs. The greatest stress ratio at 10 °C was measured for sample MT30_190C_2h and the lowest for sample CT16_165C_2h at 2.51 and 0.76, respectively. There is no requirement for the stress ratio at 10 °C test in MRWA Specification 511:2025, and according to Austroads ATS 3110:2023, the results are to be reported.

Table 4.27: Stress ratio at 10 °C results for all binders

Samples	Digestion time					
	1h	2h	4h	11h	24h	36h
TR30_165C_	1.11	0.99	1.14	1.20	–	1.19
TR30_190C_	1.05	1.20	1.36	1.40	–	1.51
TR16_165C_	0.95	0.84	0.83	1.19	–	1.15
TR16_190C_	1.01	1.23	1.24	1.15	–	1.00
CT30_165C_	1.02	0.95	1.01	1.03	–	1.10
CT30_190C_	1.08	1.04	1.05	1.22	–	1.12
CT16_165C_	0.90	0.76	0.98	0.98	–	1.07
CT16_190C_	0.96	1.01	0.96	1.11	–	1.23
CB30_165C_	1.02	1.08	1.28	1.03	–	1.23
CB30_190C_	0.99	1.15	1.34	1.40	–	1.73
CB16_165C_	0.92	0.90	0.89	0.96	–	1.03
CB16_190C_	0.95	0.98	1.11	0.93	–	1.31
MT30_165C_	1.80	1.73	2.17	1.89	–	1.86
MT30_190C_	1.68	2.51	1.63	1.85	–	1.57
MT16_165C_	1.33	1.02	1.54	1.64	–	1.12
MT16_190C_	1.10	1.79	1.49	1.61	–	1.70

Figure 4.69 graphically presents the stress ratio at 10 °C results. The stress ratio at 10 °C of TR-derived CRMBs varied by 0.68, that of CT-derived CRMBs varied by 0.46, that of CB-derived CRMBs varied by 0.84 and that of MT-derived CRMBs by 1.49, depending on the parameters.

Figure 4.69: Blending time versus stress ratio at 10 °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



The ANOVA results of Table 4.28 reveal that all investigated parameters had a statistically significant effect on the stress ratio at 10 °C results.

Table 4.28: ANOVA table of the effect of crumb rubber type and size and blending temperature and time on stress ratio at 10 °C

Source	DF	Adj SS	Adj MS	F-value	P-value
Type	3	9.6177	3.20589	66.56	0.000
Size	1	1.9031	1.90314	39.51	0.000
Temperature	1	0.6163	0.61628	12.79	0.000
Time	4	0.7649	0.19123	3.97	0.004
Error	150	7.2251	0.04817		
Lack of fit	70	4.6642	0.06663	2.08	0.001
Pure error	80	2.5608	0.03201		
Total	159	20.1271			

Notes: DF = degrees of freedom, Adj = adjusted, SS = sum of squares, MS = mean square.

Figure 4.70 presents the mean main effects of the investigated parameters on the stress ratio at 10 °C results. It is shown that MT-derived CRMBs resulted in the greatest mean stress ratio at 10 °C. In addition, the mean stress ratio at 10 °C increased with a change in crumb rubber gradation from S16 to S30 and in

blending temperature from 165 to 190 °C. It is further shown that the mean stress ratio at 10 °C increased with an increase in blending time.

Figure 4.70: Main effects plots of crumb rubber type, crumb rubber size, blending temperature and blending time on stress ratio at 10 °C

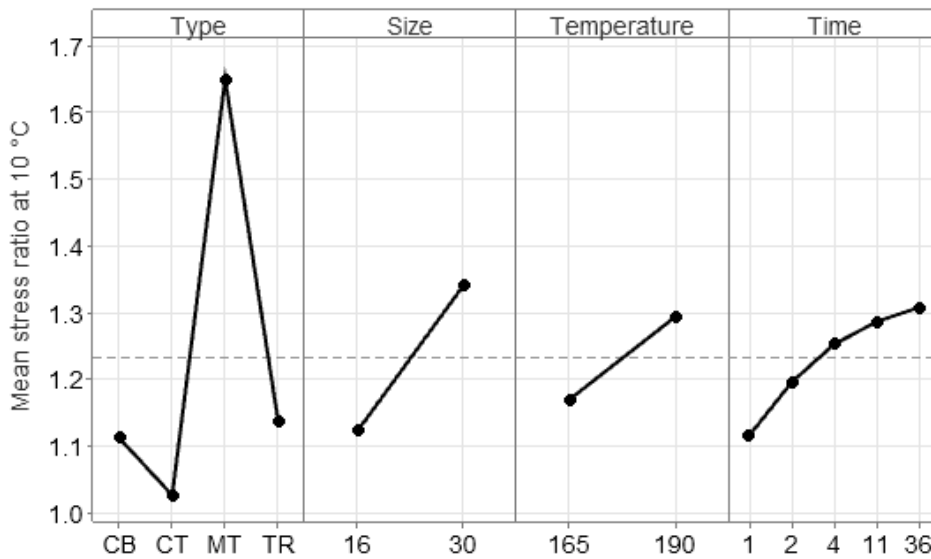
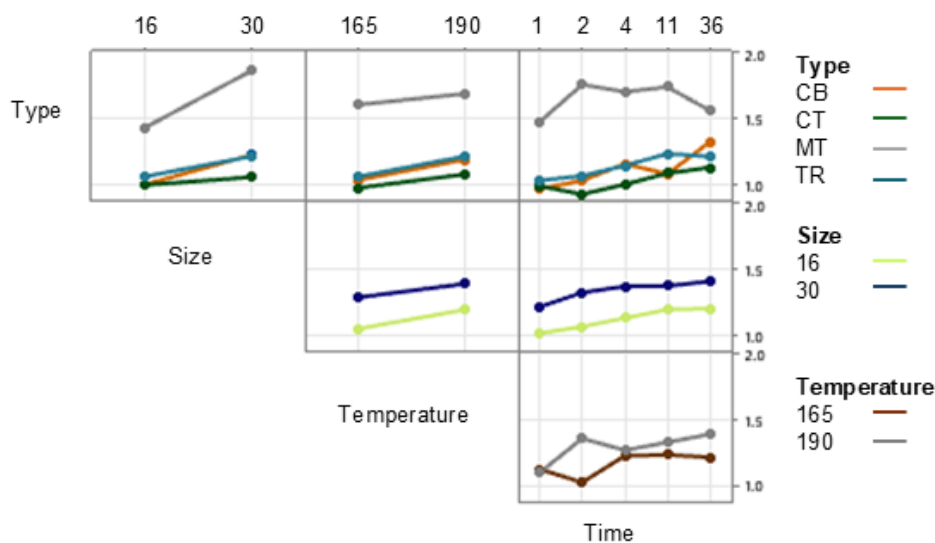


Figure 4.71 presents the interaction plots between the investigated parameters. The crumb rubber type was found to interact with blending time. Between 1 and 2 hours of blending, the stress ratio at 10 °C of CT-derived CRMBs was found to decrease, whereas that of all other binders was found to increase. Between 2 and 4 hours of blending, the stress ratio of MT-derived CRMBs was found to decrease, whereas that of all others was found to increase. Between 4 and 11 hours of blending, the stress ratio at 10 °C of CB-derived CRMBs was found to decrease, whereas that of all the others was found to increase, and between 11 and 36 hours of blending the stress ratio at 10 °C of CT- and CB-derived CRMBs was found to increase, whereas that of the other 2 decreased.

Crumb rubber size was not found to interact with any of the other investigated parameters affecting the stress ratio at 10 °C results. Interaction between blending temperature and time were, however, observed throughout the blending times investigated.

Figure 4.71: Interaction plots of crumb rubber type, crumb rubber size, blending temperature, and blending time for stress ratio at 10 °C



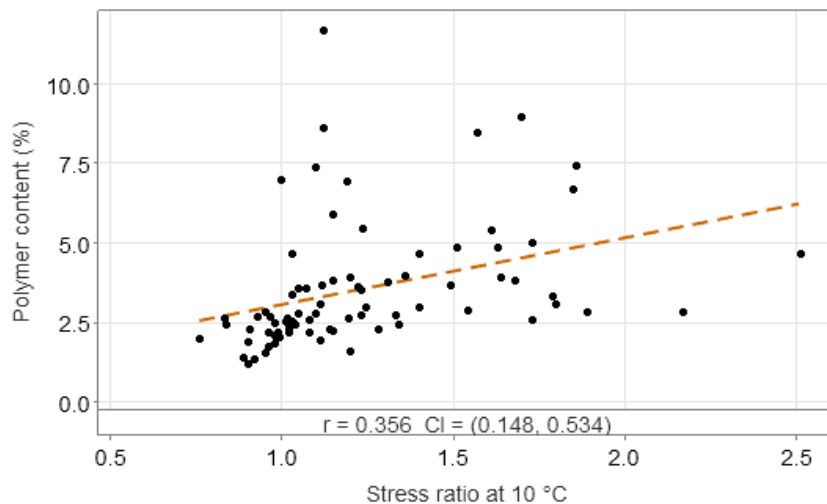
4.7.2 Summary of Findings and Discussion

The stress ratio at 10 °C results suggest that asphalt produced with MT-derived CRMBs will have a greater fatigue life when compared to asphalt produced with any of the other crumb rubber types, as is also supported by the main effects plot of Figure 4.70.

Austrroads (2017, Figure 7.2) presented a correlation between stress ratio at 10 °C and asphalt fatigue life. Based on these findings, the test was determined to be able to reasonably rank the binder impacts on asphalt fatigue life. According to Figure 7.2 in Austrroads (2017), a notable increase in fatigue life of the asphalt (by an order of magnitude, in cycles) could be achieved when the stress ratio at 10 °C result increased from approximately 1 to > 1.5. This increase was only evident between the stress ratio at 10 °C of MT-derived CRMBs when compared to CRMBs produced using TR-, CT- and CB-derived crumb rubber.

The fatigue life of asphalt is expected to be positively impacted by improved elasticity in the binder. According to Sections 2.2 and 4.5, binder elasticity is expected to increase with an increase in polymer content (presence of long molecular chains) in the liquid phase of the binder. Figure 4.72 shows a weak correlation ($|r| < 0.39$) between the stress ratio at 10 °C results and the polymer content in the binders' liquid phase.

Figure 4.72: Correlation between stress ratio at 10 °C and polymer content as measured by HPLC-GPC



It is worth noting the morphology of the stress-strain plots of the tested binders, shown in Appendix C.2, based on which the stress ratio at 10 °C was calculated. In most cases, the morphology of the curve between the 2 specimens tested per sample show significant differences and inconsistent fluctuations, suggesting that the results were impacted not only by the elasticity and stiffness of the binders but also by physical interactions among the crumb rubber particles present. As expected, these were typically observed at shear strain above 5 (increased displacement) and were more pronounced for binders produced with S16 crumb rubber particles.

5 Discussion

5.1 Binder Oxidation

It has previously been recognised that short-term ageing of bitumen is likely to occur during blending at elevated temperatures (Hofko et al. 2018). Therefore, this section summarises the effect of bitumen oxidation on the results of Section 4, in an effort to distinguish it from observations relating to the digestion of the investigated crumb rubbers.

Based on the mean viscosity effects of Sections 4.4.1, and 4.6.1, a spike in viscosity is observed after 4 hours of blending, which is followed by a decrease after 11 hours of blending. Provided that this decrease is then followed by subsequent increases in viscosity, it was hypothesised that crumb rubber degradation was not the cause for that decrease in viscosity between 4 and 11 hours. Rather, it is suspected that this spike in viscosity at 4 hours of blending is due to increased oxidation caused by the increased surface area of the binder and the consequent increased headspace in the tin, which resulted from the 2 subsampling occurrences at 1 and 2 hours of blending.

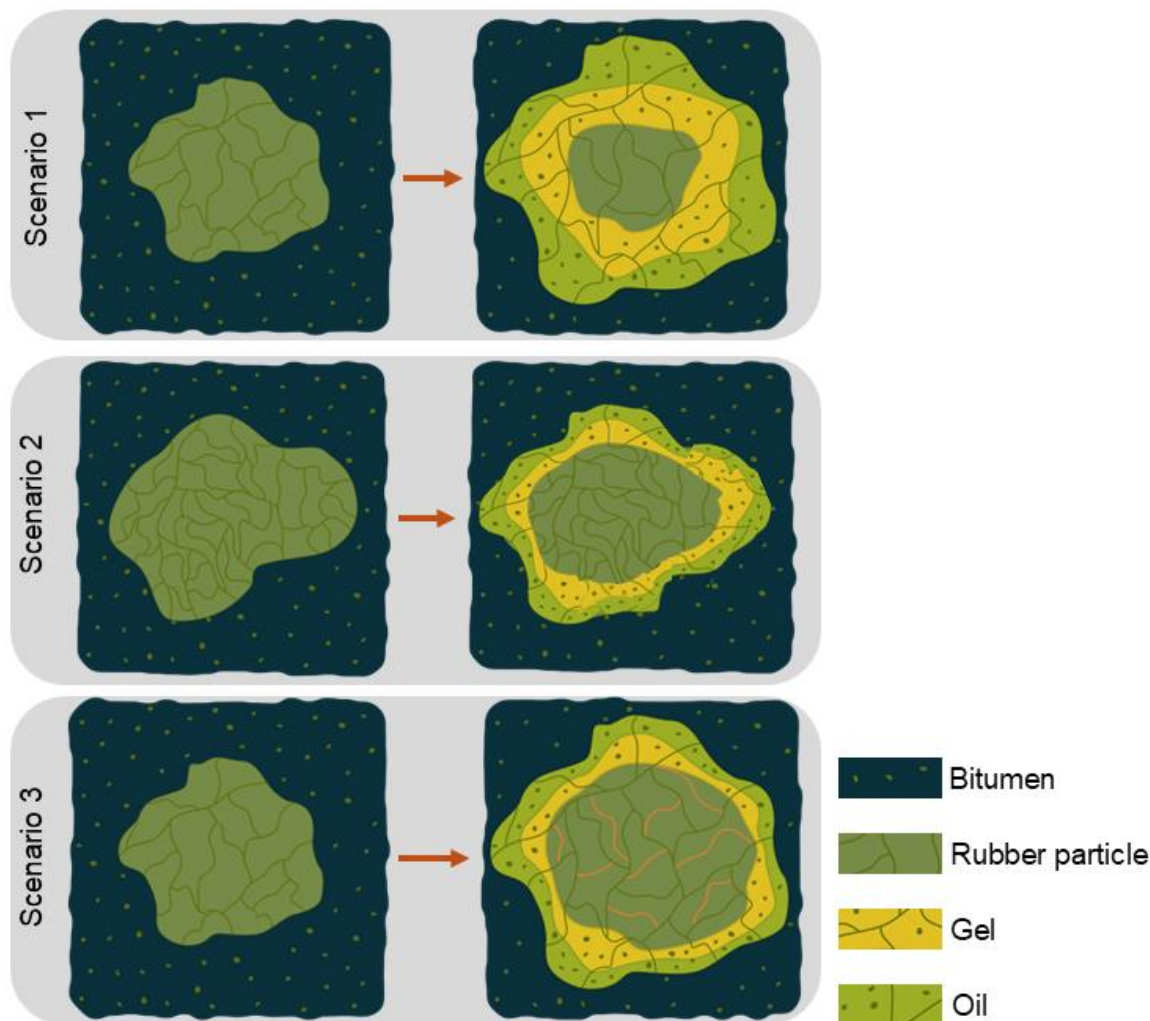
From the viscosity at 60, 165 and 175 °C, as well as the softening point results for the unmodified C170 presented in Sections 4.1.2, 4.4.1 and 4.6.1, it is indicated that the bitumen was oxidised during blending. Oxidation of bitumen in the CRMBs is expected to affect their performance. However, this is not to suggest that the oxidation mechanism would be always the same or that it would always occur at the same rate and to the same extent. It has previously been discussed that ageing effects are more prominent for unmodified bitumen when compared to CRMBs (Ali et al. 2013). Regardless, attempts to correlate observed stiffening effects with measured maltenes and apparent asphaltenes failed to demonstrate a strong dependence. This is suspected to be due to both bitumen oxidation and crumb rubber swelling contributing to measured increased stiffness.

5.2 Effect of Crumb Rubber Type on Digestion

According to Section 2, during the digestion of crumb rubber in bitumen, light components from the bitumen are absorbed by the crumb rubber at early stages, then NR dissolves into the bitumen, and at elevated temperatures and extended digestion times some SR or other polymers present may also dissolve into the bitumen. Increased crumb rubber particle surface area (smaller particles, irregular surface morphology), higher temperature and greater free volume within the rubber molecular structure all facilitate this digestion process. Hence, during the production of CRMBs, these stages are understood to be taking place simultaneously due to crumb rubber gradation, whereby smaller particles progress through these stages faster than larger particles.

The results of Section 4.2 show that TR-derived rubber had the greatest rate of swelling, followed by CB-derived rubber, then MT-derived rubber and, lastly, by CT-derived rubber. In all cases, the rate of swelling at 190 °C was greater than that at 165 °C. Based on these findings and theoretical expectations set by Section 2, it is hypothesised that there are 3 possible scenarios of the behaviour of these different types of rubber in bitumen, as schematically represented in Figure 5.1. Scenario 1 represents a rubber type that has a relatively greater free volume and upon exposure to bitumen at elevated temperatures, it notably swells, absorbing the light components from the bitumen. Scenario 2 schematically depicts a rubber type with a comparatively greater number of cross-links and, so, a condensed molecular structure. Scenario 3 shows a rubber type that in its as-received form has a relatively sparse molecular structure, but upon exposure to elevated temperatures, it has the propensity to further cross-link.

Figure 5.1: Schematic illustration of swelling behaviour dependence on rubber type



Note: Orange lines represent subsequent cross-links.

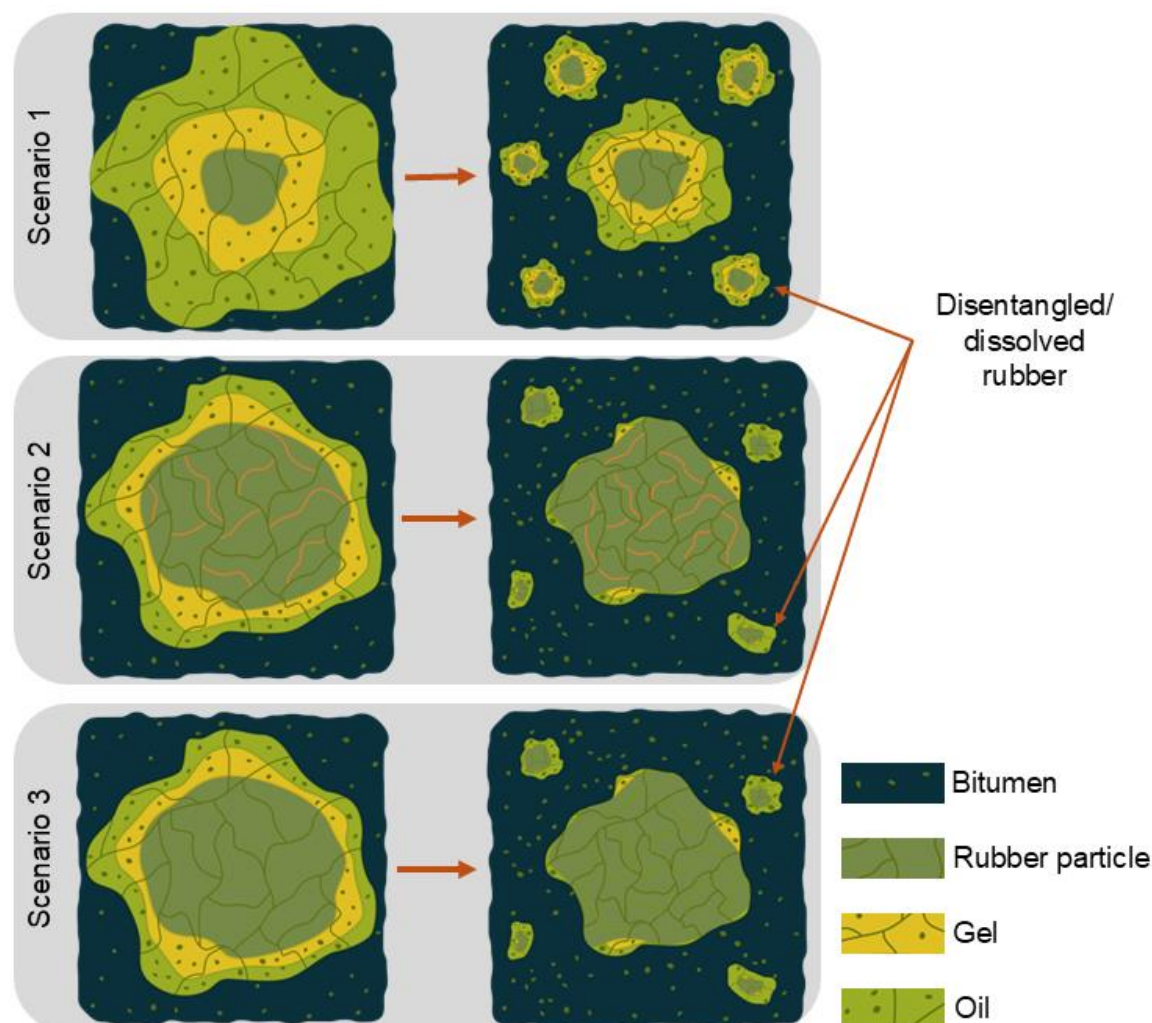
According to Ren et al. (2021), NR is more prone to swelling when compared to SR. Based on the results of Section 4.2.4 (Figure 4.16), MT-derived crumb rubber had the greatest NR content at $42.7 \pm 0.6\%$, followed by TR-derived crumb rubber at $35.0 \pm 4.6\%$, then CT-derived crumb rubber at $33.3 \pm 0.6\%$ and lastly CB-derived crumb rubber at $28.0 \pm 1.1\%$. These do not agree with the ranking for swelling rate in this research. It is, therefore, possible to suggest that more than just the content of NR determines the swelling of the different rubber types. Based on the findings of this research, it is thought that the rubber types investigated in this research can be related to the hypothesised scenarios of Figure 5.1 as:

- TR-derived crumb rubber belongs to Scenario 1: relatively greater NR content that does not subsequently cross-link upon exposure to elevated temperature
- CT-derived crumb rubber belongs to Scenario 2: comparative content of NR to that of TR-derived crumb rubber, but with less flexible/more dense chains restricting the absorption of the bitumen's light components
- CB-derived crumb rubber belongs to Scenario 2: relatively lower NR concentration
- MT-derived crumb rubber belongs to Scenario 3: greatest NR concentration; however, with the propensity to cross-link upon exposure to elevated temperature consequently hindering the absorption of the bitumen's light components.

The HPLC-GPC results of Section 4.2.3 showed a dependency of polymer dissolution on crumb rubber particle size, rubber type and blending temperature. Three scenarios for crumb rubber dissolution have been hypothesised to support these findings, which are schematically illustrated in Figure 5.2. Scenario 1 shows dissolution that occurs after extensive exposure to bitumen at elevated temperatures. Scenario 1 would require rubber with relatively high free volume, comparatively low temperature and rubber that does not subsequently cross-link upon exposure to elevated temperature. Scenario 2 shows dissolution that occurs

after prolonged exposure (comparatively low temperature or large particle size) for rubbers that can cross-link during said exposure or for rubbers with a relatively low free volume (rigid molecular structure or high concentration of cross-links) irrespective of temperature. Scenario 3 illustrates dissolution that occurs after a relatively short exposure time at a relatively high temperature.

Figure 5.2: Schematic illustration of rubber dissolution scenarios



Note: Orange lines represent subsequent cross-links or a denser cross-link network.

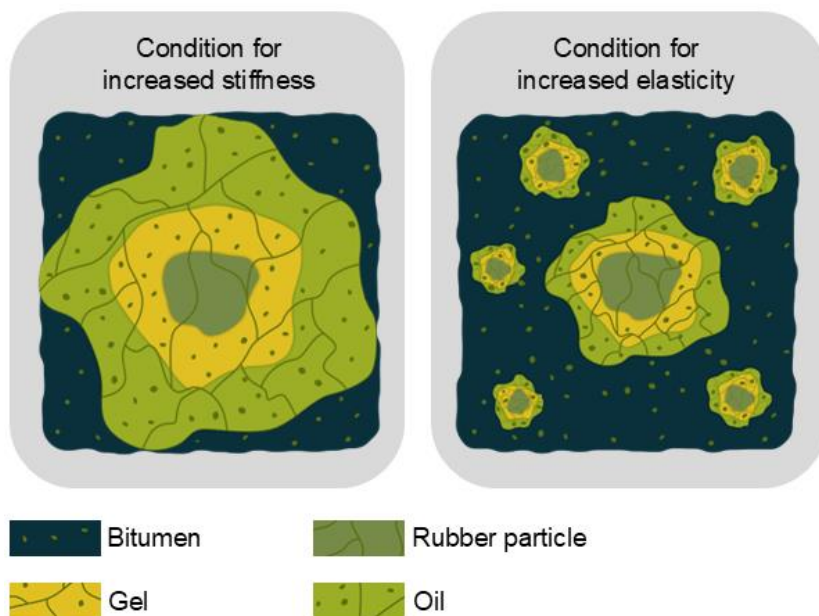
According to Ghavibazoo and Abdelrahman (2013), the oily components are the ones mainly impacted at lower temperatures, whereas NR has a greater propensity to dissolution when compared to SR. Based on the TGA results of Section 4.2.4, the concentration of oil and moisture content of all crumb rubber types notably decreased following blending of as little as 1 hour irrespective of temperature. In addition, the total rubber content (NR+SR) generally decreased with blending temperature for all investigated rubber types, suggesting that some polymer dissolution in bitumen may be expected, while the SR/NR ratio generally increased, suggesting that NR is the rubber component preferentially dissolving, as also suggested by Ghavibazoo and Abdelrahman (2013).

It needs to be noted that the scenarios of Figure 5.1 and Figure 5.2 do not consider changes in the bitumen as a result of oxidation, which as noted in Section 5.1, is expected to have taken place in this work. These scenarios do not suggest that bitumen oxidation does not happen either, though. They only assume that it is the same in all cases, which is reasonable as the binders were handled as consistently as possible.

5.3 Effect of Crumb Rubber Type on Binder Properties

According to the findings of Section 2, the process of crumb rubber swelling results in the increase in stiffness as a result of the bitumen light components being absorbed by the rubber particles and increased interparticle interactions due to the increased particle size (due to swelling). Crumb rubber dissolution, on the other hand has been linked to increased binder elasticity due to the presence of larger molecular weight chains in the liquid phase of the binder (portion of rubber < 75 µm). These 2 conditions are schematically represented in Figure 5.3.

Figure 5.3: Schematic illustration of crumb rubber/bitumen conditions for increased stiffness and elasticity of the crumb rubber-modified binders



The results of this research, as presented in Section 4.5, suggest that MT- and CB-derived CRMBs may present a greater elasticity when compared to TR- and CT-derived CRMBs. The results of Section 4.6 suggest that MT- and CB-derived CRMBs may present a greater stiffness when compared to TR- and CT-derived CRMBs as well, while MT-derived CRMBs may also produce asphalt with improved fatigue life, according to Section 4.7. This is not true for all combinations of crumb rubber size and blending temperatures and time. It is based on the mean main effect observations.

These observations could not be directly linked to the crumb rubber digestion analysis of Section 4.2, probably because of, as previously mentioned, the gradation of the crumb rubber particles introduced, which progress through the various digestion stages at a different rate. It is also suspected that some of the tests used in this research are more suitable to assess the liquid phase of the CRMBs; others best describe the solid phase, and some may be impacted by the composite as a whole.

Appendix E summarises how the tested CRMBs behaved against MRWA Specification 511:2025 and Austroads ATS 3110:2023 requirements, respectively. The results of this research indicate that, if used in isolation (i.e. the rubber types in the CRMB are not mixed), S16 MT-derived crumb rubber may not be suitable for use while S30 MT-derived crumb rubber may only be used if blended at comparatively lower temperatures and for short blending times (transportation only required for a short distance). Therefore, despite the fact that MT-derived CRMBs showed potential to support the production of asphalt with comparatively improved properties, they may only be introduced under controlled processing conditions. CB-derived CRMBs, on the other hand, were found to be able to more readily replace the commonly used TR- and CT-derived CRMBs (noting that exposure at elevated temperatures according to MRWA Specification 511:2025 is only permitted for up to 11 hours, including the first hour of blending).

However, in practice, it is likely that the various rubber types will be combined at some point throughout the production process. In addition, a demonstration trial conducted by Austroads (2024) did not report the

requirement for significant adjustments to asphalt practices during the construction of asphalt with S30 TR-, CT- and MT-derived CRMBs.

In addition, there is a wide range of factors that affect asphalt mix performance, and the range of binder performance test results should be considered in this context.

Compliance with Main Roads Western Australia and Austroads Technical Specification requirements

Appendix E summarises the compliance of all assessed CRMBs according to MRWA Specification 511:2025 and Austroads ATS 3110:2023 in a tabulated format. Overall, with only one minor exception, the results for all TR-, CT- and CB-derived CRMBs were within the limits of MRWA Specification 511:2025 and Austroads ATS 3110:2023. This suggests that crumb rubber from these 3 sources could be used for asphalt applications with relatively high confidence.

Results from MT-derived CRMBs presented great variability and inconsistencies. Test results from these CRMBs were often not within the limits of MRWA Specification 511:2025 and Austroads ATS 3110:2023. It is not known how the manufacturing process for deriving consistent crumb rubber from mining tyres or the composition of the tyres may affect these findings. Further work is necessary.

6 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 in 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 the 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. 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.

Analytical methods, including rubber swelling, TGA and HPLC-GPC, were found to be useful in assessing the digestion behaviour of the different rubber types under the different conditions investigated. It was, however, not possible to directly link these findings with the results of the handling and performance properties of the resultant CRMBs. An extensive study conducted by Shen et al. (2009), which included the testing of 108 different CRMBs, revealed that even though some trends in the results could be observed, many of the tested CRMBs did not follow them. This highlights the complexity of the crumb rubber/bitumen composite system and supports the observations of this research.

The test results relating to handling properties, as well as observations by handling MT-derived CRMBs in the laboratory, suggested that they may introduce challenges during production. In addition, MT-derived CRMBs were found to not comply with both MRWA Specification 511:2025 and Austroads ATS 3110:2023 under all conditions.

A demonstration trial by Austroads (2024) reported that there was no need to modify current practice during the manufacture and construction of asphalt produced with TR-, CT- and MT-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.

Recommendations

Overall, regarding WA practice, it may be recommended that CB-derived crumb rubber can be used to replace, fully or partially, TR- and/or CT-derived crumb rubber in the production of CRMBs. MT-derived crumb rubber, however, may need to be considered more carefully, as even though it was found to meet the limits of MRWA Specification 511:2025, its comparatively increased viscosity at 165 and 175 °C suggests that it may introduce some handling challenges. In practice, it is likely that the different crumb rubber types will be introduced as a blend from EoL rubber product recyclers, which will probably mean that any such observations will be somewhat diminished.

Regardless, it is recommended that additional work, including better understanding of the range of rubber composition and processing methods, be undertaken before MT-derived crumb rubber is considered a direct alternative to the other 3 sources investigated in this research.

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Appendix A Summary of Samples Assessed per Test

Table A.1: Samples assessed through thermogravimetric analysis

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓			✓		✓
TR30_190C_		✓			✓		✓
TR16_165C_		✓			✓		✓
TR16_190C_		✓			✓		✓
CT30_165C_		✓			✓		✓
CT30_190C_		✓			✓		✓
CT16_165C_		✓			✓		✓
CT16_190C_		✓			✓		✓
CB30_165C_		✓			✓		✓
CB30_190C_		✓			✓		✓
CB16_165C_		✓			✓		✓
CB16_190C_		✓			✓		✓
MT30_165C_		✓			✓		✓
MT30_190C_		✓			✓		✓
MT16_165C_		✓			✓		✓
MT16_190C_		✓			✓		✓
TR	✓						
CT	✓						
CB	✓						
MT	✓						
TR30 after Soxhlet	✓						
TR16 after Soxhlet	✓						
CT30 after Soxhlet	✓						
CT16 after Soxhlet	✓						
CB30 after Soxhlet	✓						
CB16 after Soxhlet	✓						
MT30 after Soxhlet	✓						
MT16 after Soxhlet	✓						

Table A.2: Samples assessed through high-performance liquid chromatography – gas permeation chromatography

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓			✓		✓
TR30_190C_		✓			✓		✓
TR16_165C_		✓			✓		✓
TR16_190C_		✓			✓		✓
CT30_165C_		✓			✓		✓
CT30_190C_		✓			✓		✓
CT16_165C_		✓			✓		✓
CT16_190C_		✓			✓		✓
CB30_165C_		✓			✓		✓
CB30_190C_		✓			✓		✓
CB16_165C_		✓			✓		✓
CB16_190C_		✓			✓		✓
MT30_165C_		✓			✓		✓
MT30_190C_		✓			✓		✓
MT16_165C_		✓			✓		✓
MT16_190C_		✓			✓		✓
C170	✓						

Table A.3: Samples assessed through Fourier-transform infrared spectroscopy

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓			✓		✓
TR30_190C_		✓			✓		✓
TR16_165C_		✓			✓		✓
TR16_190C_		✓			✓		✓
CT30_165C_		✓			✓		✓
CT30_190C_		✓			✓		✓
CT16_165C_		✓			✓		✓
CT16_190C_		✓			✓		✓
CB30_165C_		✓			✓		✓
CB30_190C_		✓			✓		✓
CB16_165C_		✓			✓		✓
CB16_190C_		✓			✓		✓
MT30_165C_		✓			✓		✓
MT30_190C_		✓			✓		✓
MT16_165C_		✓			✓		✓
MT16_190C_		✓			✓		✓
C170	✓						
TR	✓						
CT	✓						
CB	✓						
MT	✓						

Table A.4: Samples assessed for the dissolution of crumb rubber in the binder

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓			✓		✓
TR30_190C_		✓			✓		✓
TR16_165C_		✓			✓		✓
TR16_190C_		✓			✓		✓
CT30_165C_		✓			✓		✓
CT30_190C_		✓			✓		✓
CT16_165C_		✓			✓		✓
CT16_190C_		✓			✓		✓
CB30_165C_		✓			✓		✓
CB30_190C_		✓			✓		✓
CB16_165C_		✓			✓		✓
CB16_190C_		✓			✓		✓
MT30_165C_		✓			✓		✓
MT30_190C_		✓			✓		✓
MT16_165C_		✓			✓		✓
MT16_190C_		✓			✓		✓

Table A.5: Samples assessed through particle size distribution

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR16_165C_		✓			✓		✓
TR16_190C_		✓			✓		✓
CT16_165C_		✓			✓		✓
CT16_190C_		✓			✓		✓
CB16_165C_		✓			✓		✓
CB16_190C_		✓			✓		✓
TR30	✓						
TR16	✓						
CT30	✓						
CT16	✓						
CB30	✓						
CB16	✓						
MT30	✓						
MT16	✓						

Table A.6: Samples assessed through optical microscopy

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR16_165C_		✓			✓		✓
TR16_190C_		✓			✓		✓
CT16_165C_		✓			✓		✓
CT16_190C_		✓			✓		✓
CB16_165C_		✓			✓		✓
CB16_190C_		✓			✓		✓
MT16_165C_		✓			✓		✓
MT16_190C_		✓			✓		✓
TR	✓						
CT	✓						
CB	✓						
MT	✓						

Table A.7: Samples assessed according to ATM 111 for viscosity at 165 and 175 °C

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓	✓	✓	✓	✓	✓
TR30_190C_		✓	✓	✓	✓	✓	✓
TR16_165C_		✓	✓	✓	✓	✓	✓
TR16_190C_		✓	✓	✓	✓	✓	✓
CT30_165C_		✓	✓	✓	✓	✓	✓
CT30_190C_		✓	✓	✓	✓	✓	✓
CT16_165C_		✓	✓	✓	✓	✓	✓
CT16_190C_		✓	✓	✓	✓	✓	✓
CB30_165C_		✓	✓	✓	✓	✓	✓
CB30_190C_		✓	✓	✓	✓	✓	✓
CB16_165C_		✓	✓	✓	✓	✓	✓
CB16_190C_		✓	✓	✓	✓	✓	✓
MT30_165C_		✓	✓	✓	✓	✓	✓
MT30_190C_		✓	✓	✓	✓	✓	✓
MT16_165C_		✓	✓	✓	✓	✓	✓
MT16_190C_		✓	✓	✓	✓	✓	✓
C170	✓						
C170_165C_		✓					✓
C170_190C_		✓	✓	✓	✓	✓	✓

Table A.8: Samples assessed following AGPT-T125

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓	✓	✓	✓		✓
TR30_190C_		✓	✓	✓	✓		✓
TR16_165C_		✓	✓	✓	✓		✓
TR16_190C_		✓	✓	✓	✓		✓
CT30_165C_		✓	✓	✓	✓		✓
CT30_190C_		✓	✓	✓	✓		✓
CT16_165C_		✓	✓	✓	✓		✓
CT16_190C_		✓	✓	✓	✓		✓
CB30_165C_		✓	✓	✓	✓		✓
CB30_190C_		✓	✓	✓	✓		✓
CB16_165C_		✓	✓	✓	✓		✓
CB16_190C_		✓	✓	✓	✓		✓
MT30_165C_		✓	✓	✓	✓		✓
MT30_190C_		✓	✓	✓	✓		✓
MT16_165C_		✓	✓	✓	✓		✓
MT16_190C_		✓	✓	✓	✓		✓

Table A.9: Samples assessed for torsional recovery at 25 °C according to ATM 122

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓	✓	✓	✓	✓	✓
TR30_190C_		✓	✓	✓	✓	✓	✓
TR16_165C_		✓	✓	✓	✓	✓	✓
TR16_190C_		✓	✓	✓	✓	✓	✓
CT30_165C_		✓	✓	✓	✓	✓	✓
CT30_190C_		✓	✓	✓	✓	✓	✓
CT16_165C_		✓	✓	✓	✓	✓	✓
CT16_190C_		✓	✓	✓	✓	✓	✓
CB30_165C_		✓	✓	✓	✓	✓	✓
CB30_190C_		✓	✓	✓	✓	✓	✓
CB16_165C_		✓	✓	✓	✓	✓	✓
CB16_190C_		✓	✓	✓	✓	✓	✓
MT30_165C_		✓	✓	✓	✓	✓	✓
MT30_190C_		✓	✓	✓	✓	✓	✓
MT16_165C_		✓	✓	✓	✓	✓	✓
MT16_190C_		✓	✓	✓	✓	✓	✓

Table A.10: Samples assessed for resilience at 25 °C following ASTM D5329-20

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓					✓
TR30_190C_		✓					✓
TR16_165C_		✓					✓
TR16_190C_		✓					✓
CT30_165C_		✓					✓
CT30_190C_		✓					✓
CT16_165C_		✓					✓
CT16_190C_		✓					✓
CB30_165C_		✓					✓
CB30_190C_		✓					✓
CB16_165C_		✓					✓
CB16_190C_		✓					✓
MT30_165C_		✓					✓
MT30_190C_		✓					✓
MT16_165C_		✓					✓
MT16_190C_		✓					✓

Table A.11: Samples assessed for softening point following AGPT-T131

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓	✓	✓	✓	✓	✓
TR30_190C_		✓	✓	✓	✓	✓	✓
TR16_165C_		✓	✓	✓	✓	✓	✓
TR16_190C_		✓	✓	✓	✓	✓	✓
CT30_165C_		✓	✓	✓	✓	✓	✓
CT30_190C_		✓	✓	✓	✓	✓	✓
CT16_165C_		✓	✓	✓	✓	✓	✓
CT16_190C_		✓	✓	✓	✓	✓	✓
CB30_165C_		✓	✓	✓	✓	✓	✓
CB30_190C_		✓	✓	✓	✓	✓	✓
CB16_165C_		✓	✓	✓	✓	✓	✓
CB16_190C_		✓	✓	✓	✓	✓	✓
MT30_165C_		✓	✓	✓	✓	✓	✓
MT30_190C_		✓	✓	✓	✓	✓	✓
MT16_165C_		✓	✓	✓	✓	✓	✓
MT16_190C_		✓	✓	✓	✓	✓	✓
C170	✓						
C170_165C_		✓					✓
C170_190C_		✓	✓	✓	✓	✓	✓

Table A.12: Samples assessed following AGPT-T121

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓	✓	✓	✓		✓
TR30_190C_		✓	✓	✓	✓		✓
TR16_165C_		✓	✓	✓	✓		✓
TR16_190C_		✓	✓	✓	✓		✓
CT30_165C_		✓	✓	✓	✓		✓
CT30_190C_		✓	✓	✓	✓		✓
CT16_165C_		✓	✓	✓	✓		✓
CT16_190C_		✓	✓	✓	✓		✓
CB30_165C_		✓	✓	✓	✓		✓
CB30_190C_		✓	✓	✓	✓		✓
CB16_165C_		✓	✓	✓	✓		✓
CB16_190C_		✓	✓	✓	✓		✓
MT30_165C_		✓	✓	✓	✓		✓
MT30_190C_		✓	✓	✓	✓		✓
MT16_165C_		✓	✓	✓	✓		✓
MT16_190C_		✓	✓	✓	✓		✓

Table A.13: Samples assessed for loss on heating following ATM 103

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓					✓
TR30_190C_		✓					✓
TR16_165C_		✓					✓
TR16_190C_		✓					✓
CT30_165C_		✓					✓
CT30_190C_		✓					✓
CT16_165C_		✓					✓
CT16_190C_		✓					✓
CB30_165C_		✓					✓
CB30_190C_		✓					✓
CB16_165C_		✓					✓
CB16_190C_		✓					✓
MT30_165C_		✓					✓
MT30_190C_		✓					✓
MT16_165C_		✓					✓
MT16_190C_		✓					✓

Table A.14: Samples assessed following ATM 132

Samples	Digestion						
	0h	1h	2h	4h	11h	24h	36h
TR30_165C_		✓					✓
TR30_190C_		✓					✓
TR16_165C_		✓					✓
TR16_190C_		✓					✓
CT30_165C_		✓					✓
CT30_190C_		✓					✓
CT16_165C_		✓					✓
CT16_190C_		✓					✓
CB30_165C_		✓					✓
CB30_190C_		✓					✓
CB16_165C_		✓					✓
CB16_190C_		✓					✓
MT30_165C_		✓					✓
MT30_190C_		✓					✓
MT16_165C_		✓					✓
MT16_190C_		✓					✓

Appendix B Blended Sample Morphology

Table B.1: Images of truck tyre-derived crumb rubber-modified binders

	1h	2h	4h	11h	24h	36h
TR30_165C						
TR30_190C						
TR16_165C						
TR16_190C						

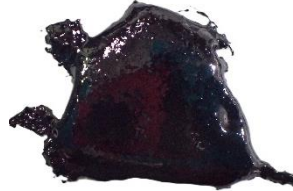
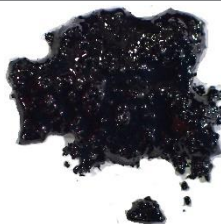
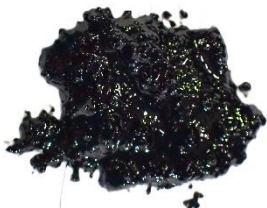
Table B.2: Images of car tyre-derived crumb rubber-modified binders

	1h	2h	4h	11h	24h	36h
CT30_165C						
CT30_190C						
CT16_165C						
CT16_190C						

Table B.3: Images of conveyor belt–derived crumb rubber-modified binders

	1h	2h	4h	11h	24h	36h
CB30_165C						
CB30_190C						
CB16_165C						
CB16_190C						

Table B.4: Images of mining tyre-derived crumb rubber-modified binders

	1h	2h	4h	11h	24h	36h
MT30_165C						
MT30_190C						
MT16_165C						
MT16_190C						

Appendix C Supplementary Results on Binder Performance Tests

C.1 Rotational Viscosity Results

Table C.1, Table C.2, Table C.3 and Table C.4 show the measured viscosity at 165 and 175 °C along with the rotations per minute (rpm) at which these measurements were taken for all binders investigated.

Table C.1: Truck tyre–derived crumb rubber-modified binders

Sample	Viscosity at 165 °C (Pa·s)	Viscosity at 165 °C rpm	Viscosity at 175 °C (Pa·s)	Viscosity at 175 °C rpm
TR30_165C_1h	1.67	50	1.33	60
TR30_165C_2h	1.68	50	1.35	60
TR30_165C_4h	2.35	30	1.75	50
TR30_165C_11h	1.78	50	1.18	60
TR30_165C_24h	1.86	50	1.35	60
TR30_165C_36h	2.33	30	1.46	60
TR30_190C_1h	4.17	20	2.90	30
TR30_190C_2h	3.80	20	2.77	30
TR30_190C_4h	5.28	12	2.94	30
TR30_190C_11h	2.88	30	1.85	50
TR30_190C_24h	2.64	30	1.69	50
TR30_190C_36h	2.04	30	1.35	60
TR16_165C_1h	1.24	50	0.90	100
TR16_165C_2h	1.26	60	0.82	100
TR16_165C_4h	1.73	50	1.27	60
TR16_165C_11h	1.44	50	1.05	60
TR16_165C_24h	1.46	60	1.18	60
TR16_165C_36h	1.64	50	1.16	60
TR16_190C_1h	2.85	30	1.59	50
TR16_190C_2h	1.60	50	1.10	60
TR16_190C_4h	2.43	30	1.36	60
TR16_190C_11h	2.31	30	1.41	60
TR16_190C_24h	1.62	50	1.15	60
TR16_190C_36h	1.15	60	0.76	100

Table C.2: Car tyre–derived crumb rubber-modified binders

Sample	Viscosity at 165 °C (Pa·s)	Viscosity at 165 °C rpm	Viscosity at 175 °C (Pa·s)	Viscosity at 175 °C rpm
CT30_165C_1h	2.35	30	1.57	50
CT30_165C_2h	2.24	30	1.37	60
CT30_165C_4h	2.50	30	1.71	50
CT30_165C_11h	1.78	50	1.24	60
CT30_165C_24h	2.65	30	1.68	50
CT30_165C_36h	2.83	30	1.75	50
CT30_190C_1h	3.75	20	1.81	50
CT30_190C_2h	2.87	30	1.63	50
CT30_190C_4h	2.88	30	1.71	50
CT30_190C_11h	3.56	20	2.59	30
CT30_190C_24h	2.10	30	1.42	60
CT30_190C_36h	1.85	50	1.27	60
CT16_165C_1h	1.16	60	0.76	100
CT16_165C_2h	1.38	50	1.02	60
CT16_165C_4h	1.53	50	0.89	100
CT16_165C_11h	1.28	60	0.87	100
CT16_165C_24h	1.35	60	0.89	100
CT16_165C_36h	1.79	50	1.23	60
CT16_190C_1h	1.64	50	1.08	60
CT16_190C_2h	1.35	60	0.88	100
CT16_190C_4h	1.86	30	1.28	60
CT16_190C_11h	2.53	30	1.39	60
CT16_190C_24h	1.43	50	1.12	60
CT16_190C_36h	1.73	50	1.22	60

Table C.3: Conveyor belt–derived crumb rubber-modified binders

Sample	Viscosity at 165 °C (Pa·s)	Viscosity at 165 °C rpm	Viscosity at 175 °C (Pa·s)	Viscosity at 175 °C rpm
CB30_165C_1h	2.28	30	1.38	60
CB30_165C_2h	2.42	30	1.75	50
CB30_165C_4h	2.31	30	1.64	50
CB30_165C_11h	1.80	50	1.29	60
CB30_165C_24h	2.37	30	1.48	60
CB30_165C_36h	2.68	30	1.87	50
CB30_190C_1h	2.95	30	1.81	50
CB30_190C_2h	2.85	30	1.80	50
CB30_190C_4h	2.92	30	1.85	50
CB30_190C_11h	3.06	30	1.89	50
CB30_190C_24h	3.02	30	1.85	50
CB30_190C_36h	3.88	20	2.89	30
CB16_165C_1h	0.75	100	0.56	100
CB16_165C_2h	0.82	100	0.58	100
CB16_165C_4h	1.11	60	0.81	100
CB16_165C_11h	2.78	30	1.37	60
CB16_165C_24h	1.56	50	1.10	60
CB16_165C_36h	2.86	30	1.72	50
CB16_190C_1h	0.87	100	0.62	100
CB16_190C_2h	1.27	60	0.88	100
CB16_190C_4h	1.32	60	0.84	100
CB16_190C_11h	1.49	20	0.97	30
CB16_190C_24h	1.56	50	1.10	60
CB16_190C_36h	2.28	30	1.40	60

Table C.4: Mining tyre-derived crumb rubber-modified binders

Sample	Viscosity at 165 °C (Pa·s)	Viscosity at 165 °C rpm	Viscosity at 175 °C (Pa·s)	Viscosity at 175 °C rpm
MT30_165C_1h	2.78	10	1.83	12
MT30_165C_2h	4.67	20	3.71	20
MT30_165C_4h	17.58	5	7.69	12
MT30_165C_11h	12.35	6	8.05	10
MT30_165C_24h	13.88	6	9.02	10
MT30_165C_36h	0.74	30	0.49	60
MT30_190C_1h	18.26	5	13.20	6
MT30_190C_2h	14.47	6	9.25	10
MT30_190C_4h	13.71	6	8.44	10
MT30_190C_11h	11.62	6	8.89	10
MT30_190C_24h	2.95	30	2.40	30
MT30_190C_36h	1.27	60	0.87	100
MT16_165C_1h	0.95	60	0.74	100
MT16_165C_2h	2.11	30	1.28	60
MT16_165C_4h	8.53	10	6.11	12
MT16_165C_11h	5.57	12	3.91	20
MT16_165C_24h	13.43	6	8.65	10
MT16_165C_36h	1.19	60	0.73	100
MT16_190C_1h	7.32	12	4.31	20
MT16_190C_2h	14.89	6	3.82	20
MT16_190C_4h	14.28	5	6.07	12
MT16_190C_11h	12.19	6	6.16	12
MT16_190C_24h	3.12	30	2.22	30
MT16_190C_36h	1.46	60	1.06	60

C.2 Stress Ratio at 10 °C Stress-strain Plots

Stress-strain curves as derived from the stress ratio test are provided in Figure C.1 to Figure C.126.

Figure C.1: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 truck tyre-derived crumb rubber at 165 °C

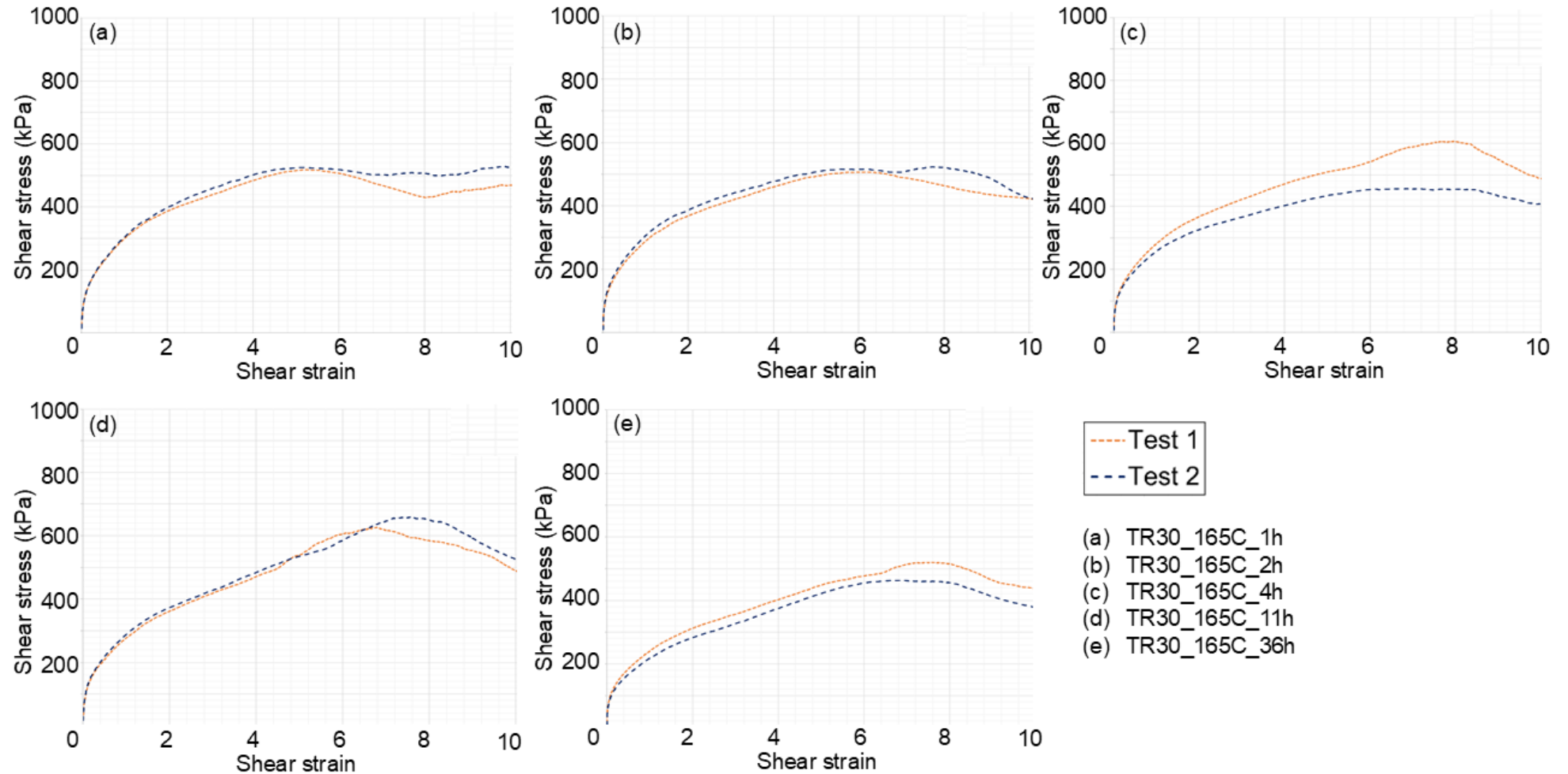


Figure C.2: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 truck tyre–derived crumb rubber at 190 °C

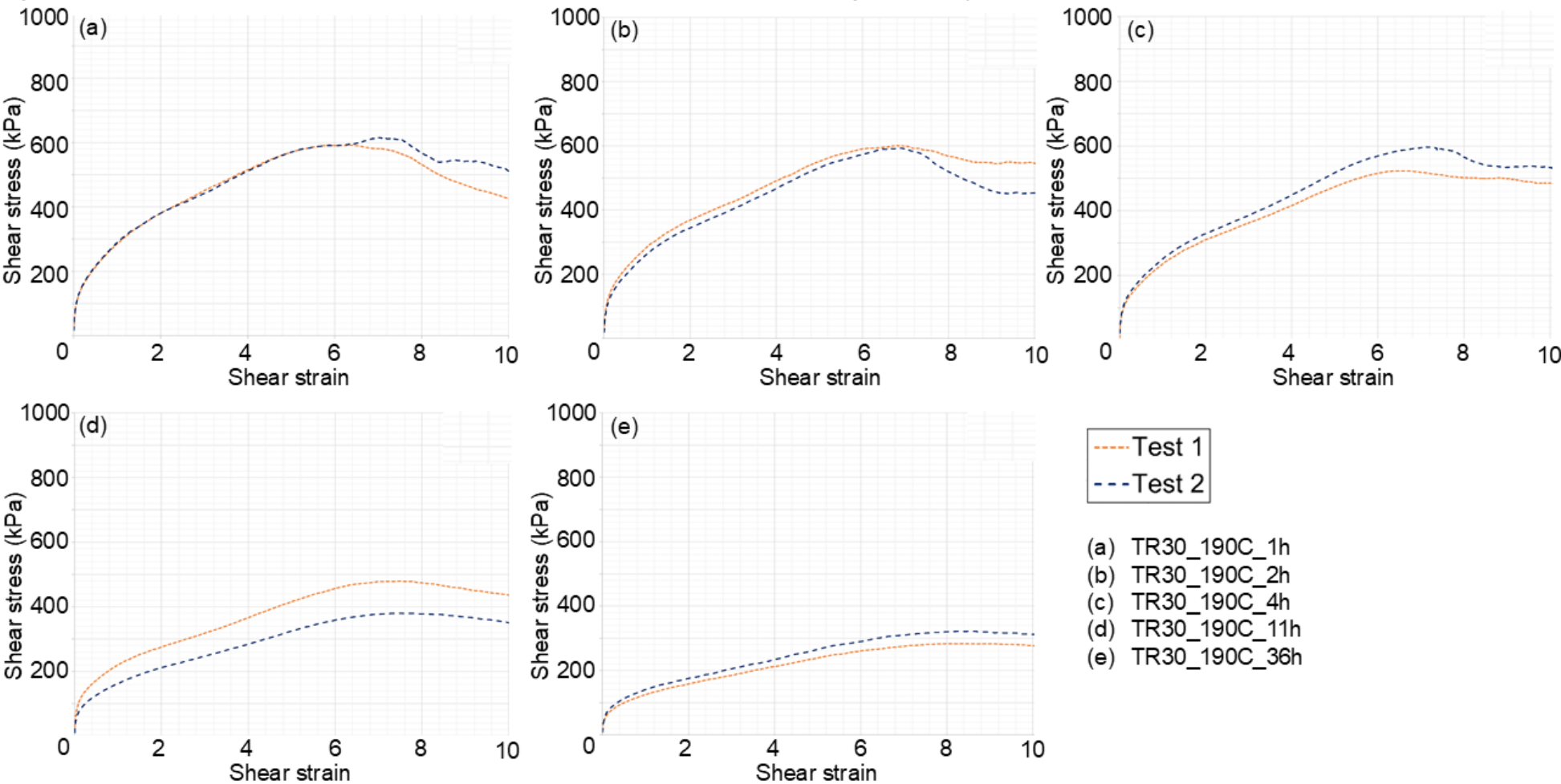


Figure C.3: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 truck tyre–derived crumb rubber at 165 °C

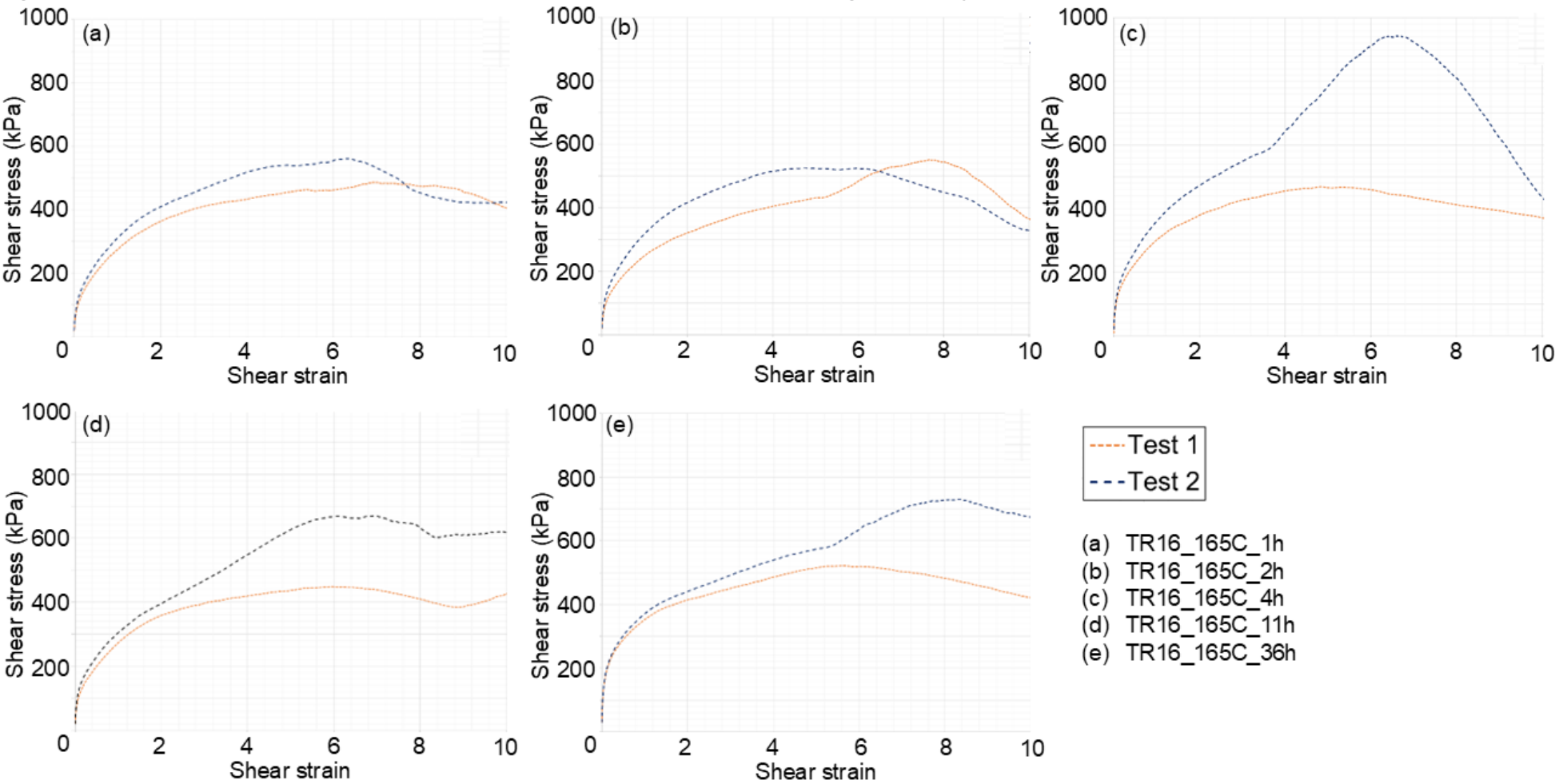


Figure C.4: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 truck tyre-derived crumb rubber at 190 °C

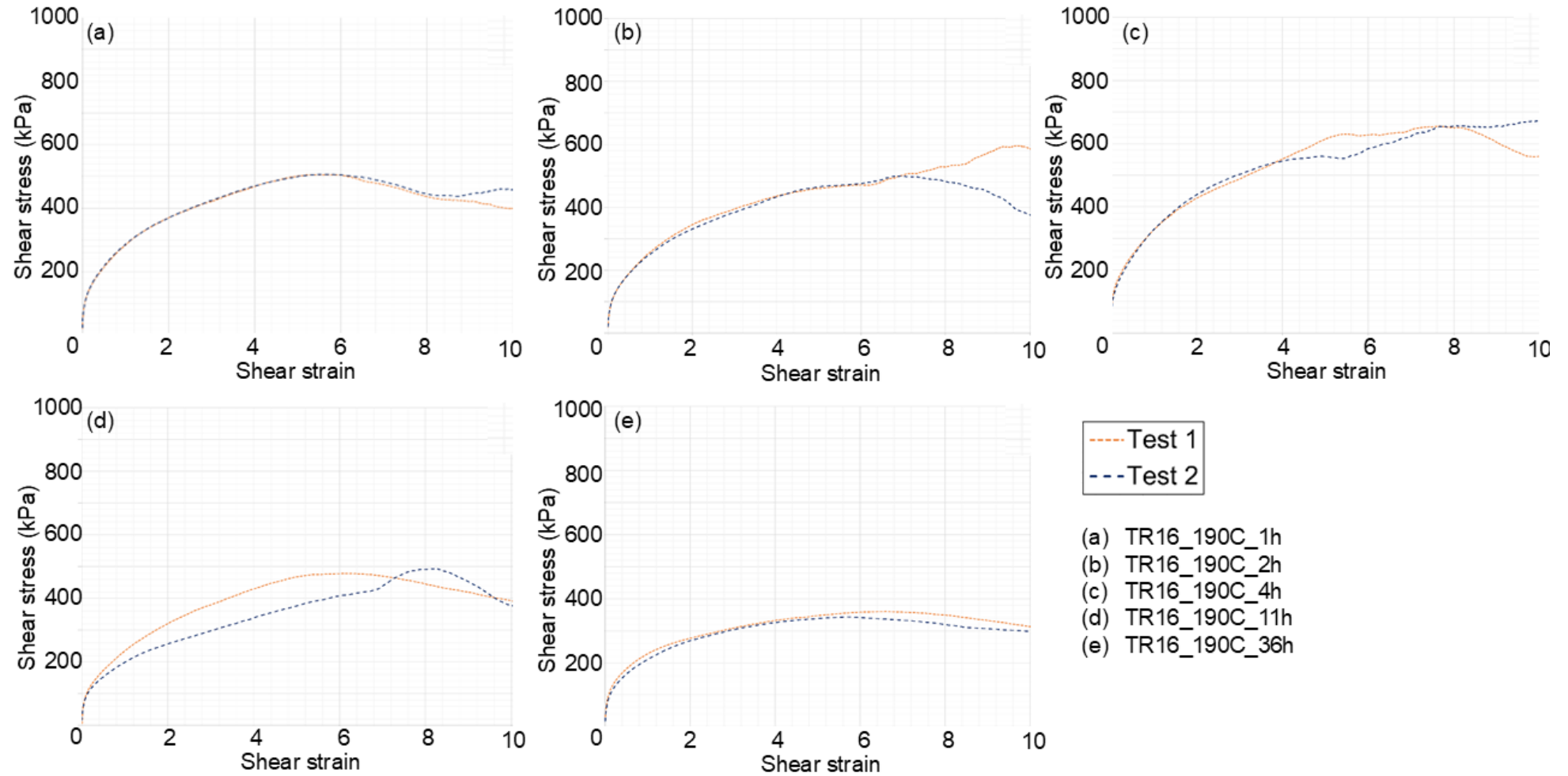


Figure C.5: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 car tyre-derived crumb rubber at 165 °C

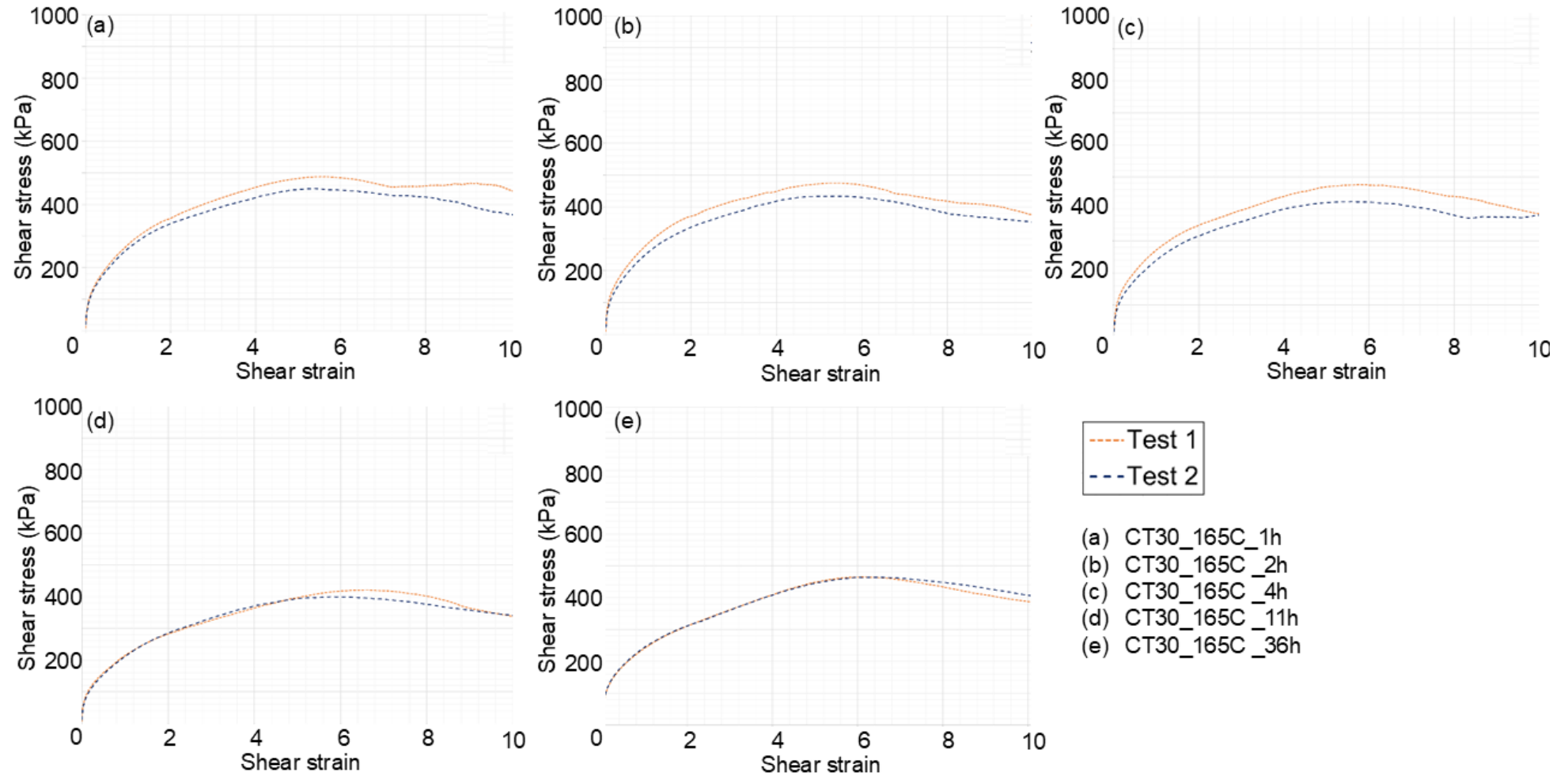


Figure C.6: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 car tyre-derived crumb rubber at 190 °C

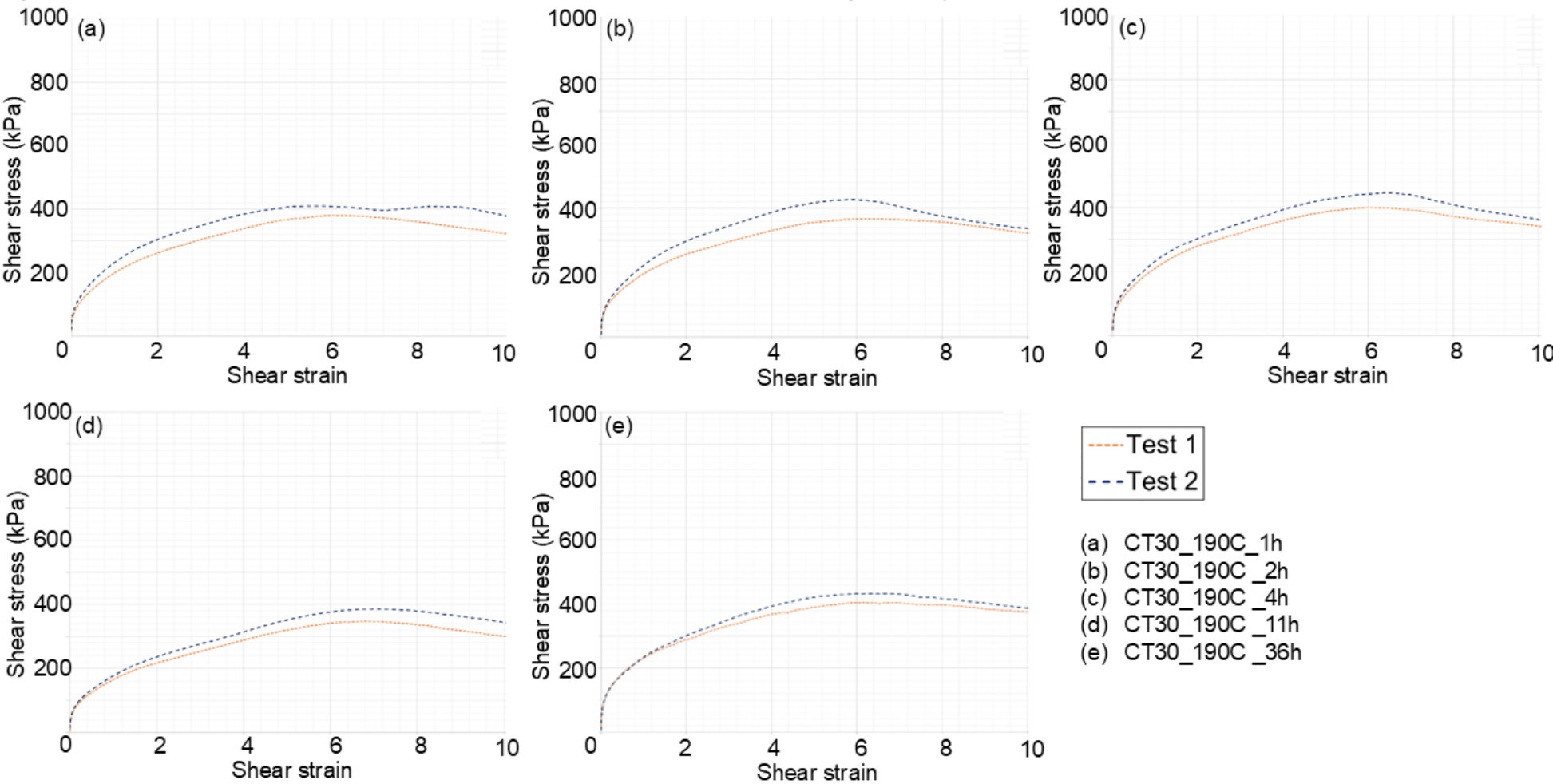


Figure C.7: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 car tyre-derived crumb rubber at 165 °C

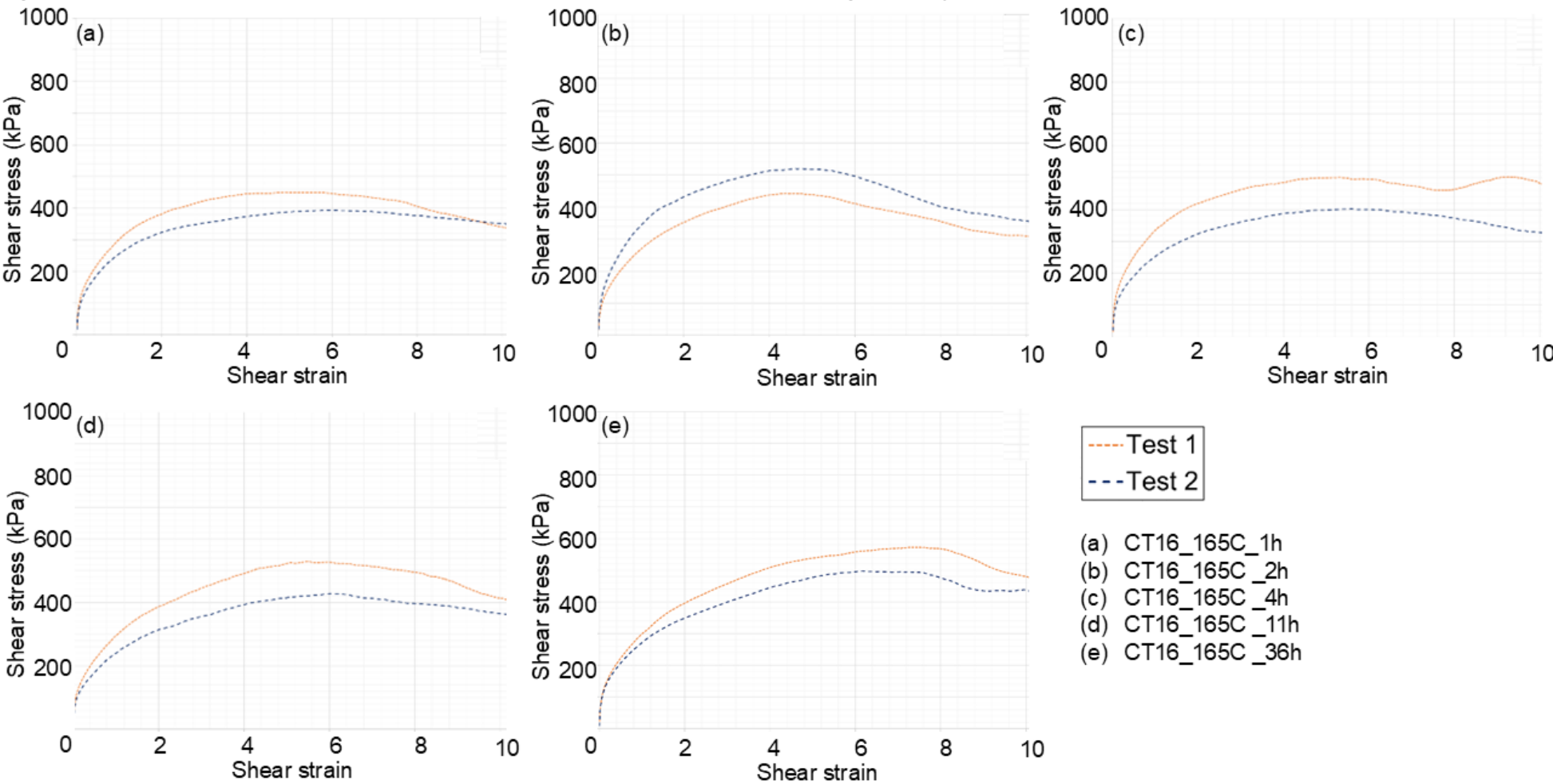


Figure C.8: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 car tyre-derived crumb rubber at 190 °C

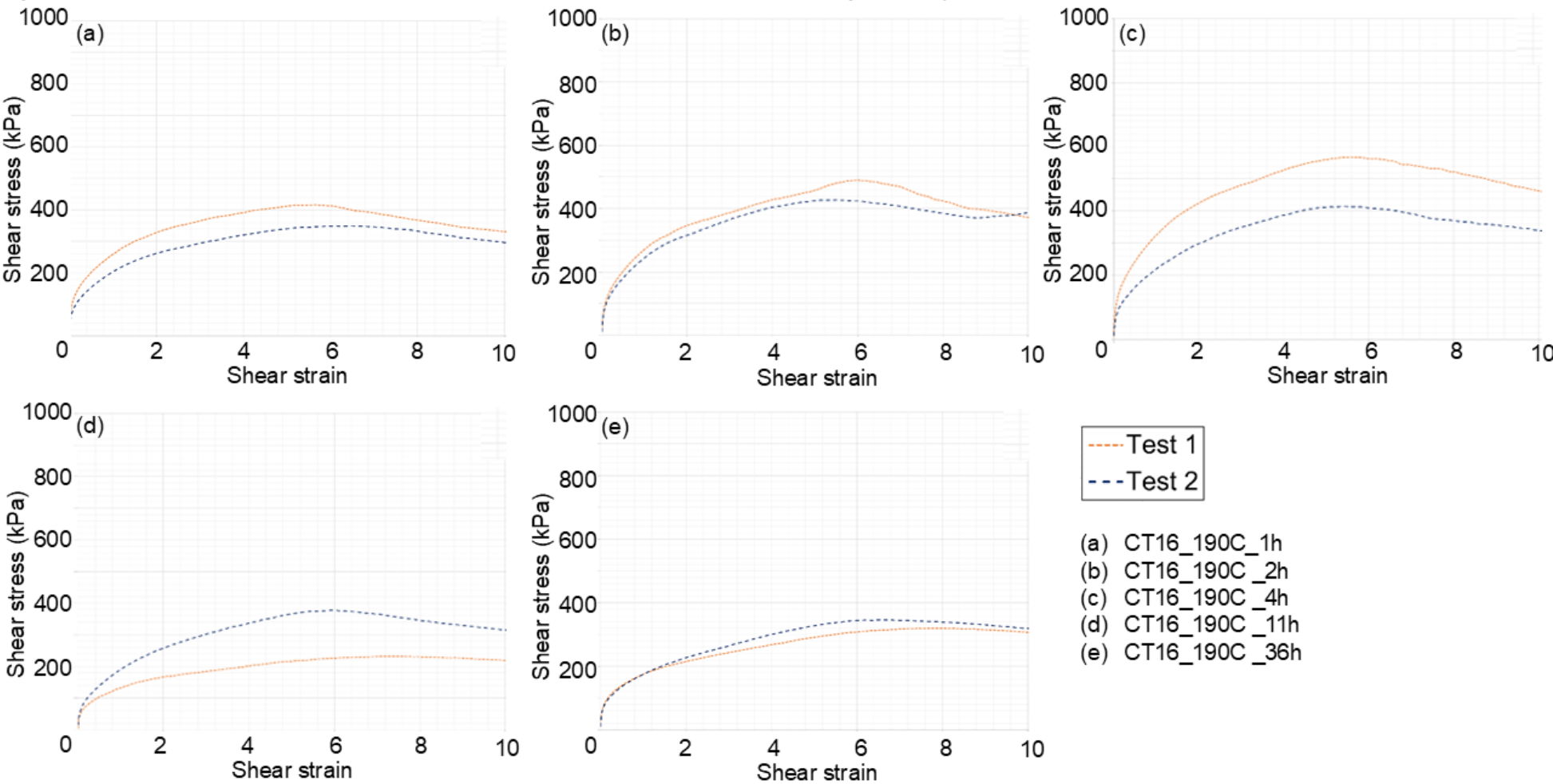


Figure C.9: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 conveyor belt–derived crumb rubber at 165 °C

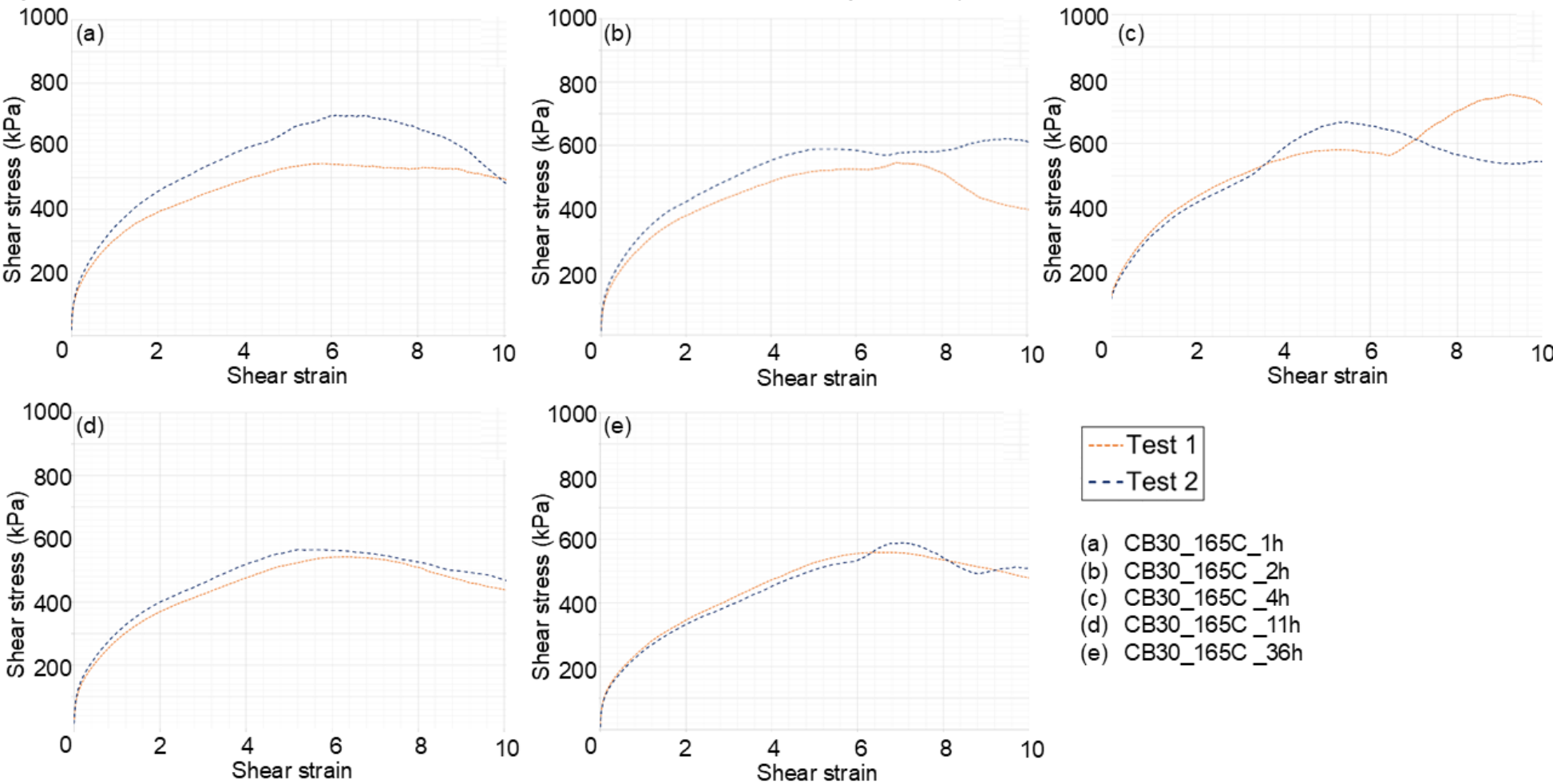


Figure C.10: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 conveyor belt–derived crumb rubber at 190 °C

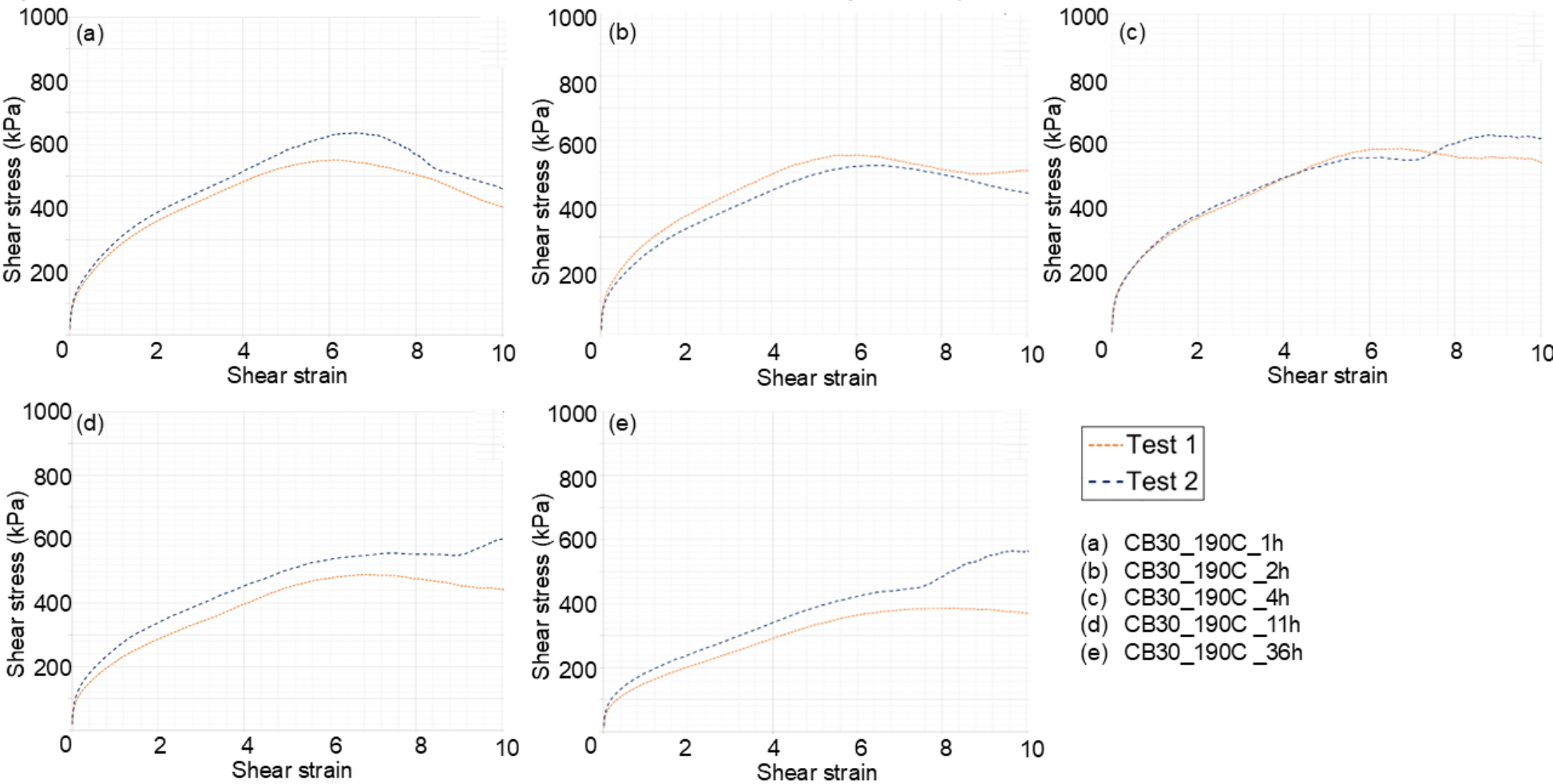


Figure C.11: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 conveyor belt–derived crumb rubber at 165 °C

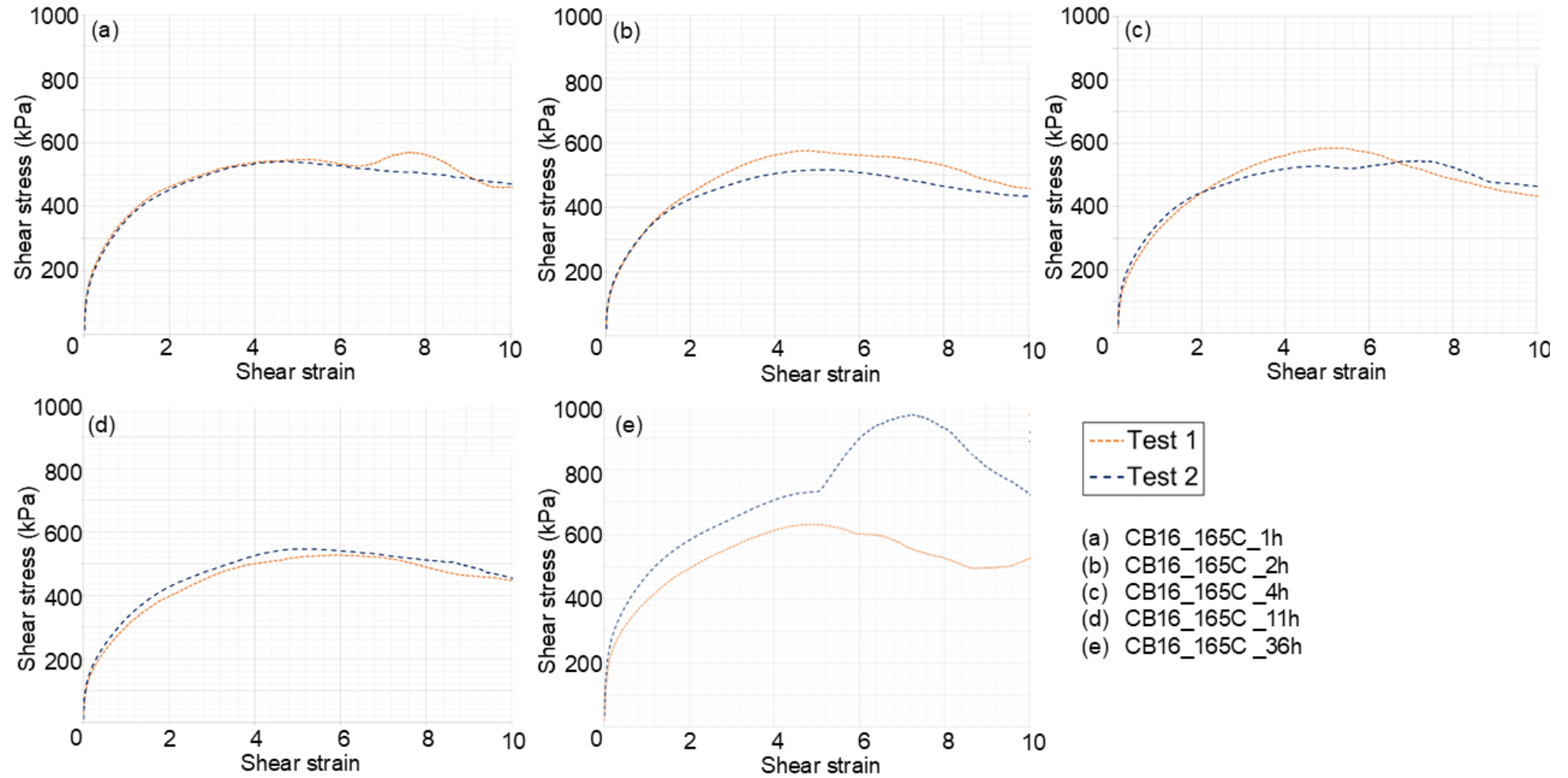


Figure C.12: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 conveyor belt–derived crumb rubber at 190 °C

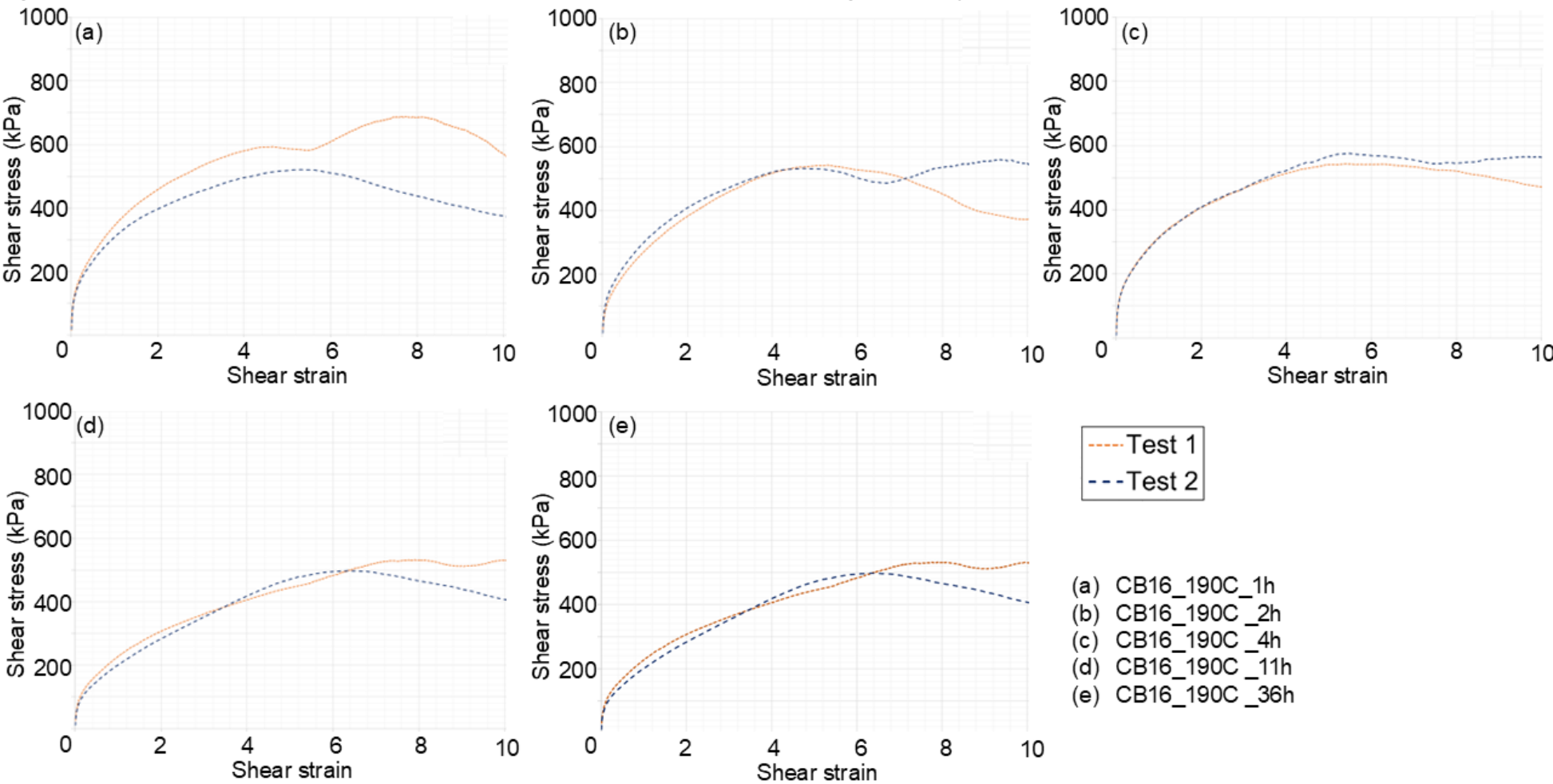


Figure C.13: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 mining tyre-derived crumb rubber at 165 °C

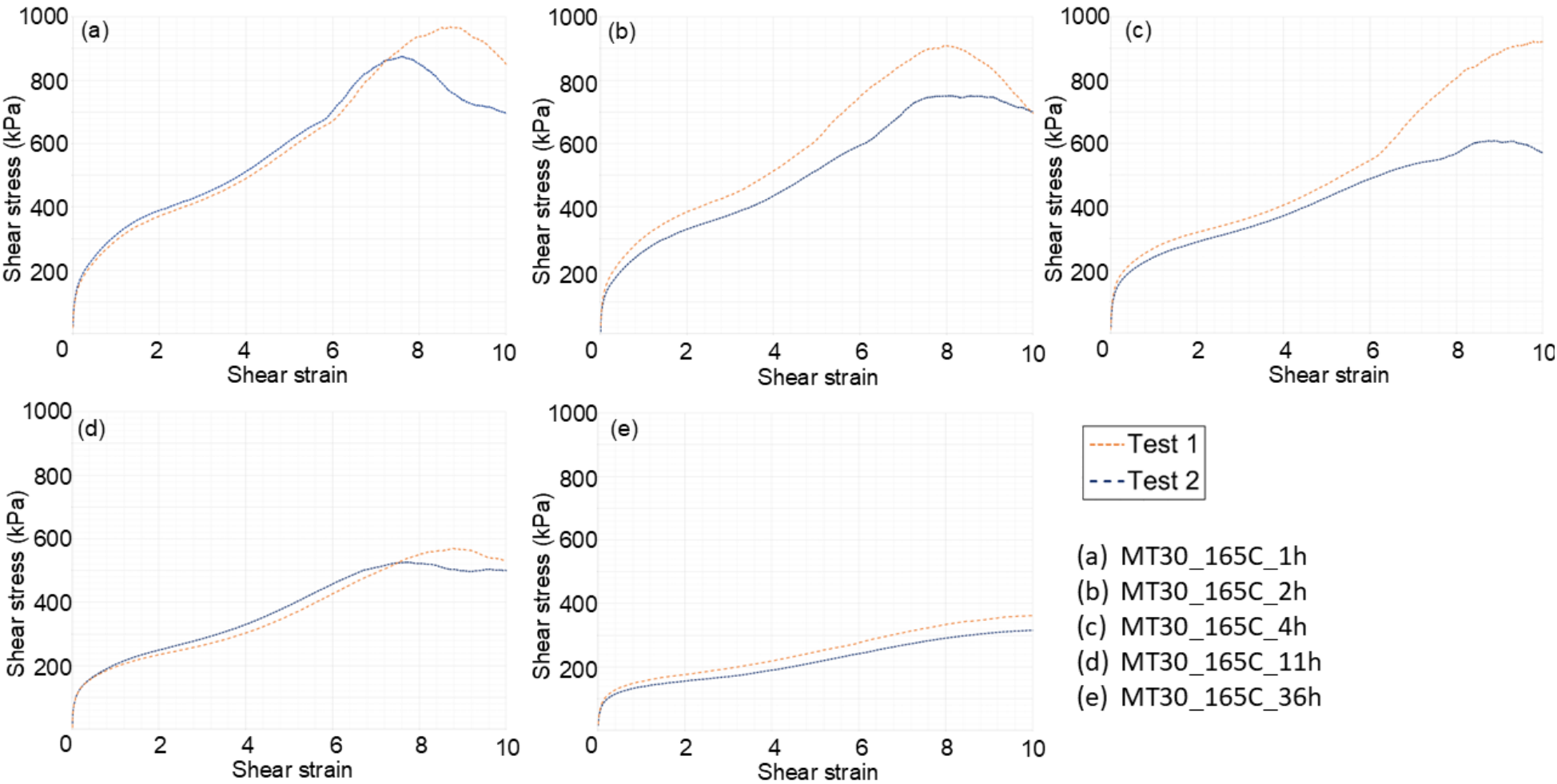


Figure C.14: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S30 mining tyre-derived crumb rubber at 190 °C

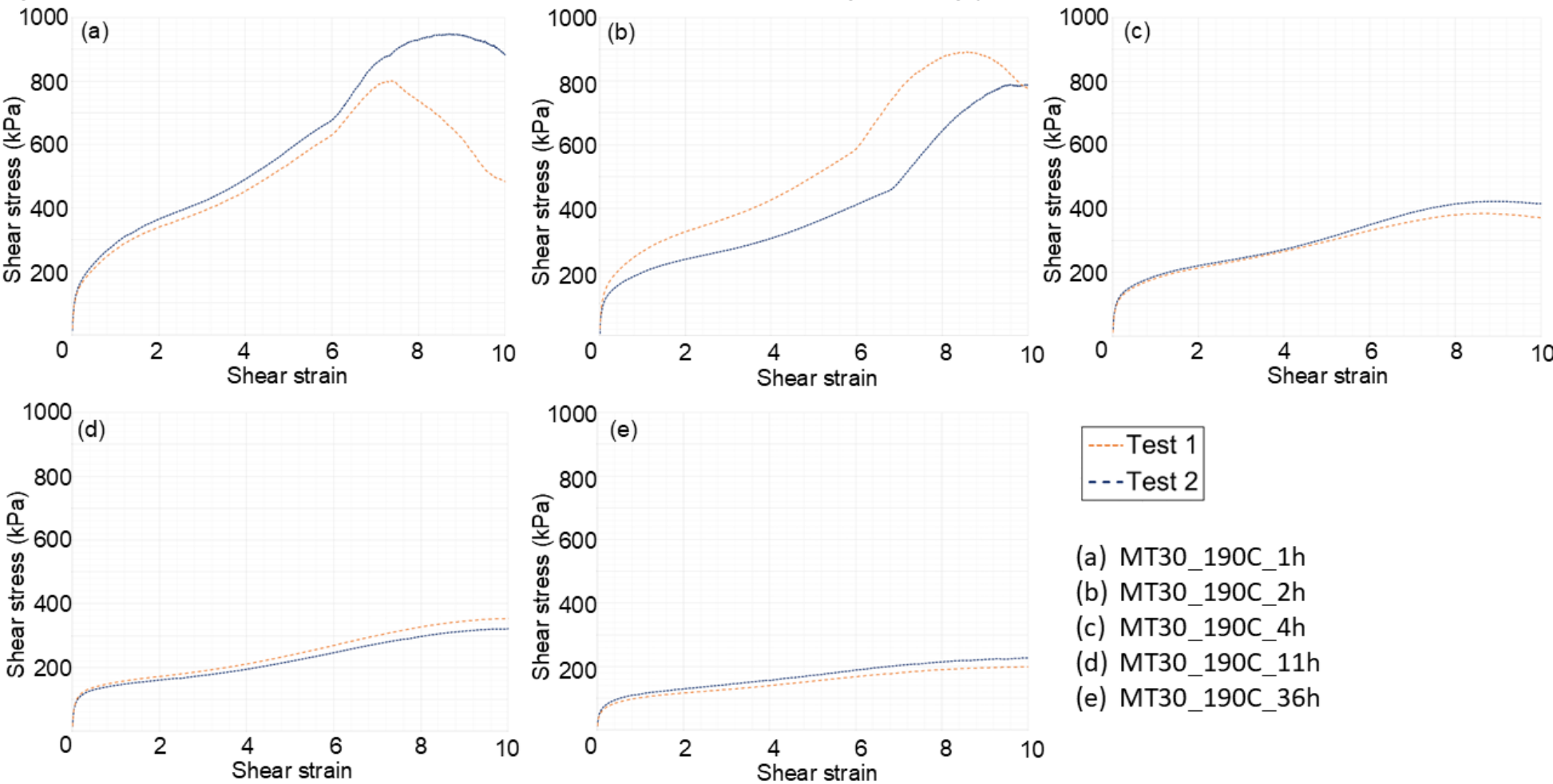


Figure C.15: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 mining tyre-derived crumb rubber at 165 °C

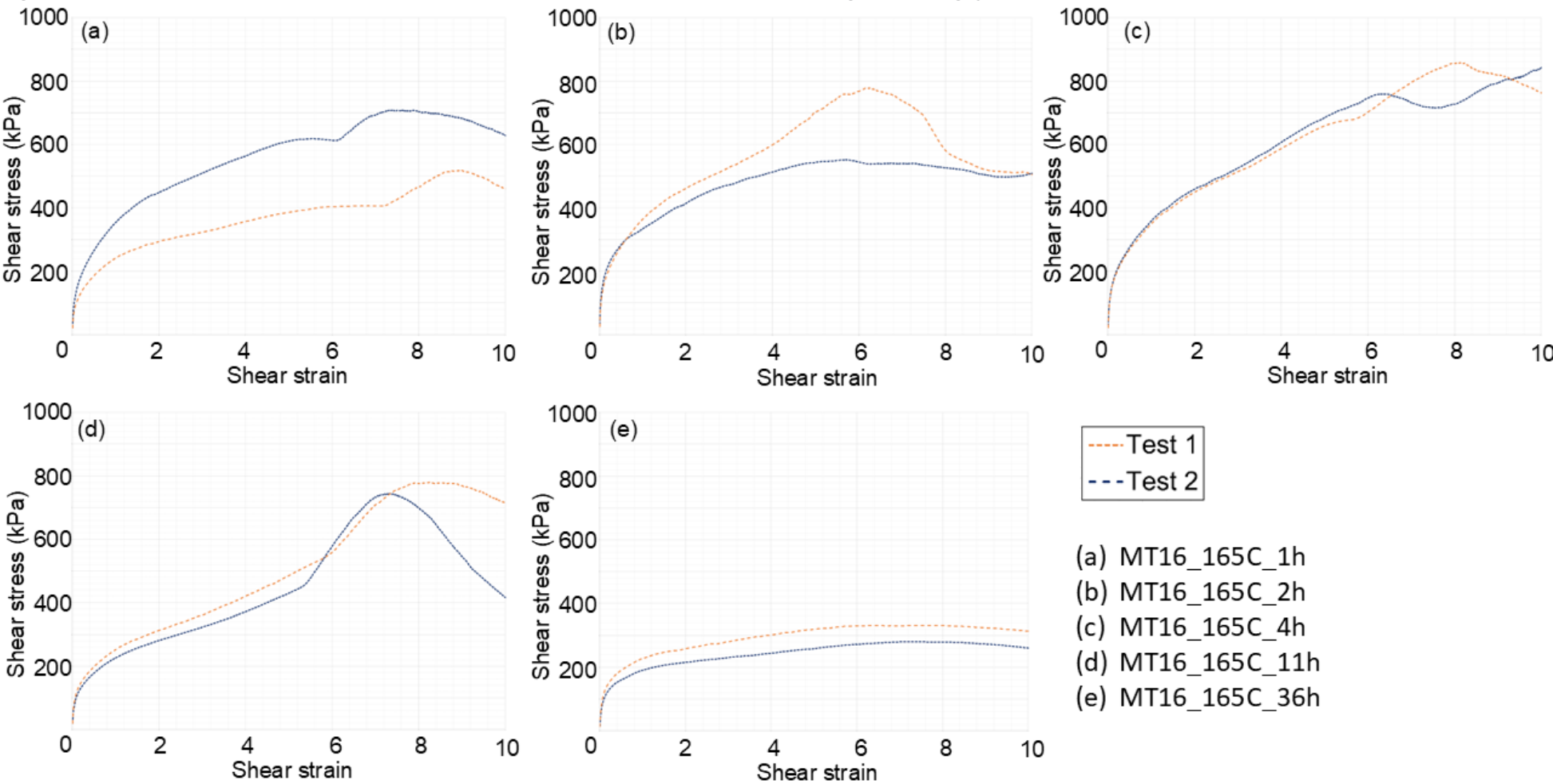
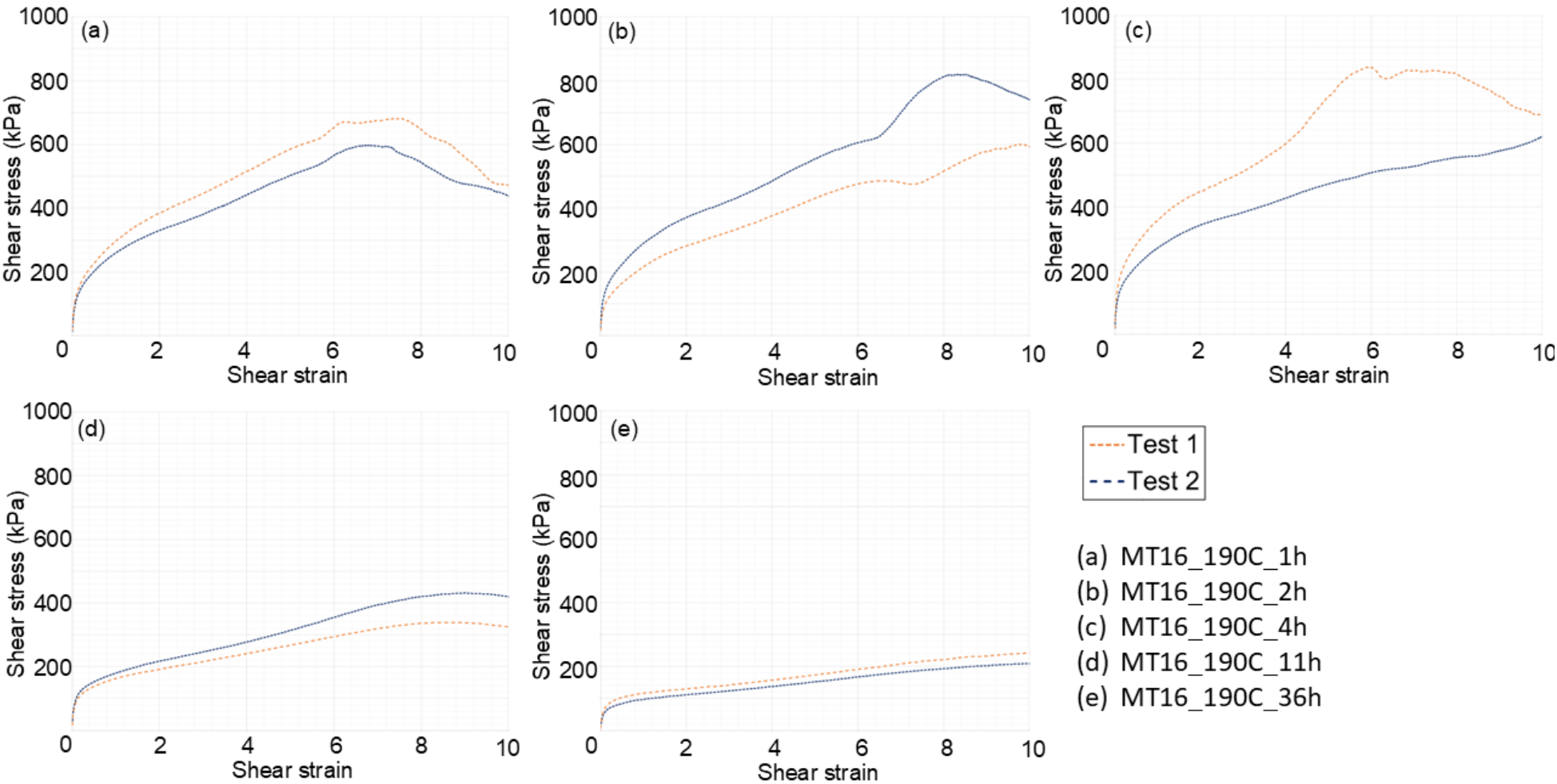


Figure C.16: Stress-strain plots for stress ratio at 10 °C tests for samples manufactured using S16 mining tyre-derived crumb rubber at 190 °C

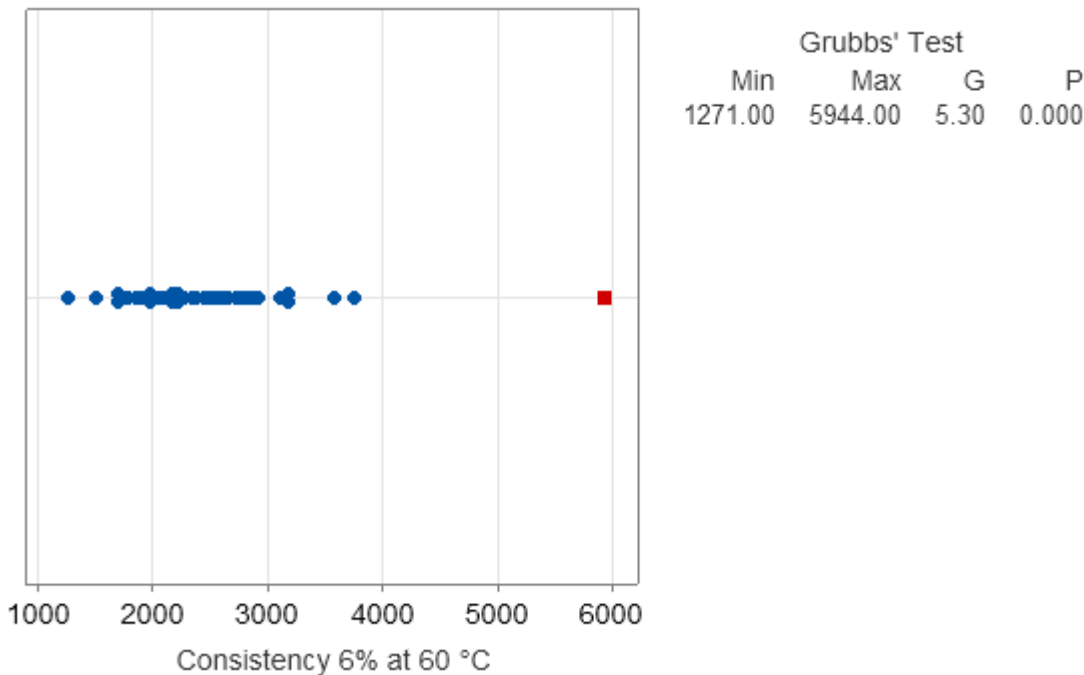


C.3 Outlier Test for Consistency 6% at 60 °C

During the analysis of the results for CB16_165C_36h, it was noted that consistency 6% at 60 °C was notably greater than all other results. Therefore, a Grubbs' test was conducted to confirm whether the reported value was a statistical outlier. The results are presented in Figure C.17.

The results revealed that CB16_165C_36h was indeed an outlier with a p-value of 0.000. To confirm that the result was not erroneous, the test was repeated for this sample. The second result confirmed the accuracy of the first test with a report value of 6,254 Pa.s. Provided that the test equipment was appropriately calibrated and the laboratory operator was qualified to undertake the relevant test, it was decided that the results were to be reported as true.

Figure C.17: Grubbs' test results for consistency 6% at 60 °C



C.4 Outlier Test for Viscosity at 165 and 175 °C

During the analysis of the Brookfield viscosity results, it was noted that those for CB16_190C_11h for both 165 and 175 °C were notably greater than what was measured for other samples. Therefore, an outlier test was performed. The results are presented in Figure C.18 and Figure C.19.

The test revealed that the results were indeed outliers with p-values of 0.001 and 0.000 for viscosity at 165 and 175 °C, respectively.

Figure C.18: Grubbs' test results for viscosity at 165 °C

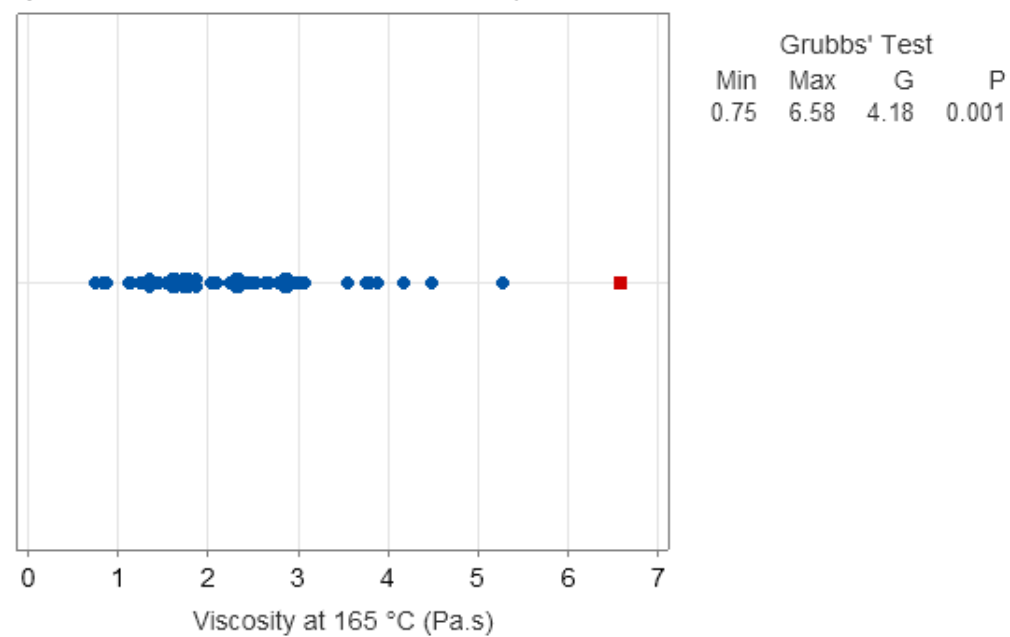
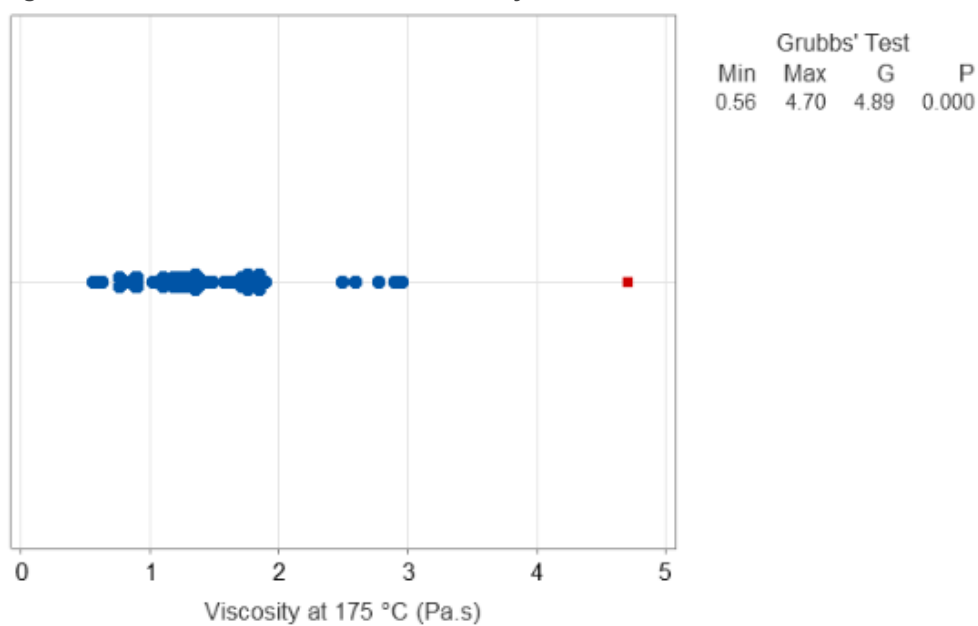


Figure C.19: Grubbs' test results for viscosity at 175 °C



Therefore, the tests were repeated a second time. The results of the second repetition notably varied from the first tests run, and therefore, to confirm which tests were most repeatable, the tests were repeated a third time. The results are presented in Table C.5.

Table C.5: Viscosity at 165 and 175 °C results for each repeated test for sample CB16_190C_11h

	1st test	2nd test	3rd test
Viscosity at 165 °C (Pa.s)	6.58	1.47	1.52
Viscosity at 175 °C (Pa.s)	4.70	0.98	0.97

The results for the second and third tests appeared to be closer with each other as well as the rest of the samples. Therefore, the results reported and analysed in Section 4.4.1 are the average of the second and third tests performed for sample CB16_190C_11h.

C.5 Repeat Testing for Viscosity at 165 and 175 °C for Mining Tyre-derived Crumb Rubber-modified Binders

Table C.6 presents the viscosity at 165 and 175 °C test results for the first test and repeat tests for selected MT-derived CRMBs.

Table C.6: Repeat testing results of viscosity at 165 and 175 °C for mining tyre-derived crumb rubber-modified binders

Sample	Viscosity (Pa·s) – Test 1	Viscosity rpm – Test 1	Viscosity (Pa·s) – Test 2	Viscosity rpm – Test 2
	at 165 °C			
MT16_165C_4h	8.53	10	7.98	10
MT16_165C_11h	5.57	12	12.89	6
MT16_190C_1h	7.32	12	13.05	6
MT16_190C_2h	14.89	6	14.41	6
MT16_190C_4h	14.28	5	8.36	10
MT16_190C_11h	12.19	6	12.03	6
MT30_165C_2h	4.67	20	7.28	12
MT30_165C_4h	17.58	5	17.29	5
MT30_165C_11h	12.35	6	13.62	6
MT30_165C_24h	13.88	6	15.39	6
MT30_190C_1h	18.26	5	36.39	2.5
MT30_190C_2h	14.47	6	30.17	3
MT30_190C_4h	13.71	6	14.82	6
MT30_190C_11h	11.62	6	13.86	6
Sample	at 175 °C			
MT16_165C_4h	6.11	12	4.56	20
MT16_165C_11h	3.91	20	7.43	12
MT16_190C_1h	4.31	20	8.55	10
MT16_190C_2h	3.82	20	8.78	10
MT16_190C_4h	6.07	12	5.89	12
MT16_190C_11h	6.16	12	5.92	12
MT30_165C_2h	3.71	20	6.32	12
MT30_165C_4h	7.69	12	8.72	10
MT30_165C_11h	8.05	10	9.10	10
MT30_165C_24h	9.02	10	9.26	10
MT30_190C_1h	13.20	6	23.41	4
MT30_190C_2h	9.25	10	17.40	5
MT30_190C_4h	8.44	10	8.43	10
MT30_190C_11h	8.89	10	9.37	10

Table C.6 shows a dependency between the report values for viscosity at both 165 and 175 °C and the speed at which the spindle was rotating at the time of the measurement. Accepting that the relationship between viscosity and shear rate for shear thinning non-Newtonian fluids follows a power law, the results of Table C.6 whose viscosity measurements were undertaken at different spindle speeds were plotted and fitted. Using the fit equations for each, the results were extrapolated to the spindle speeds at which measurements for their TR-, CT- and CB-derived CRMB counterparts were taken according to Table C.1, Table C.2 and Table C.3, respectively. These calculated viscosities at 165 and 175 °C are shown in Table C.7. It is highlighted that the 2 data points acquired in this research are not sufficient to suggest that the equations generated by said fits are representative of these CRMBs. They are merely used here to demonstrate an approximate value for the viscosity at 165 and 175 °C results accepting known rheological

theoretical models. It is also noted that these models better reflect homogeneous fluids, whereas in the case of CRMBs, such as those assessed in this research, the material is a composite of solid crumb rubber particles (undigested portion) and a modified bituminous liquid.

Table C.7: Calculated viscosity at 165 and 175 °C for mining tyre-derived crumb rubber-modified binders for different spindle speeds

Sample	MT measured		TR		MT calculated	CT		MT calculated	CB		MT calculated
	Pa.s	rpm	Pa.s	rpm	Pa.s	Pa.s	rpm	Pa.s	Pa.s	rpm	Pa.s
Sample	at 165 °C										
-16_165C_11h	5.57	12	1.44	50	0.99	1.28	60	0.79	2.78	30	1.83
	12.89	6									
-16_190C_1h	7.32	12	2.85	30	3.41	1.64	50	2.23	0.87	100	1.25
	13.05	6									
-16_190C_4h	14.28	5	2.43	30	3.58	1.86	30	3.58	1.32	30	2.10
	8.36	10									
-30_165C_2h	4.27	20	1.68	50	1.64	2.24	30	2.78	2.42	30	2.80
	7.28	12									
-30_190C_1h	18.26	5	4.17	20	4.60	3.75	20	4.60	2.95	30	3.07
	36.39	2.5									
-30_190C_2h	14.47	6	3.80	20	4.04	2.87	30	2.63	2.85	30	2.63
	30.17	3									
	at 175 °C										
-16_165C_4h	6.11	12	1.27	60	2.43	0.89	100	1.81	0.81	100	1.81
	4.56	20									
-16_165C_11h	3.91	20	1.05	60	0.98	0.87	100	0.52	1.37	60	0.98
	7.43	12									
-16_190C_1h	4.31	20	1.59	50	1.74	1.08	60	1.46	0.62	100	0.88
	8.55	10									
-16_190C_2h	3.82	20	1.10	60	1.02	0.88	100	0.55	0.88	100	0.55
	8.78	10									
-30_165C_2h	3.71	20	1.35	60	1.18	1.37	60	1.18	1.75	50	1.43
	6.32	12									
-30_165C_4h	7.69	12	1.75	50	2.88	1.71	50	2.88	1.64	50	2.88
	8.72	10									
-30_190C_1h	13.20	6	2.90	30	1.36	1.81	50	0.66	1.81	50	0.66
	23.41	4									
-30_190C_2h	9.25	10	2.77	30	3.39	1.63	50	2.13	1.80	50	2.13
	17.40	5									

From Table C.7, it is reasonable to anticipate that had the results for viscosity at 165 and 175 °C for MT-derived CRMBs been obtained at the same spindle speeds as their TR-, CT- and CB-derived counterparts, they would have been closer than the actuals discussed in Section 4.4.1. The ANOVA observations, in turn, would have also potentially been different. It is highlighted once again that it is not suggested that the calculated values in Table C.7 would be accurate, they are simply a sensible approximation.

C.6 Additional Results for Conveyor Belt– and Mining Tyre–derived Crumb Rubber-modified Binders

C.6.1 Stiffness at 25 °C

Stiffness at 25 °C was quantified for CB- and MT-derived CRMBs following AGPT-T121:2014. In this method, the stiffness of the binder is assessed using the ARRB elastometer, which measures the resistance of the binder to elastic deformation at peak stress. The results are presented in Table C.8 and Table C.9.

MRWA Specification 511:2025 poses a maximum requirement of 180 kPa for binders blended for 1 hour. For greater digestion times, no requirement is specified. In all cases, the stiffness at 25 °C for all CB- and MT-derived CRMBs was found to be below 180 kPa.

Table C.8: Results for stiffness at 25 °C for conveyor belt–derived crumb rubber-modified binders; results in kPa

Sample	Digestion time	
	1h	36h
CB30_165C_	94	80
CB30_190C_	88	52
CB16_165C_	112	159
CB16_190C_	106	65

Table C.8 shows the stiffness at 25 °C decreasing for all binders when the blending time increased from 1 to 36 hours, except for CB16_165C, which increased by 47 kPa. An increase in stiffness is probably observed due to the swelling of the crumb rubber particles in the binder. A decrease in stiffness, on the other hand, is an indication of devulcanisation and depolymerisation of the rubber particles (Daly et al. 2019).

According to the literature presented in Sections 2.2.3 and 2.2.6, binders blended at higher temperatures using crumb rubber particles of smaller sizes are expected to progress through the digestion stages faster than larger particles digested at lower temperatures. The observations made in Table C.8, therefore, agree with these expectations, suggesting the smaller particles have potentially progressed to decomposition after 36 hours of blending irrespective of temperature, whereas the larger particles decomposed only when blended for an extended time at elevated temperatures.

Table C.9 presents the stiffness at 25 °C results for the MT-derived CRMBs. In all cases, the stiffness notably decreased with an increase in blending time, with that decrease being more pronounced for binders blended at 190 °C. These findings support that the crumb rubber particles have undergone some depolymerisation and/or devulcanisation under these extended blending conditions.

Table C.9: Results for stiffness at 25 °C for mining tyre–derived crumb rubber-modified binders; results in kPa

Sample	Digestion time	
	1h	36h
MT30_165C_	98	43
MT30_190C_	85	27
MT16_165C_	99	85
MT16_190C_	98	34

C.6.2 Penetration at 4 °C

Penetration at 4 °C was measured following AS 2341.12:2020 *Methods of Testing Bitumen and Related Roadmaking Products, Method 12: Determination of Penetration*. The penetration at 4 °C results for CB-derived CRMBs are presented in Table C.10. All the binders pass the minimum requirement of 15 p.u. according to MRWA Specification 511:2025. All results are comparable and no specific trends can be identified.

Table C.10: Results for penetration at 4 °C for conveyor belt–derived crumb rubber-modified binders; results in p.u.

Sample	Digestion time					
	1h	2h	4h	11h	24h	36h
CB30_165C_	20	17	19	19	–	19
CB30_190C_	19	18	20	18	–	18
CB16_165C_	17	18	18	18	–	18
CB16_190C_	18	18	17	19	–	17

The penetration at 4 °C results for MT-derived CRMBs are presented in Table C.11. All the results are above the minimum requirement of 15 p.u according to MRWA Specification 511:2025. The results present little variation and generally slightly increase with blending time.

Table C.11: Results for penetration at 4 °C for mining tyre–derived crumb rubber-modified binders; results in p.u.

Sample	Digestion time					
	1h	2h	4h	11h	24h	36h
MT30_165C_	22	22	21	22	–	24
MT30_190C_	17	20	21	22	–	24
MT16_165C_	20	21	21	21	–	24
MT16_190C_	19	22	22	22		22

C.6.3 Elastic Recovery

The elastic recovery results for CB-derived CRMBs are presented in Table C.12 and Table C.13 for testing at 25 and 60 °C, respectively. Elastic recovery at 25 °C was found to increase with an increase in temperature when blending was undertaken for 1 hour irrespective of the size of crumb rubber. It was also found to increase with an increase in blending time from 1 to 36 hours for all samples except CB30_190C. This could be attributed to the relatively accelerated progression through the digestion stages for this sample, as it was manufactured at elevated temperatures using crumb rubber with greater surface area.

Table C.12: Results for elastic recovery at 25 °C for conveyor belt–derived crumb rubber-modified binders; results in %

Sample	Digestion time	
	1h	36h
CB30_165C_	73	79
CB30_190C_	79	77
CB16_165C_	70	78
CB16_190C_	75	78

The elastic recovery at 60 °C was found to increase with an increase in blending temperature for all samples except those manufactured with S16 CB-derived crumb rubber when blended for 36 hours. A consistent trend with blending time could not be observed.

Table C.13: Results for elastic recovery at 60 °C for conveyor belt–derived crumb rubber-modified binders; results in %

Sample	Digestion time					
	1h	2h	4h	11h	24h	36h
CB30_165C_	23	22	22	23	–	24
CB30_190C_	28	27	26	30	–	29
CB16_165C_	16	17	18	19	–	31
CB16_190C_	20	24	22	27	–	24

The elastic recovery of MT-derived CRMBs is presented in Table C.14 for samples tested at 25 °C and in Table C.15 for samples tested at 60 °C. The elastic recovery at 25 °C was found to decrease for all MT-derived CRMBs as the blending time increased from 1 to 36 hours.

Table C.14: Results for elastic recovery at 25 °C for mining tyre–derived crumb rubber-modified binders; results in %

Sample	Digestion time	
	1h	36h
MT30_165C_	80	68
MT30_190C_	82	58
MT16_165C_	73	64
MT16_190C_	82	63

The elastic recovery at 60 °C of S30 MT-derived CRMBs was found to increase for blending up to 4 hours and decrease beyond that, whereas that of S16 MT-derived CRMBs was found to increase for blending up to 11 hours and decrease after that. The greatest elastic recovery for both crumb rubber gradations was achieved by binders blended at 190 °C.

Table C.15: Results for elastic recovery at 60 °C for mining tyre–derived crumb rubber-modified binders; results in %

Sample	Digestion time					
	1h	2h	4h	11h	24h	36h
MT30_165C_	36	32	61	49	–	53
MT30_190C_	48	53	65	64	–	33
MT16_165C_	24	24	33	39	–	34
MT16_190C_	39	39	54	65	–	34

Appendix D Supplementary Results on Analytical Assessment

D.1 Thermogravimetric Analysis

Table D.1 presents the distribution of constituents as measured through TGA.

Table D.1: Composition of undigested, undigested and Soxhlet extracted, and digested and Soxhlet extracted crumb rubbers; average of 4 specimens per sample with standard deviation

Sample	Moisture and light oils (%)	Natural rubber (%)	Synthetic rubber and other polymers (%)	Carbon black and fillers (%)
TR30_165C_1h	2.19 ± 0.27	38.36 ± 0.90	15.57 ± 0.65	43.88 ± 1.04
TR30_165C_11h	2.63 ± 0.73	31.55 ± 4.76	17.48 ± 1.89	48.33 ± 2.28
TR30_165C_36h	1.70 ± 0.35	33.67 ± 1.26	16.44 ± 0.57	48.19 ± 0.39
TR30_190C_1h	1.85 ± 0.18	37.14 ± 1.18	17.44 ± 0.90	43.58 ± 0.82
TR30_190C_11h	1.59 ± 0.14	31.03 ± 0.72	21.32 ± 0.66	46.07 ± 0.70
TR30_190C_36h	1.52 ± 0.28	28.92 ± 2.15	16.96 ± 1.50	52.60 ± 1.89
TR16_165C_1h	1.98 ± 0.24	37.28 ± 5.55	17.35 ± 3.53	43.39 ± 2.26
TR16_165C_11h	1.79 ± 0.33	35.45 ± 3.77	17.92 ± 3.17	44.84 ± 1.25
TR16_165C_36h	1.70 ± 0.25	28.56 ± 3.56	18.70 ± 3.94	18.70 ± 3.94
TR16_190C_1h	1.66 ± 0.11	33.13 ± 1.35	19.25 ± 0.48	45.96 ± 1.66
TR16_190C_11h	1.70 ± 0.29	33.61 ± 0.92	17.12 ± 1.18	47.57 ± 1.51
TR16_190C_36h	1.61 ± 0.17	23.57 ± 2.41	18.67 ± 3.78	56.15 ± 2.63
CT30_165C_1h	1.97 ± 0.28	29.32 ± 0.84	21.65 ± 2.26	47.06 ± 1.79
CT30_165C_11h	1.76 ± 0.23	27.49 ± 2.48	25.02 ± 1.99	45.74 ± 2.01
CT30_165C_36h	1.56 ± 0.37	21.43 ± 1.60	25.89 ± 2.47	51.12 ± 0.81
CT30_190C_1h	1.93 ± 0.29	26.07 ± 1.39	24.01 ± 0.52	47.99 ± 1.26
CT30_190C_11h	1.42 ± 0.26	27.09 ± 0.81	24.50 ± 0.51	47.00 ± 0.68
CT30_190C_36h	1.29 ± 0.23	14.92 ± 1.55	29.04 ± 1.51	54.74 ± 2.04
CT16_165C_1h	1.75 ± 0.23	29.66 ± 6.42	23.31 ± 4.54	45.28 ± 2.03
CT16_165C_11h	2.24 ± 0.18	31.09 ± 1.76	22.00 ± 2.95	44.67 ± 2.23
CT16_165C_36h	1.52 ± 0.35	27.80 ± 1.47	23.62 ± 0.59	47.06 ± 1.50
CT16_190C_1h	2.04 ± 0.59	27.24 ± 3.25	22.23 ± 3.09	48.49 ± 1.91
CT16_190C_11h	1.62 ± 0.46	29.43 ± 1.14	18.51 ± 1.61	50.44 ± 2.21
CT16_190C_36h	1.43 ± 0.22	22.90 ± 3.82	24.51 ± 4.75	51.15 ± 2.03
CB30_165C_1h	1.76 ± 0.12	24.41 ± 1.67	30.68 ± 0.81	43.16 ± 1.85
CB30_165C_11h	1.65 ± 0.16	22.58 ± 0.72	30.44 ± 0.99	45.33 ± 0.78
CB30_165C_36h	1.52 ± 0.32	21.54 ± 1.66	30.89 ± 0.93	46.05 ± 0.92
CB30_190C_1h	1.49 ± 0.08	21.39 ± 1.64	31.98 ± 1.42	45.14 ± 1.42
CB30_190C_11h	1.78 ± 0.39	18.81 ± 1.82	30.38 ± 2.20	49.03 ± 2.43
CB30_190C_36h	1.81 ± 0.08	15.25 ± 1.77	31.14 ± 3.27	51.80 ± 1.59
CB16_165C_1h	1.54 ± 0.08	23.18 ± 2.61	32.08 ± 2.38	43.19 ± 0.35
CB16_165C_11h	1.88 ± 0.26	23.89 ± 4.11	29.56 ± 3.56	44.67 ± 0.86
CB16_165C_36h	1.25 ± 0.23	17.29 ± 0.1	31.53 ± 0.88	49.94 ± 0.81
CB16_190C_1h	1.27 ± 0.23	22.95 ± 3.33	31.36 ± 3.53	44.42 ± 0.83
CB16_190C_11h	1.04 ± 0.25	19.23 ± 4.13	31.63 ± 3.36	48.11 ± 0.75

Sample	Moisture and light oils (%)	Natural rubber (%)	Synthetic rubber and other polymers (%)	Carbon black and fillers (%)
CB16_190C_36h	1.39 ± 0.07	18.47 ± 1.68	30.29 ± 1.83	49.84 ± 0.72
MT30_165C_1h	3.91 ± 0.33	33.94 ± 1.54	22.14 ± 0.99	40.01 ± 0.61
MT30_165C_11h	1.65 ± 0.13	33.99 ± 1.46	20.90 ± 1.32	43.46 ± 0.49
MT30_165C_36h	1.53 ± 0.03	22.94 ± 1.08	22.85 ± 1.35	52.69 ± 0.67
MT30_190C_1h	1.35 ± 0.26	36.43 ± 0.46	18.71 ± 0.91	43.50 ± 0.20
MT30_190C_11h	1.12 ± 0.10	23.97 ± 0.68	24.34 ± 1.19	50.57 ± 1.20
MT30_190C_36h	0.67 ± 0.05	17.33 ± 0.64	29.20 ± 0.42	52.80 ± 0.40
MT16_165C_1h	6.36 ± 0.17	15.54 ± 0.74	31.44 ± 0.93	46.66 ± 0.76
MT16_165C_11h	5.99 ± 0.43	16.90 ± 2.30	28.89 ± 1.99	48.22 ± 0.50
MT16_165C_36h	1.86 ± 0.35	20.94 ± 2.21	25.25 ± 2.23	51.97 ± 1.93
MT16_190C_1h	4.44 ± 0.53	30.23 ± 2.75	22.58 ± 2.66	42.75 ± 0.72
MT16_190C_11h	1.99 ± 0.15	28.80 ± 2.94	23.96 ± 1.42	52.88 ± 2.10
MT16_190C_36h	1.67 ± 0.24	19.40 ± 3.48	26.05 ± 3.93	52.88 ± 2.10
TR	7.74 ± 1.26	35.01 ± 4.56	12.86 ± 3.23	44.39 ± 1.52
CT	6.84 ± 0.22	33.26 ± 0.59	19.39 ± 2.95	40.50 ± 2.61
CB	5.00 ± 0.20	27.96 ± 1.07	28.04 ± 3.57	38.99 ± 2.89
MT	4.69 ± 0.02	42.75 ± 0.59	17.59 ± 0.35	34.98 ± 0.33
TR30 after Soxhlet	2.01 ± 0.29	43.76 ± 4.94	12.08 ± 3.91	42.15 ± 2.65
TR16 after Soxhlet	2.10 ± 0.18	40.11 ± 1.57	16.28 ± 1.57	41.52 ± 1.37
CT30 after Soxhlet	2.14 ± 0.21	30.31 ± 2.62	23.70 ± 1.04	43.85 ± 1.94
CT16 after Soxhlet	2.67 ± 0.88	30.36 ± 2.01	20.43 ± 1.49	46.54 ± 0.83
CB30 after Soxhlet	2.21 ± 0.53	27.74 ± 2.14	28.07 ± 4.13	41.99 ± 4.23
CB16 after Soxhlet	1.49 ± 0.15	27.95 ± 4.23	28.87 ± 4.34	41.68 ± 2.87
MT30 after Soxhlet	0.60 ± 0.14	17.87 ± 0.72	29.23 ± 1.58	52.30 ± 1.31
MT16 after Soxhlet	4.58 ± 0.17	34.79 ± 3.98	24.54 ± 2.28	36.08 ± 1.99

Table D.2 to Table D.9 show a representative TGA and DTG curve for each of the extracted crumb rubber samples.

Table D.2: Temperature-mass and differential curves for extracted truck tyre crumb rubber blended at 165 °C

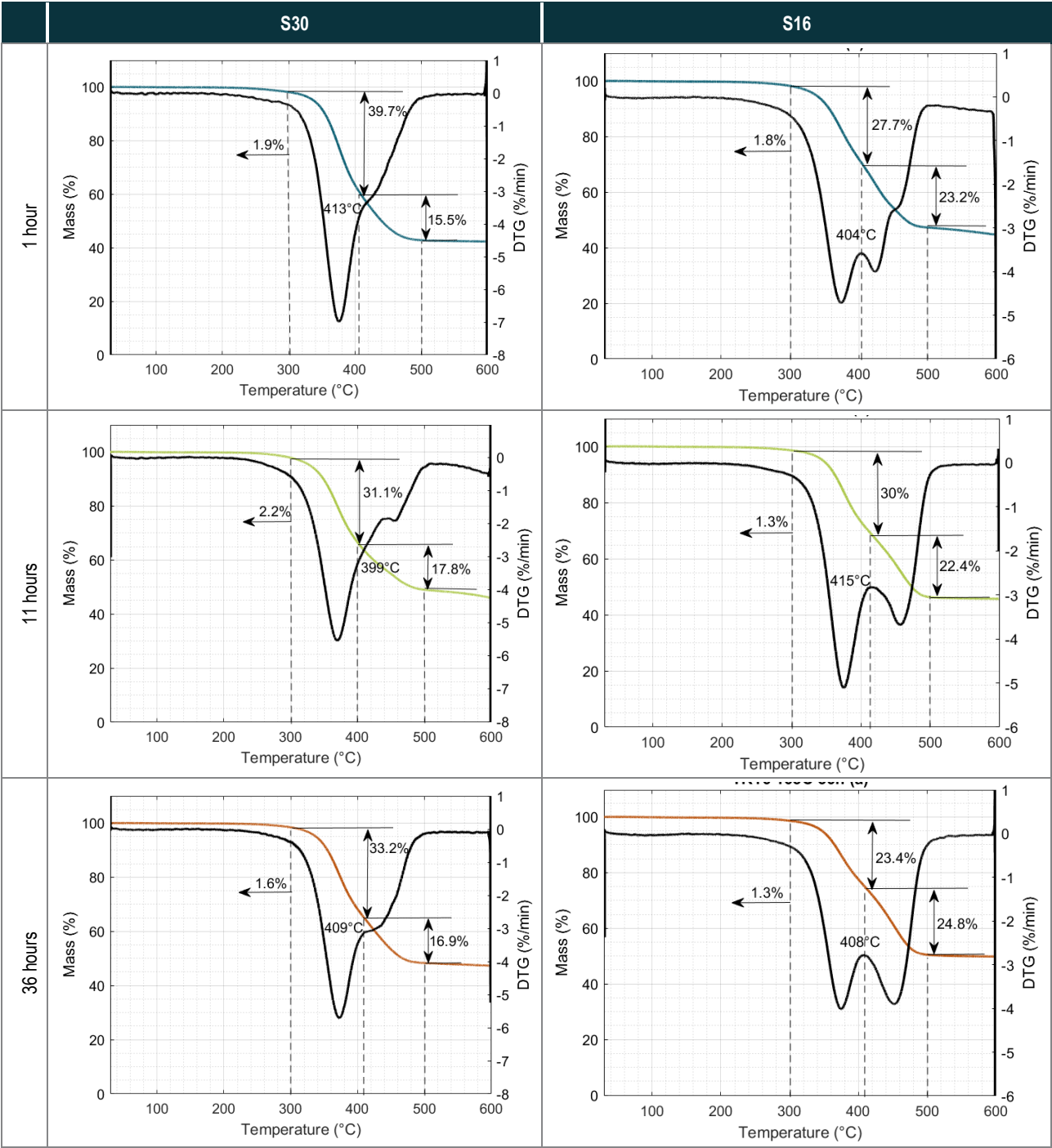


Table D.3: Temperature-mass and differential curves for extracted truck tyre crumb rubber blended at 190 °C

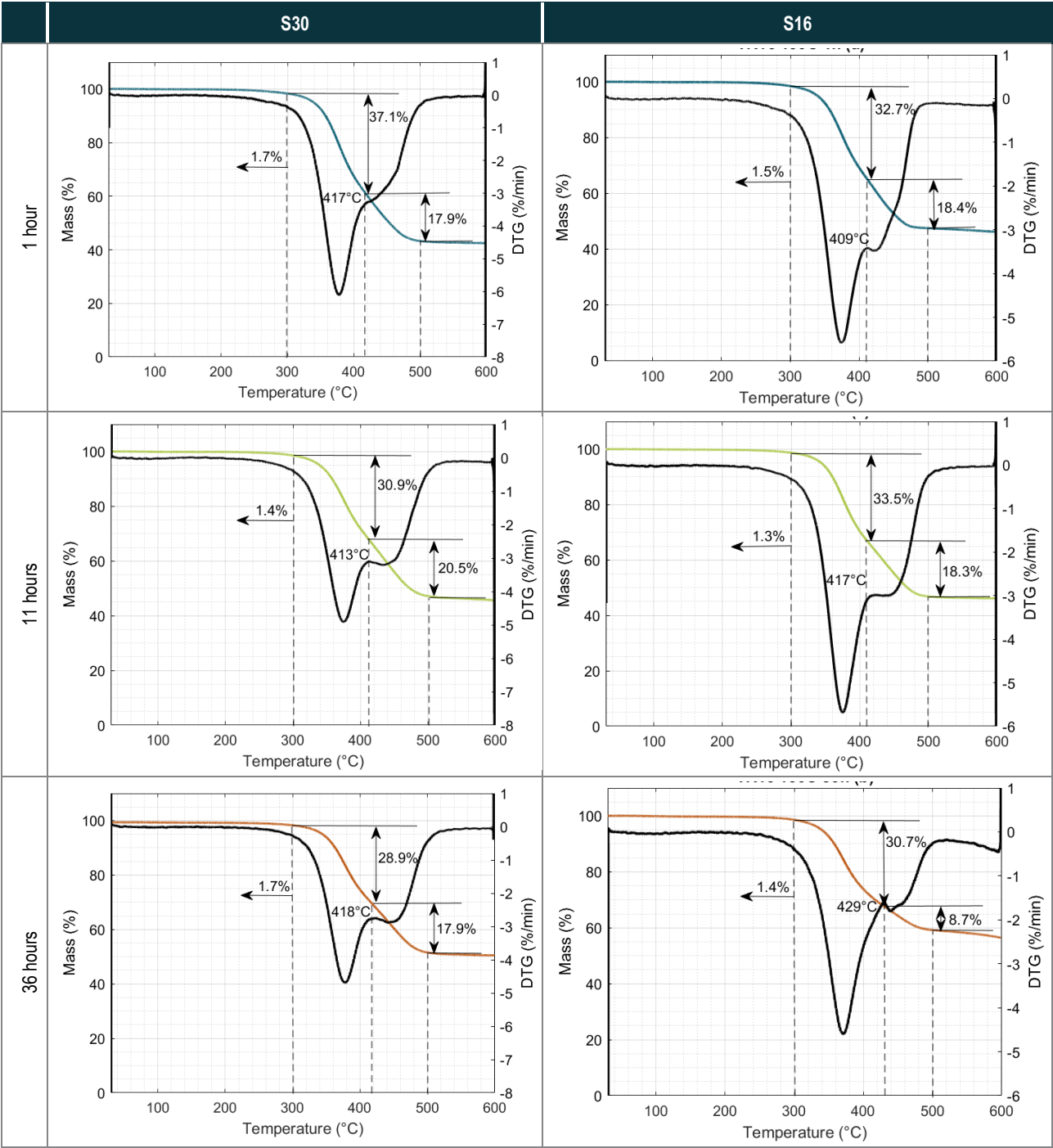


Table D.4: Temperature-mass and differential curves for extracted car tyre crumb rubber blended at 165 °C

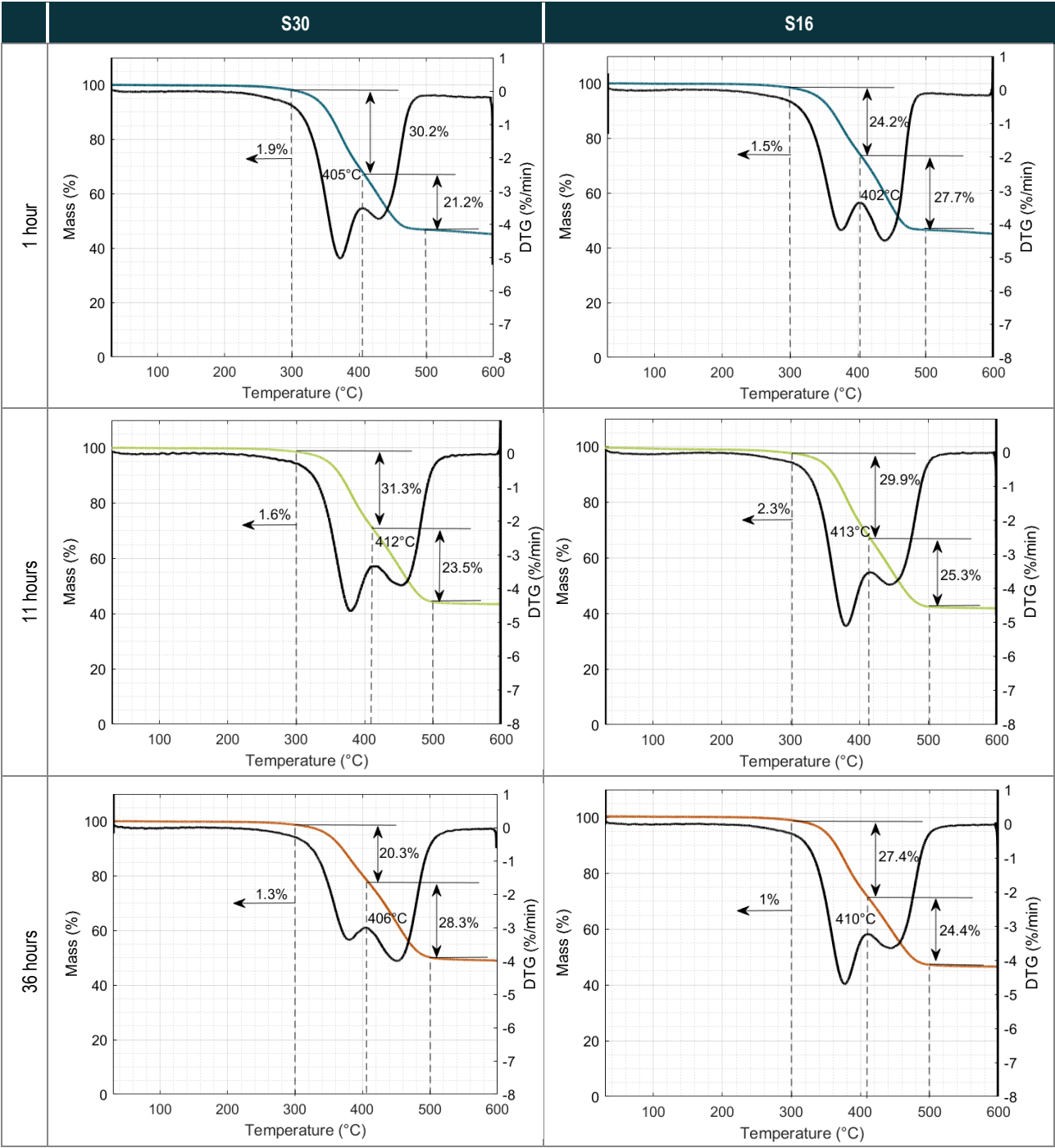


Table D.5: Temperature-mass and differential curves for extracted car tyre crumb rubber blended at 190 °C

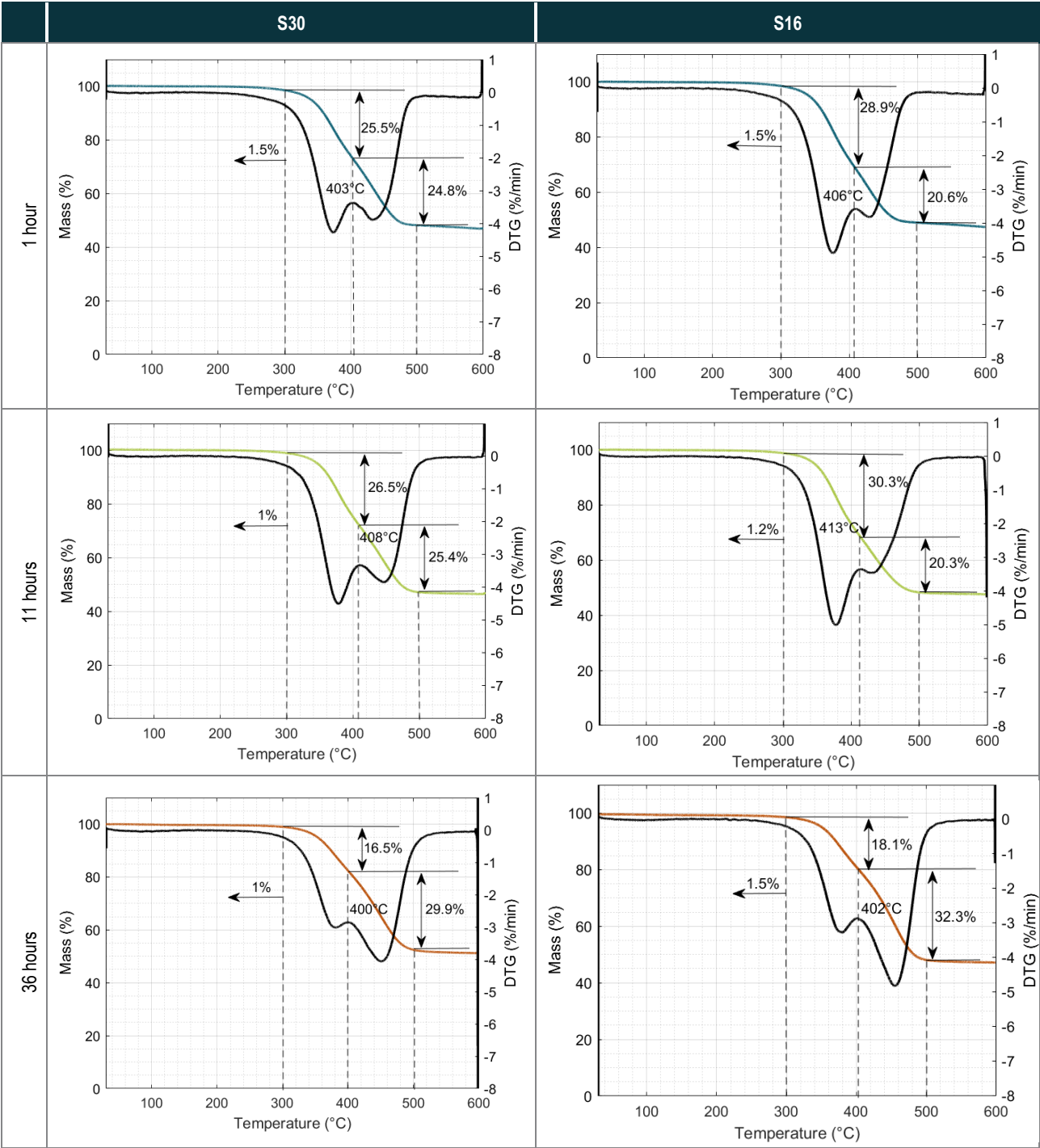


Table D.6: Temperature-mass and differential curves for extracted conveyor belt crumb rubber blended at 165 °C

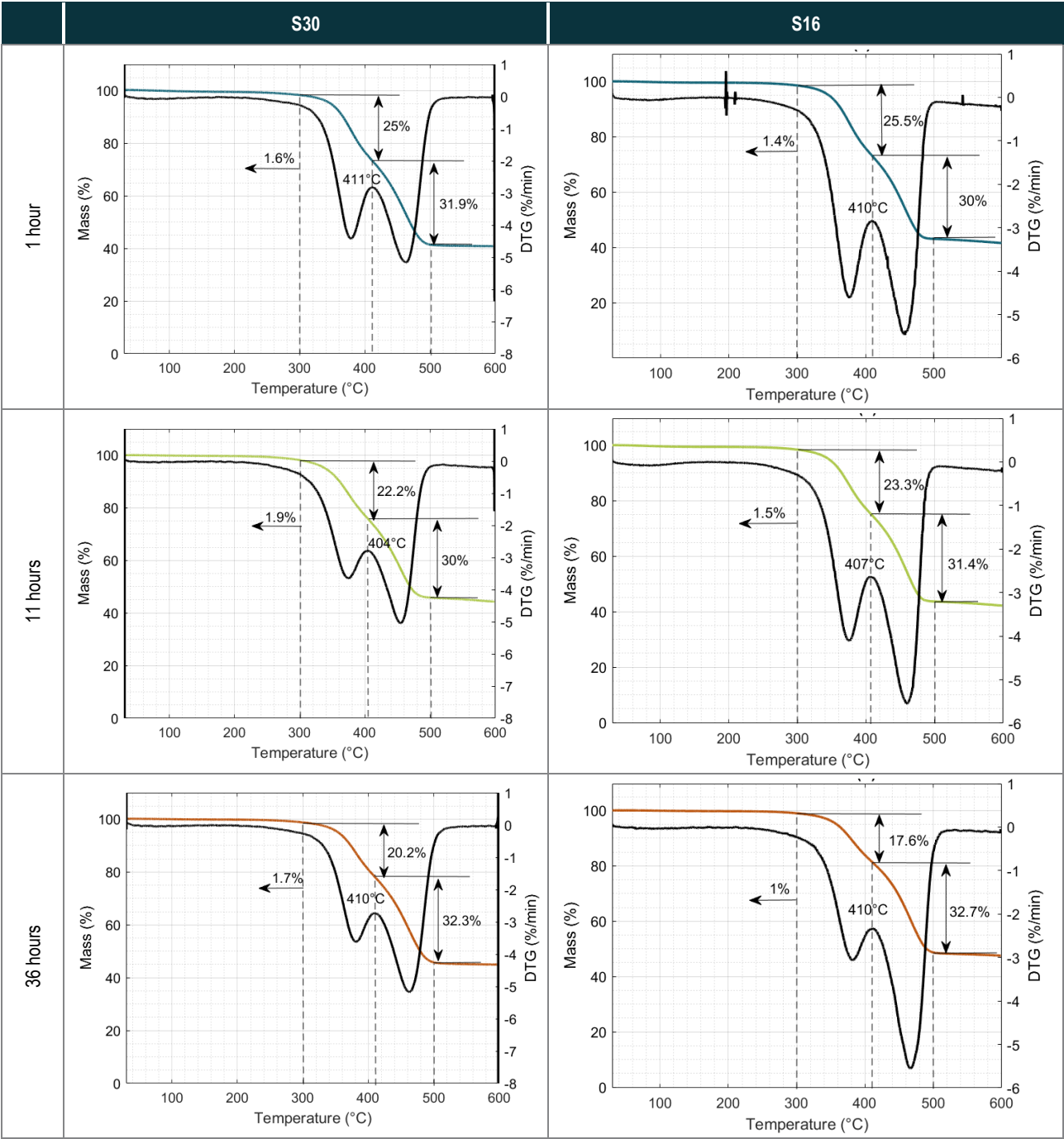


Table D.7: Temperature-mass and differential curves for extracted conveyor belt crumb rubber blended at 190 °C

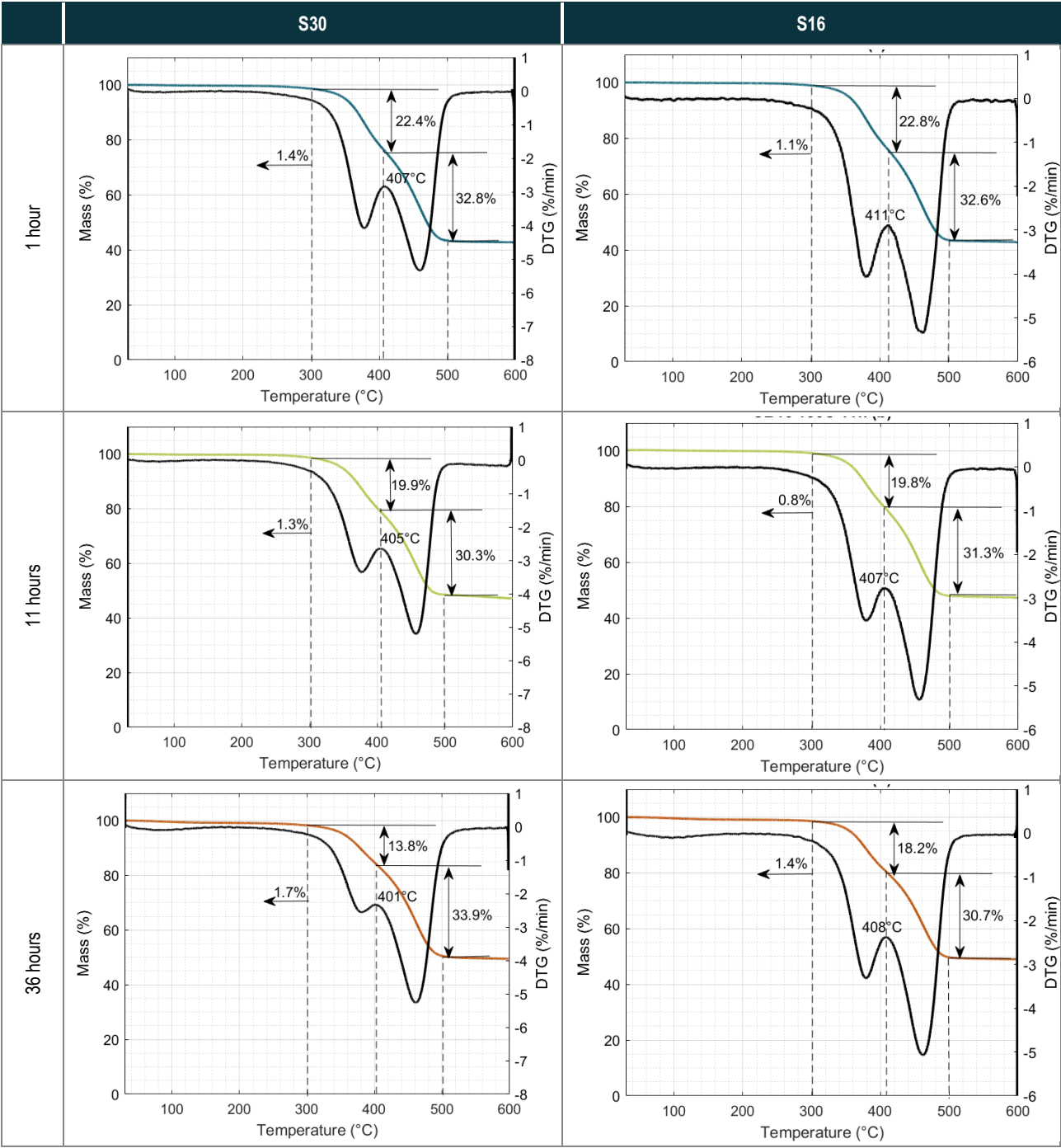


Table D.8: Temperature-mass and differential curves for extracted mining tyre crumb rubber blended at 165 °C

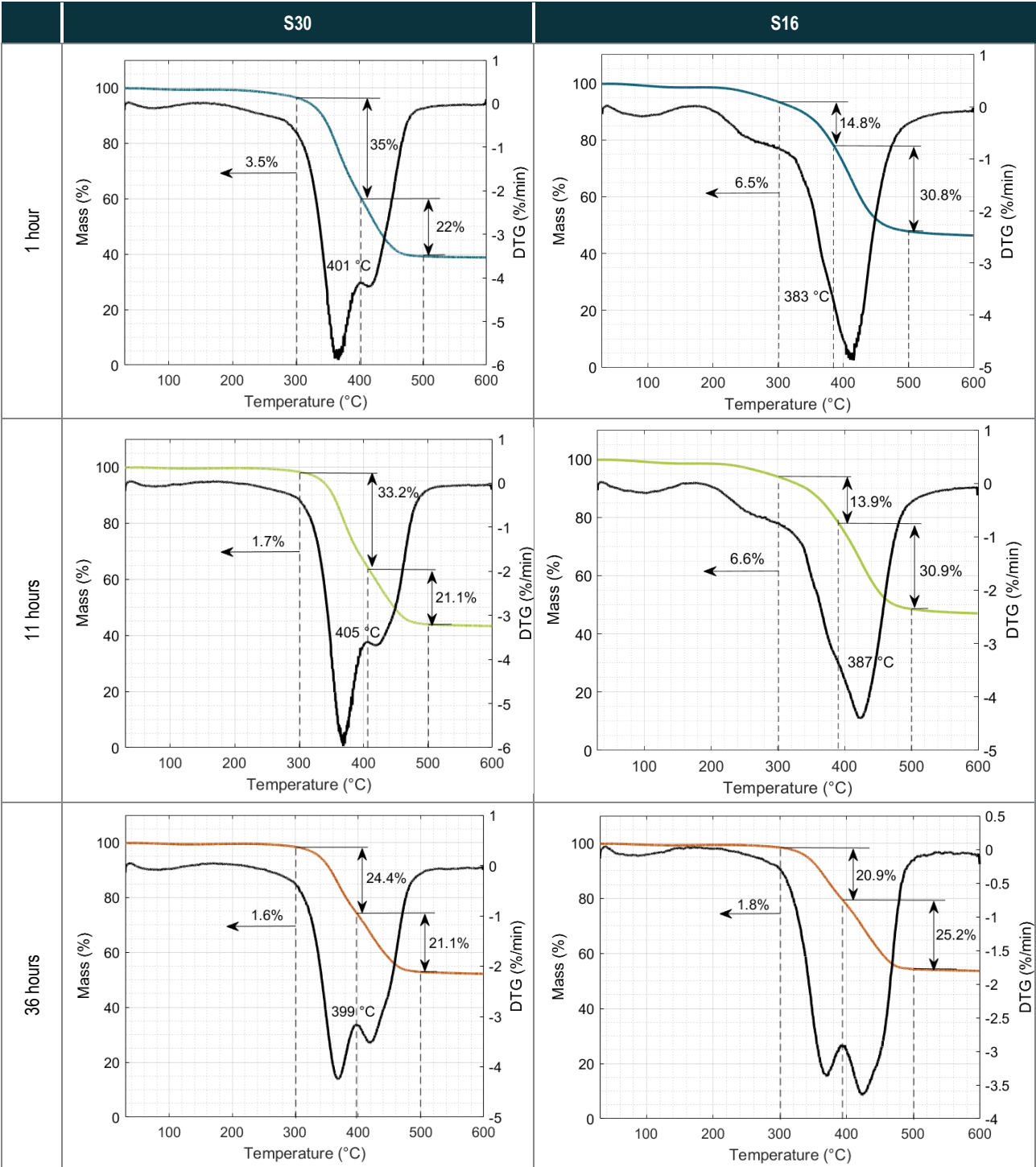
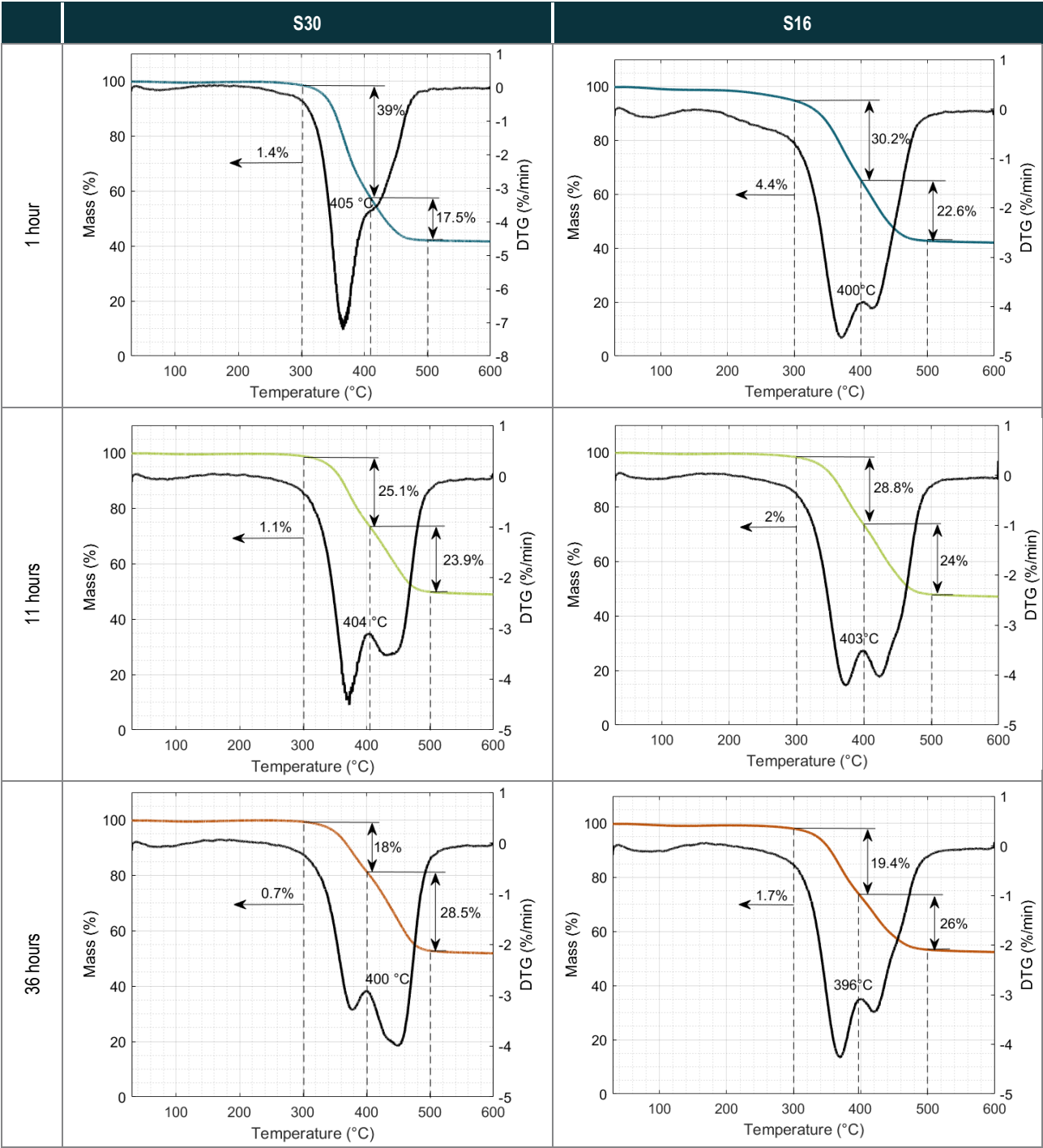


Table D.9: Temperature-mass and differential curves for extracted mining tyre crumb rubber blended at 190 °C



D.2 Particle Size Distribution

Table D.10 presents the 10th, 50th and 90th percentile and mean particle size with standard deviation for all samples listed in Table A.5.

Table D.10: Particle size distribution average percentiles with standard deviation; number of specimens for each sample are noted in 'Mean' column in parenthesis

Sample	x10.0 (µm)	x50.0 (µm)	x90.0 (µm)	Mean (no. of specimens)
TR16_165C_1h	985.2 ± 46.9	1,556.3 ± 64.0	2,389.6 ± 1633.3	1,633.3 ± 53.6 (2)
TR16_165C_11h	1,393.6 ± 128.5	2,400.7 ± 282.3	4,558.1 ± 1319.1	2,618.0 ± 423.8 (3)
TR16_165C_36h	1,428.1 ± 226.8	3,253.6 ± 1005.8	6,058.4 ± 1504.3	3,666.5 ± 991.1 (3)
TR16_190C_1h	1,477.1 ± 180.1	2,753.5 ± 372.6	4,963.8 ± 884.9	2,790.6 ± 384.4 (3)
TR16_190C_11h	2,068.2 ± 398.7	5,391.6 ± 1836.0	7,655.0 ± 293.4	4,944.3 ± 1038.3 (3)
TR16_190C_36h	1,188.7 ± 82.1	2,147.0 ± 228.7	3,900.0 ± 486.4	2,406.7 ± 234.7 (3)
CT16_165C_1h	1,016.7 ± 90.6	1,684.3 ± 122.8	2,729.4 ± 325.7	1,766.9 ± 173.5 (3)
CT16_165C_11h	1,660.9 ± 129.8	3,546.3 ± 713.6	5,706.1 ± 801.9	3,792.7 ± 829.9 (2)
CT16_165C_36h	1,509.2 ± 496.0	3,517.3 ± 1537.2	5,029.6 ± 968.4	3,445.3 ± 1099.9 (3)
CT16_190C_1h	1,409.7 ± 226.1	2,518.0 ± 492.0	3,887.6 ± 317.4	2,537.9 ± 278.3 (3)
CT16_190C_11h	1,254.9 ± 13.4	2,553.6 ± 100.1	4,111.0 ± 99.9	2,558.0 ± 59.6 (2)
CT16_190C_36h	2,000.7 ± 823.9	4,085.6 ± 1566.0	4,973.5 ± 1363.8	3,910.3 ± 1348.0 (2)
CB16_165C_1h	994.2 ± 79.4	1,451.0 ± 39.1	2,430.5 ± 260.1	1,578.1 ± 0.2 (2)
CB16_165C_11h	873.9 ± 92.0	1,394.5 ± 109.9	1,999.8 ± 404.6	1,428.0 ± 148.5 (2)
CB16_165C_36h	960.5 ± 3.6	1,858.1 ± 83.2	3,375.4 ± 141.1	2,039.7 ± 96.4 (2)
CB16_190C_1h	1,104.4 ± 43.2	1,668.1 ± 52.5	2,543.8 ± 64.1	1,728.1 ± 78.2 (4)
CB16_190C_11h	1,520.7 ± 96.6	2,686.2 ± 151.4	4,292.8 ± 387.7	2,777.7 ± 130.3 (3)
CB16_190C_36h	1,464.4 ± 346.0	2,189.7 ± 479.3	2,955.5 ± 164.4	2,173.1 ± 348.0 (3)
TR30	408.5 ± 29.4	650.8 ± 43.7	1,240.0 ± 76.6	730.8 ± 61.0 (4)
TR16	862.0 ± 43.8	1,129.0 ± 29.7	1,568.4 ± 38.2	1,169.5 ± 36.6 (2)
CT30	347.3 ± 39.4	640.0 ± 54.6	1,197.9 ± 67.1	707.5 ± 57.6 (3)
CB30	219.5 ± 72.5	419.6 ± 117.0	728.6 ± 116.5	451.8 ± 101.9 (4)
CB16	758.2 ± 24.4	1,156.4 ± 8.9	1,431.3 ± 24.3	1,119.3 ± 19.8 (2)

Appendix E Specification Compliance

E.1 Compliance with Main Roads Western Australia Specification 511 Requirements

Table E.1 summarises the binder requirements as specified in MRWA Specification 511:2025. MRWA Specifications 516 for OGA and 517 for GGA reference MRWA Specification 511 when providing the requirements for CRMB performance.

Table E.1: Properties of crumb rubber-modified binders for use in asphalt applications as per MRWA Specification 511:2025

Binder property	Test method	Digestion time				
		60 minutes	120 minutes	240 minutes	11 hours	Maximum ⁽¹⁾
Penetration at 4 °C, 200 g, 60 s, pu (minimum)	AS 2341.12	15	–	15	15	15
Resilience at 25 °C, % rebound (minimum)	ASTM D5329	20	–	20	20	20
Consistency 6% at 60 °C	AGPT-T121	Report	–	Report	Report	Report
Torsional recovery at 25 °C, 30 s, (%)	ATM 122	Report	–	Report	Report	Report
Softening point, (°C) (minimum)	AGPT-T131	55	–	55	55	55
Compressive limit at 70 °C, 2 kg, (mm) (minimum)	ATM 132	0.2	–	0.2	0.2	0.2
Stiffness at 25 °C (kPa) (maximum)	AGPT-T121	180	–	180	180	180
Viscosity at 175 °C	ASTM D7741/D7741M (Note 2 & 3)	1.5-4.0				
	ATM 111	Report				

1. The asphalt manufacturer is to nominate the maximum period of time it intends to store the crumb rubber-modified binder beyond 11 hours. The properties of the binder must comply with the table after this period of time.

Source: MRWA Specification 511:2025.

Table E.2 presents the comparison of the results obtained for S30 TR-derived CRMBs against MRWA Specification 511:2025. It is shown that the binders met all relevant specifications irrespective of blending time and temperature.

Table E.2: MRWA Specification 511:2025 pass/fail (green/red) summary for S30 truck tyre–derived crumb rubber-modified binders

Binder property	TR30_1h	TR30_2h	TR30_4h	TR30_11h	TR30_24h	TR30_36h
	165C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
	190C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						

Table E.3 shows the comparison of S16 TR-derived CRMBs against MRWA Specification 511:2025, where all binders appear to meet specification irrespective of blending time and temperature.

Table E.3: MRWA Specification 511:2025 pass/fail (green/red) summary for S16 truck tyre–derived crumb rubber-modified binders

Binder property	TR16_1h	TR16_2h	TR16_4h	TR16_11h	TR16_24h	TR16_36h
	165C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
	190C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						

According to Table E.4, all S30 CT-derived CRMBs were found to meet MRWA Specification 511:2025 irrespective of blending time and temperature.

Table E.4: MRWA Specification 511:2025 pass/fail (green/red) summary for S30 car tyre–derived crumb rubber-modified binders

Binder property	CT30_1h	CT30_2h	CT30_4h	CT30_11h	CT30_24h	CT30_36h
	165C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
	190C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						

Table E.5 shows that S16 CT-derived CRMBs also met MRWA Specification 511:2025 irrespective of blending time and temperature.

Table E.5: MRWA Specification 511:2025 pass/fail (green/red) summary for S16 car tyre–derived crumb rubber-modified binders

Binder property	CT16_1h	CT16_2h	CT16_4h	CT16_11h	CT16_24h	CT16_36h
	165C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
	190C					
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						

Additional tests were conducted for CB-derived CRMBs, the results of which are presented in Appendix C.6. From Table E.6, all S30 CB-derived CRMBs met MRWA Specification 511:2025 irrespective of blending time and temperature.

Table E.6: MRWA Specification 511:2025 pass/fail (green/red) summary for S30 conveyor belt–derived crumb rubber-modified binders

Binder property	CB30_1h	CB30_2h	CB30_4h	CB30_11h	CB30_24h	CB30_36h
	165C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Stiffness at 25 °C (kPa) (maximum)						
	190C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Stiffness at 25 °C (kPa) (maximum)						

Table E.7 shows that all S16 CB-derived CRMBs also met MRWA Specification 511:2025.

Table E.7: MRWA Specification 511:2025 pass/fail (green/red) summary for S16 conveyor belt–derived crumb rubber-modified binders

Binder property	CB16_1h	CB16_2h	CB16_4h	CB16_11h	CB16_24h	CB16_36h
	165C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Stiffness at 25 °C (kPa) (maximum)						
	190C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Stiffness at 25 °C (kPa) (maximum)						

Table E.8 shows that binders produced with S30 MT-derived crumb rubber generally met the requirements of MRWA Specification 511:2025, except from samples MT30_165C_36h and MT30_190C_36h, which failed to meet the minimum specified requirements for compressive limit at 70 °C.

Table E.8: MRWA Specification 511:2025 pass/fail (green/red) summary for S30 mining tyre-derived crumb rubber-modified binders

Binder property	MT30_1h	MT30_2h	MT30_4h	MT30_11h	MT30_24h	MT30_36h
	165C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						Lower
Stiffness at 25 °C (kPa) (maximum)						
	190C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						Lower
Stiffness at 25 °C (kPa) (maximum)						

Table E.9 shows that all CRMBs produced with S16 MT-derived crumb rubber met the requirements of MRWA Specification 511:2025.

Table E.9: MRWA Specification 511:2025 pass/fail (green/red) summary for S16 mining tyre-derived crumb rubber-modified binders

Binder property	MT16_1h	MT16_2h	MT16_4h	MT16_11h	MT16_24h	MT16_36h
	165C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Stiffness at 25 °C (kPa) (maximum)						
	190C					
Penetration at 4 °C, 200 g, 60 s, pu (minimum)						
Resilience at 25 °C, % rebound (minimum)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Stiffness at 25 °C (kPa) (maximum)						

E.2 Compliance with Austroads Technical Specification ATS 3110 Requirements

Table E.10 summarises the specifications for CRMBs according to Austroads ATS 3110:2023. It should be noted that Austroads ATS 3110:2023 specification limits only relate to the samples taken at the point of manufacture, unless specified otherwise. Point of manufacture samples are collected immediately after a batch has been produced and not after long-term storage. As the CRMBs that were sampled after 24 and 36 hours would represent samples that were manufactured and then stored for up to several days, these CRMBs would not be considered to not meet the specification limits of Austroads ATS 3110:2023 in a production/field situation.

Table E.10: Properties of crumb rubber-modified binders for use in asphalt applications (A18R) as specified in the relevant Austroads Technical Specification

Binder property	Test method	A18R
Viscosity at 165 °C (Pa.s) max.	AS/NZS 2341.4 or ATM 111	6.2
Torsional recovery at 25 °C, 30 s (%)	ATM 122	30–70
Softening point (°C)	AS 2341.18	62–80
Stress ratio at 10 °C min.	AGPT-T125	TBR
Consistency 6% at 60 °C (Pa.s) min.	AGPT-T121	1,000
Compressive limit at 70 °C, 2 kg (mm) min.	ATM 132	0.1
Segregation (%) max.	AGPT-T108	–8 to +8
Flash point (°) min.	ATM 112	250
Mass change (%)	ATM 103	–0.6 to +0.6

Source: Austroads (2023).

Table E.11 shows that all S30 TR-derived CRMBs met Austroads ATS 3110:2023 irrespective of blending temperature and time.

Table E.11: Austroads ATS 3110:2023 pass/fail (green/red) summary for S30 truck tyre–derived crumb rubber-modified binders

Binder property	TR30_1h	TR30_2h	TR30_4h	TR30_11h	TR30_24h	TR30_36h
	165C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						
	190C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						

Binders modified by S16 TR-derived crumb rubber were also found to meet Austroads ATS 3110:2023 for all blending times and temperatures investigated, as shown in Table E.12.

Table E.12: Austroads ATS 3110:2023 pass/fail (green/red) summary for S16 truck tyre–derived crumb rubber-modified binders

Binder property	TR16_1h	TR16_2h	TR16_4h	TR16_11h	TR16_24h	TR16_36h
	165C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						
	190C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						

Table E.13 reveals that S30 CT-derived CRMBs also met Austroads ATS 3110:2023 for all blending temperatures and times investigated.

Table E.13: Austroads ATS 3110:2023 pass/fail (green/red) summary for S30 car tyre–derived crumb rubber-modified binders

Binder property	CT30_1h	CT30_2h	CT30_4h	CT30_11h	CT30_24h	CT30_36h
	165C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						
	190C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						

According to Table E.14, samples CT16_190C_24h and CT16_190C_36h did not meet the softening point requirements of Austroads ATS 3110:2023, with results below the minimum 62 °C permitted. All other binders, however, were found to meet the requirements of Austroads ATS 3110:2023.

Table E.14: Austroads ATS 3110:2023 pass/fail (green/red) summary for S16 car tyre–derived crumb rubber-modified binders

Binder property	CT16_1h	CT16_2h	CT16_4h	CT16_11h	CT16_24h	CT16_36h
	165C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						
	190C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)					Lower	Lower
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						

The requirements of Austroads ATS 3110:2023 were found to be met by all binders produced using S30 CB-derived CRMBs irrespective of blending parameters, as indicated by Table E.15.

Table E.15: Austroads ATS 3110:2023 pass/fail (green/red) summary for S30 conveyor belt–derived crumb rubber-modified binders

Binder property	CB30_1h	CB30_2h	CB30_4h	CB30_11h	CB30_24h	CB30_36h
	165C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						
	190C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						

As shown in Table E.16, S16 CB-derived CRMBs were, in their majority, found to meet Austroads ATS 3110:2023, irrespective of blending time and temperature.

Table E.16: Austroads ATS 3110:2023 pass/fail (green/red) summary for S16 conveyor belt–derived crumb rubber-modified binders

Binder property	CB16_1h	CB16_2h	CB16_4h	CB16_11h	CB16_24h	CB16_36h
	165C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)	Lower	Lower				
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						
	190C					
Viscosity at 165 °C (Pa.s) max.						
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)						

Table E.17 shows that CRMBs produced with S30 MT-derived crumb rubber failed some of the investigated parameters, predominantly that of viscosity at 165 °C. As noted earlier, failure to meet the requirements of Austroads ATS 3110:2023 after blending for 24 and 36 hours does not mean that the binder is not compliant with the specification, rather that the specified limits do not consider binders that have been blended for such extended durations. Regardless, without appropriate limits having been set for binders blended for 24 and 36 hours, only samples MT30_165C_1h and MT30_165C_2h can be said to meet the requirements of Austroads ATS 3110:2023.

Table E.17: Austroads ATS 3110:2023 pass/fail (green/red) summary for S30 mining tyre–derived crumb rubber-modified binders

Binder property	MT30_1h	MT30_2h	MT30_4h	MT30_11h	MT30_24h	MT30_36h
	165C					
Viscosity at 165 °C (Pa.s) max.			Greater	Greater	Greater	
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						Lower
Mass change (%)						
	190C					
Viscosity at 165 °C (Pa.s) max.	Greater	Greater	Greater	Greater		
Consistency 6% at 60 °C (Pa.s) min.						Lower
Torsional recovery at 25 °C, 30 s, (%)						
Softening point, (°C)						Lower
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						Lower
Mass change (%)						

According to Table E.18, no binder produced using S16 MT-derived crumb rubber was found to meet all the requirements of Austroads ATS 3110:2023.

Table E.18: Austroads ATS 3110:2023 pass/fail (green/red) summary for S16 mining tyre-derived crumb rubber-modified binders

Binder property	MT16_1h	MT16_2h	MT16_4h	MT16_11h	MT16_24h	MT16_36h
	165C					
Viscosity at 165 °C (Pa.s) max.			Greater		Greater	
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)	Lower	Lower	Lower	Lower	Lower	
Softening point, (°C) (minimum)						
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)	Greater					
	190C					
Viscosity at 165 °C (Pa.s) max.	Greater	Greater	Greater	Greater		
Consistency 6% at 60 °C (Pa.s) min.						
Torsional recovery at 25 °C, 30 s, (%)	Lower				Lower	
Softening point, (°C) (minimum)						Lower
Compressive limit at 70 °C, 2 kg, (mm) (minimum)						
Mass change (%)	Greater					

The parameters not listed in Table E.3 to Table E.9 for MRWA Specification 511:2025 and Table E.11 to Table E.18 for Austroads ATS 3110:2023 require the manufacturer to 'Report' the test results as indicated by Table E.1 and Table E.10, and so, these were not assessed by the pass/fail criterion.



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