

Technical Insights of KURARAY LIQUID RUBBER

Liquid Farnesene Rubber in Tire Formulation

Elastomer R&D Department
Elastomer Division

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Technical Insights of KURARAY LIQUID RUBBER

New Bio-based Liquid Polymer in Various Formulations

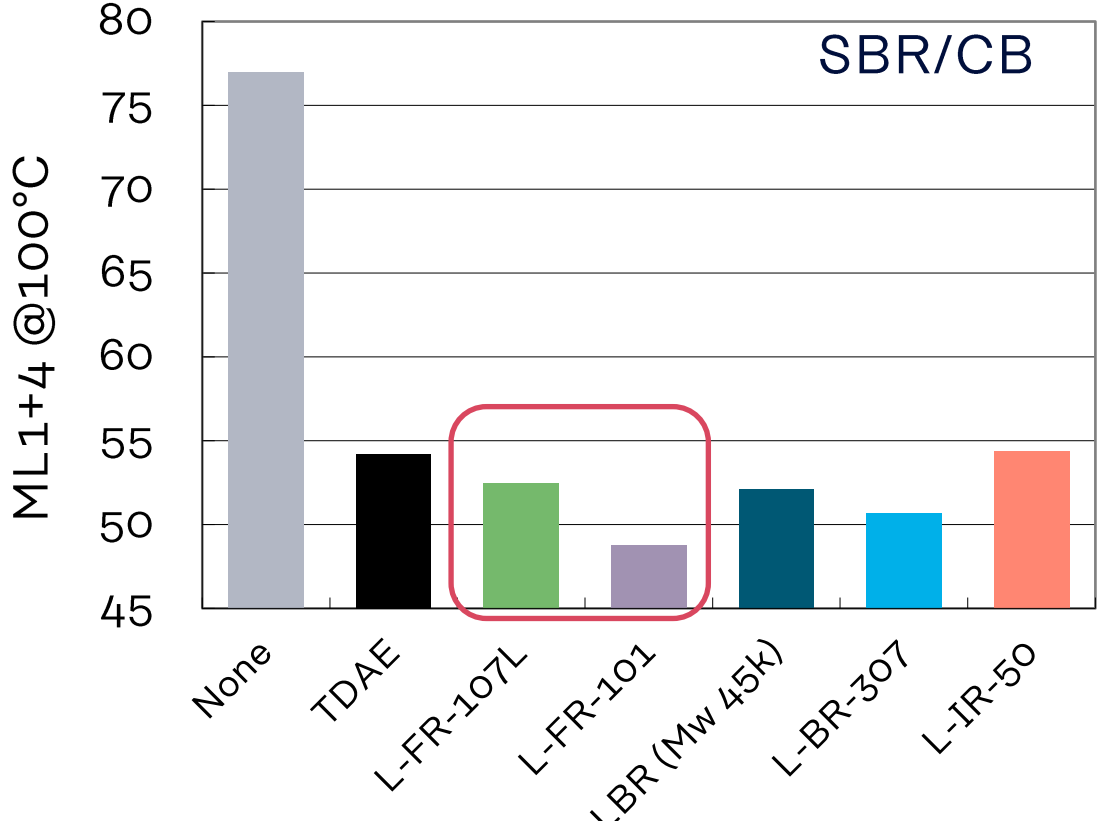
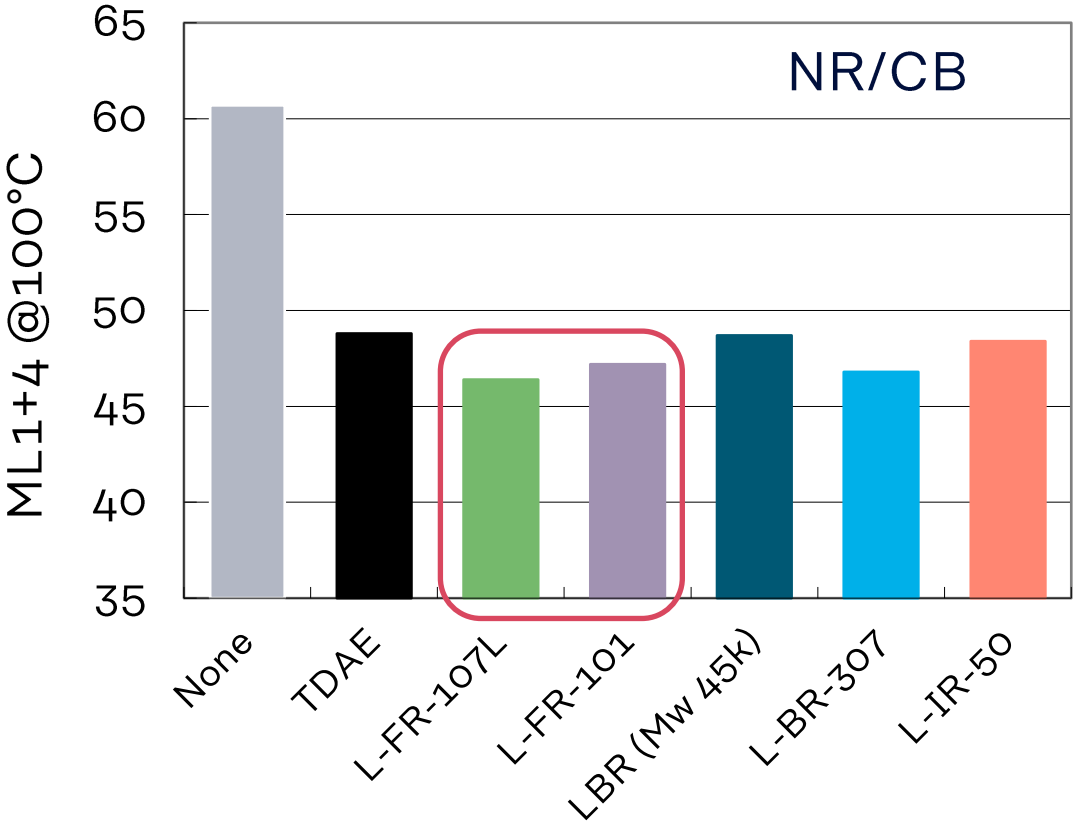
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Test Formulation

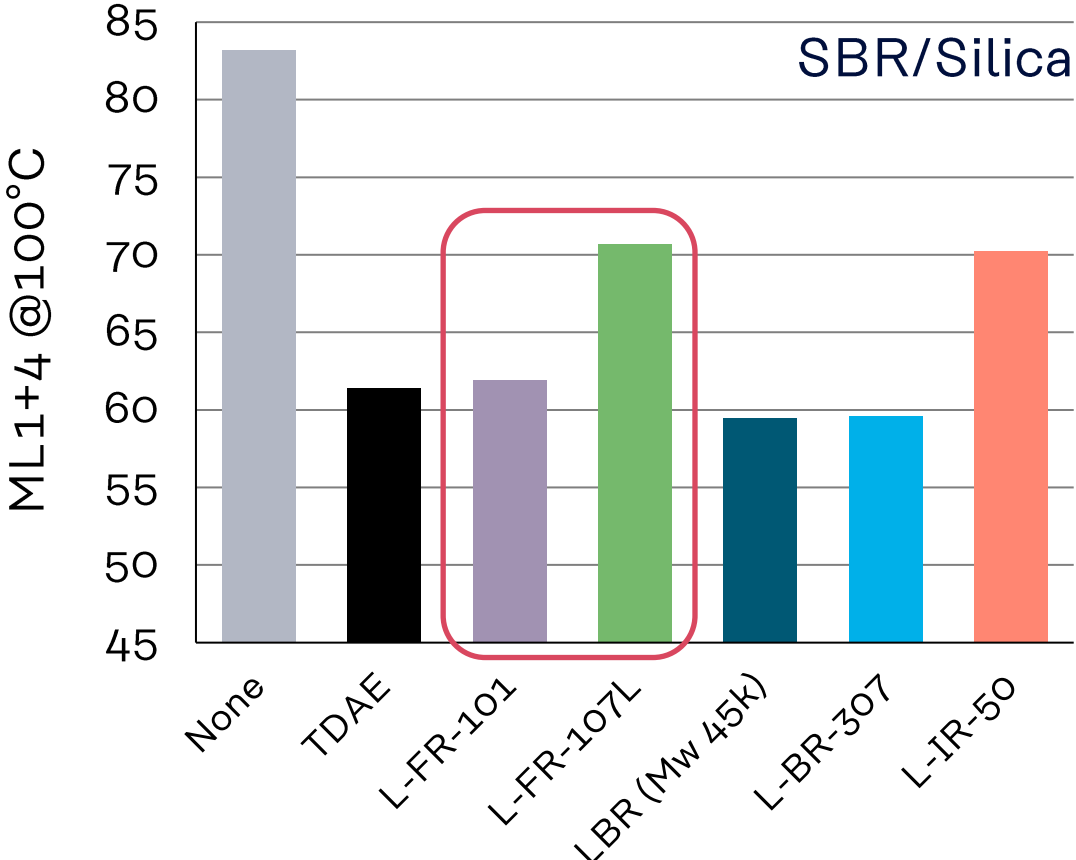
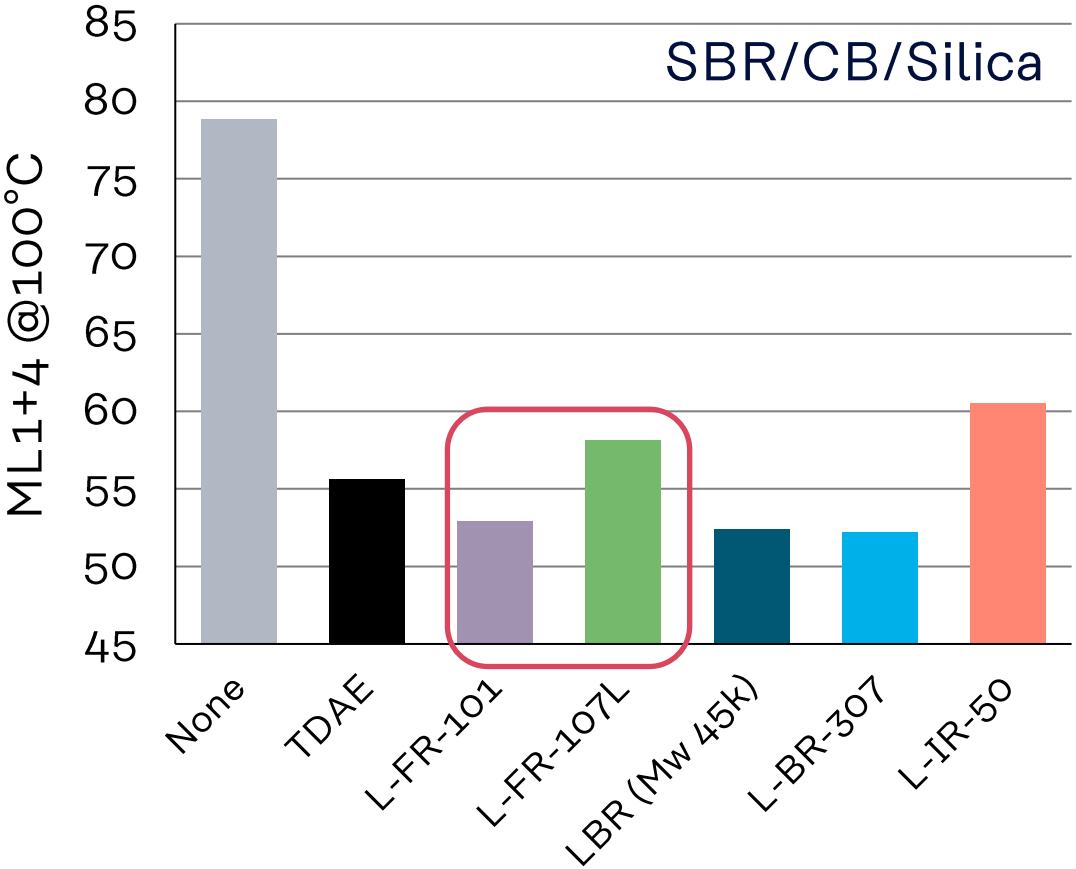
- NR/ CB/ Plasticizer = 100/ 50/ 10
- SBR/ CB/ Plasticizer = 100/ 50/ 10
- SBR/ CB/ Silica/ Plasticizer = 100/ 25/ 25/ 10
- SBR/ Silica/ Plasticizer = 100/ 50/ 10

Mooney Viscosity 1



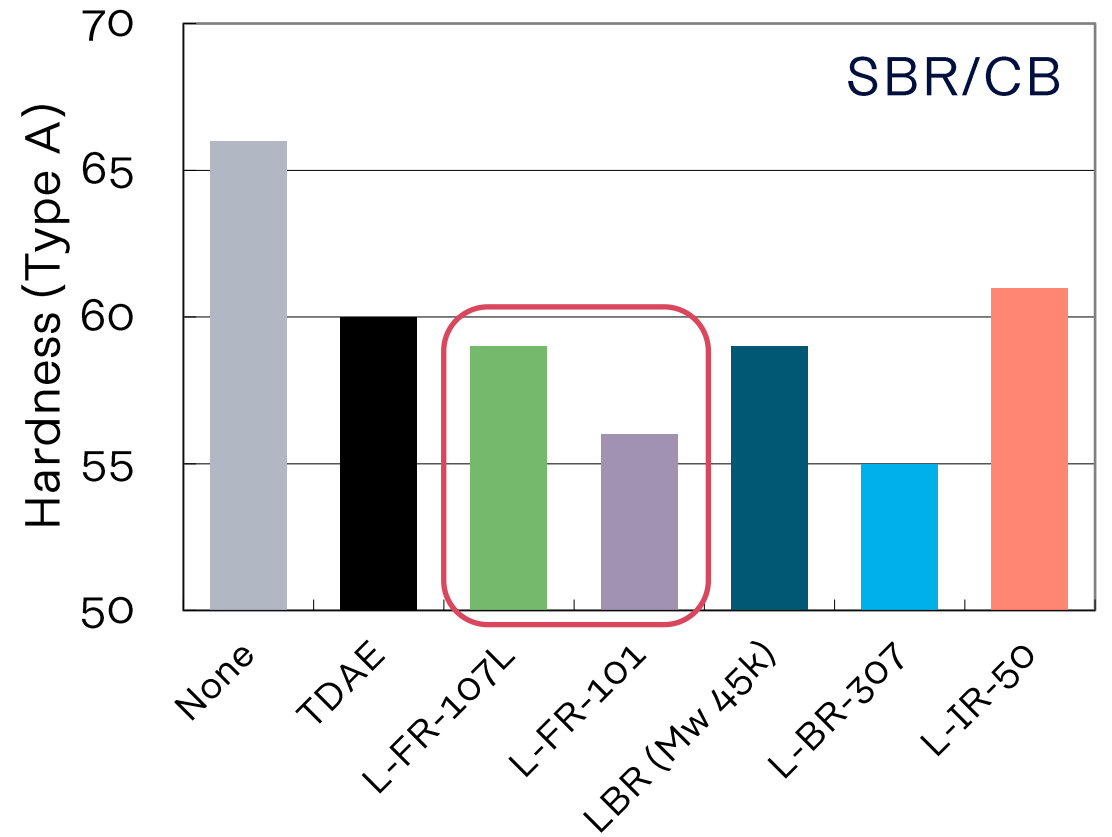
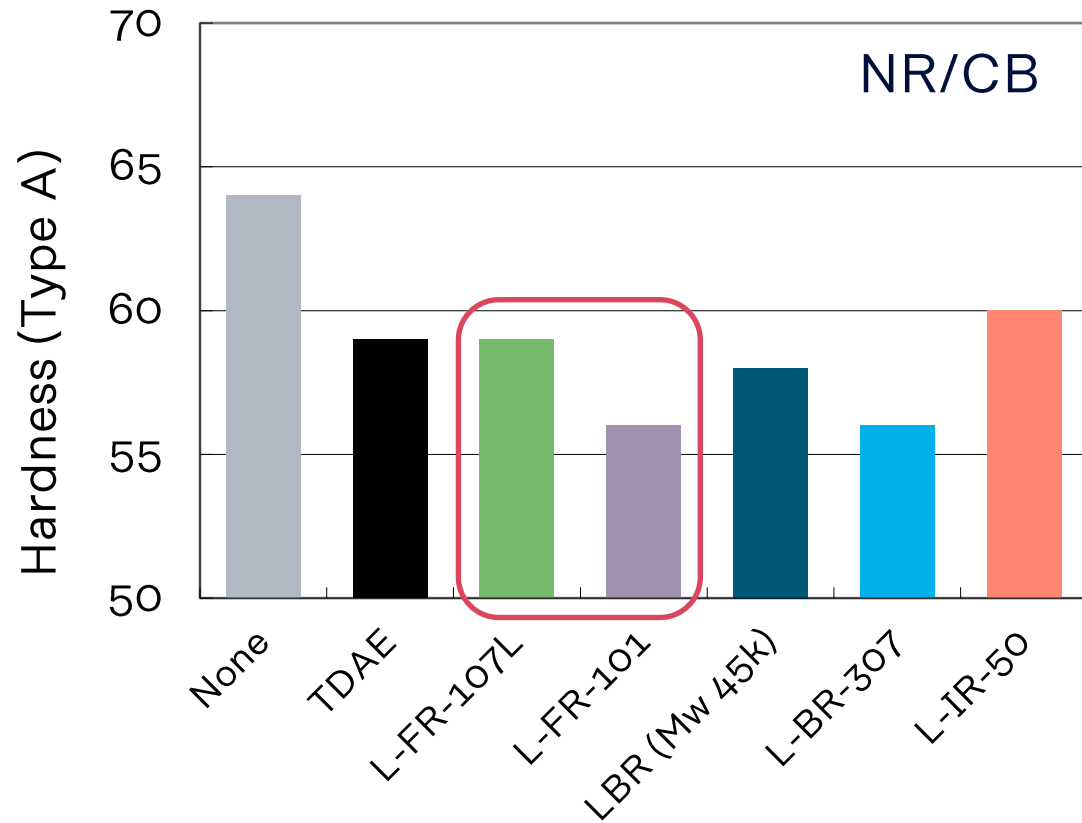
Better Plasticizing Effect

Mooney Viscosity 2

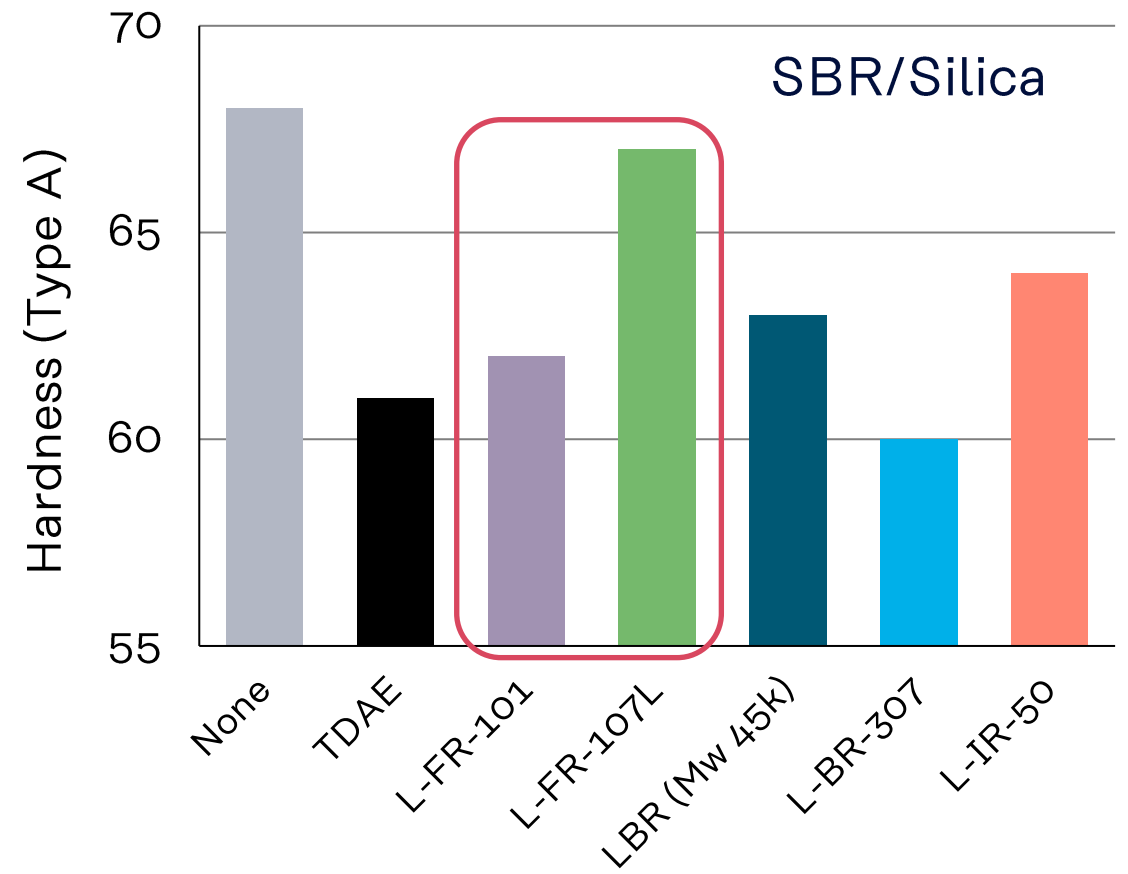
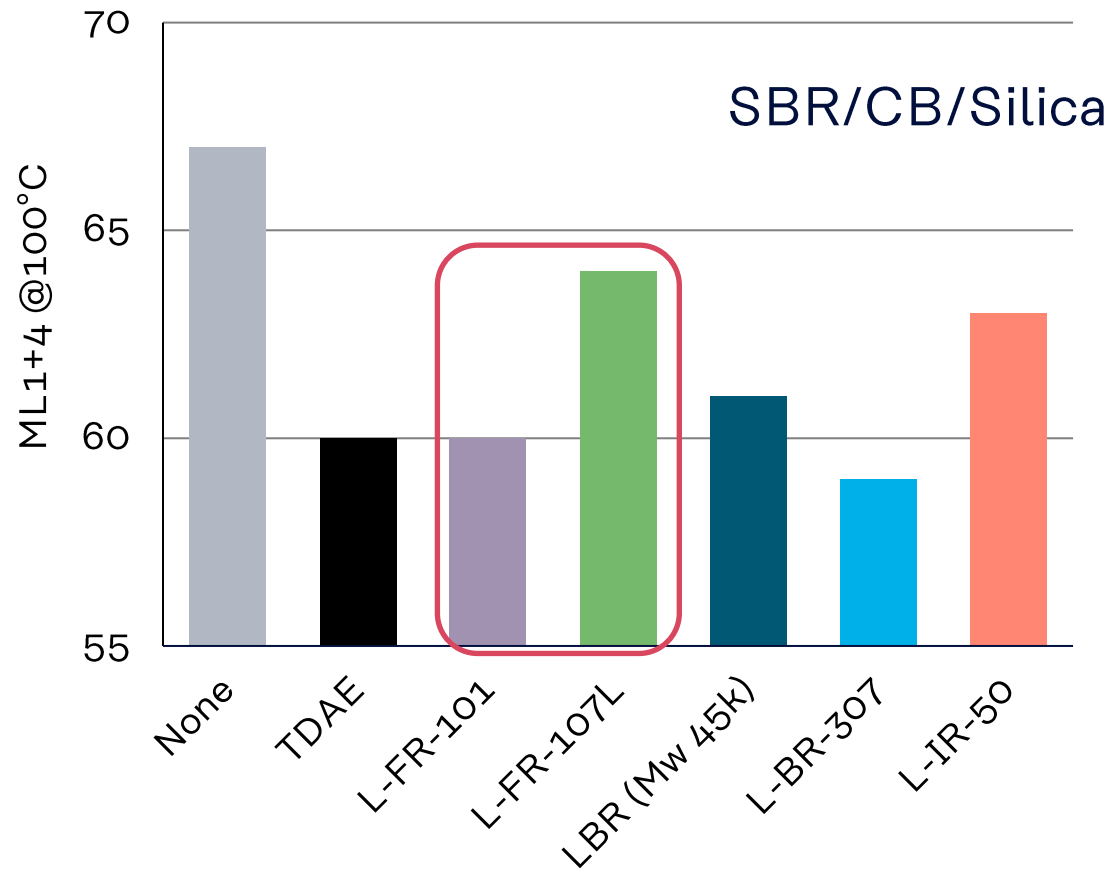


Good Plasticizing Effect

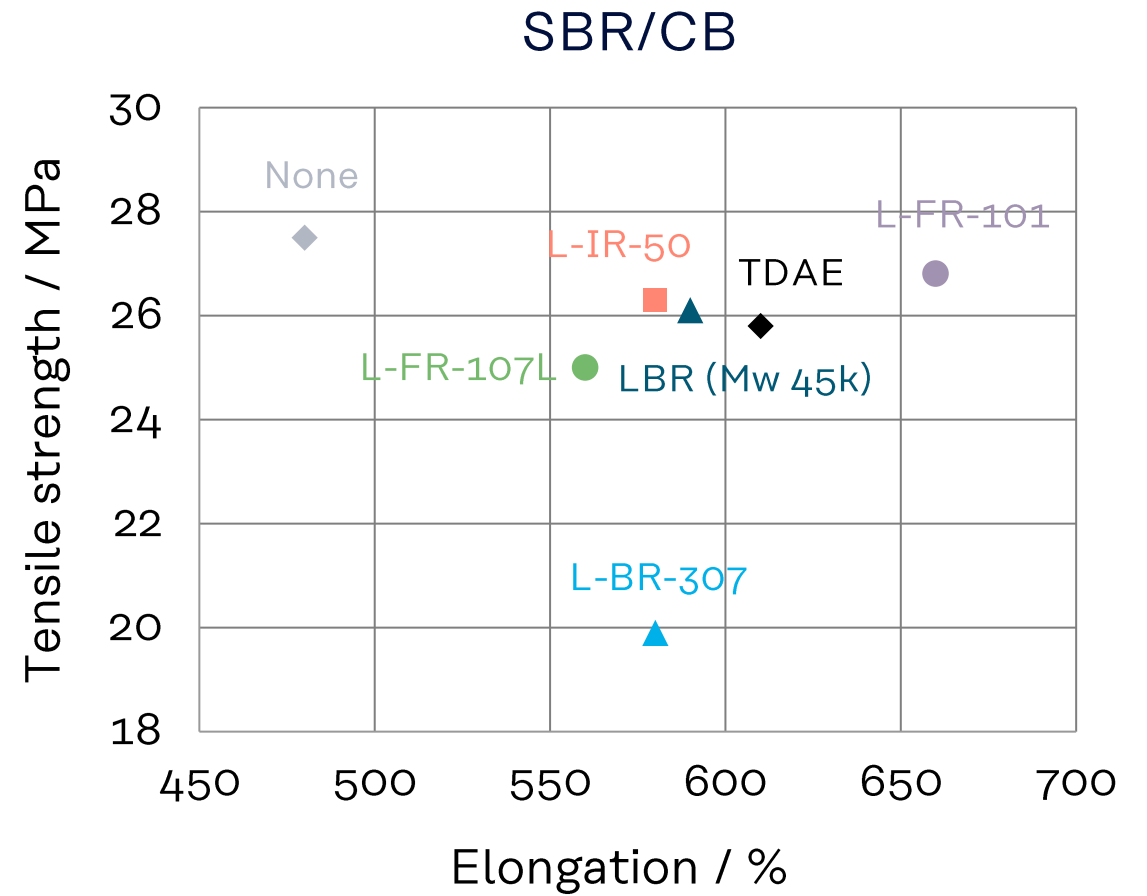
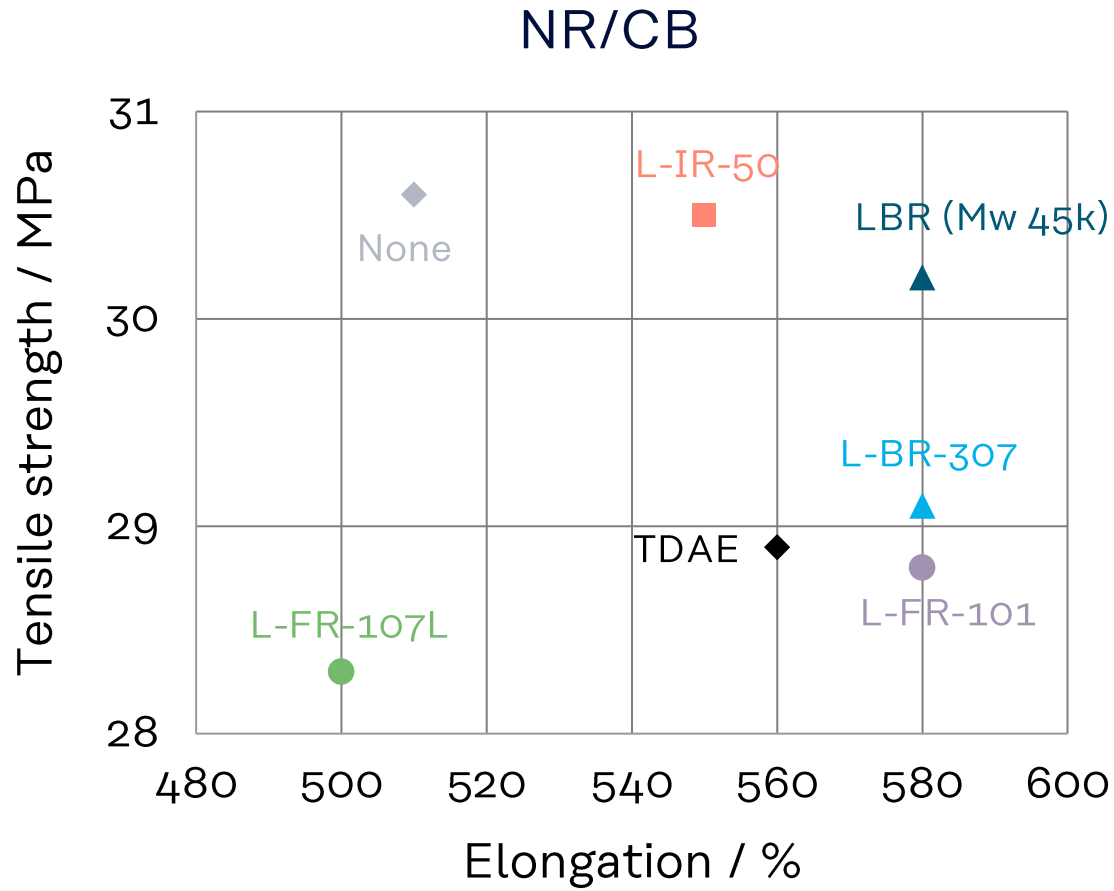
Hardness (Type A) 1



Hardness (Shore A) 2

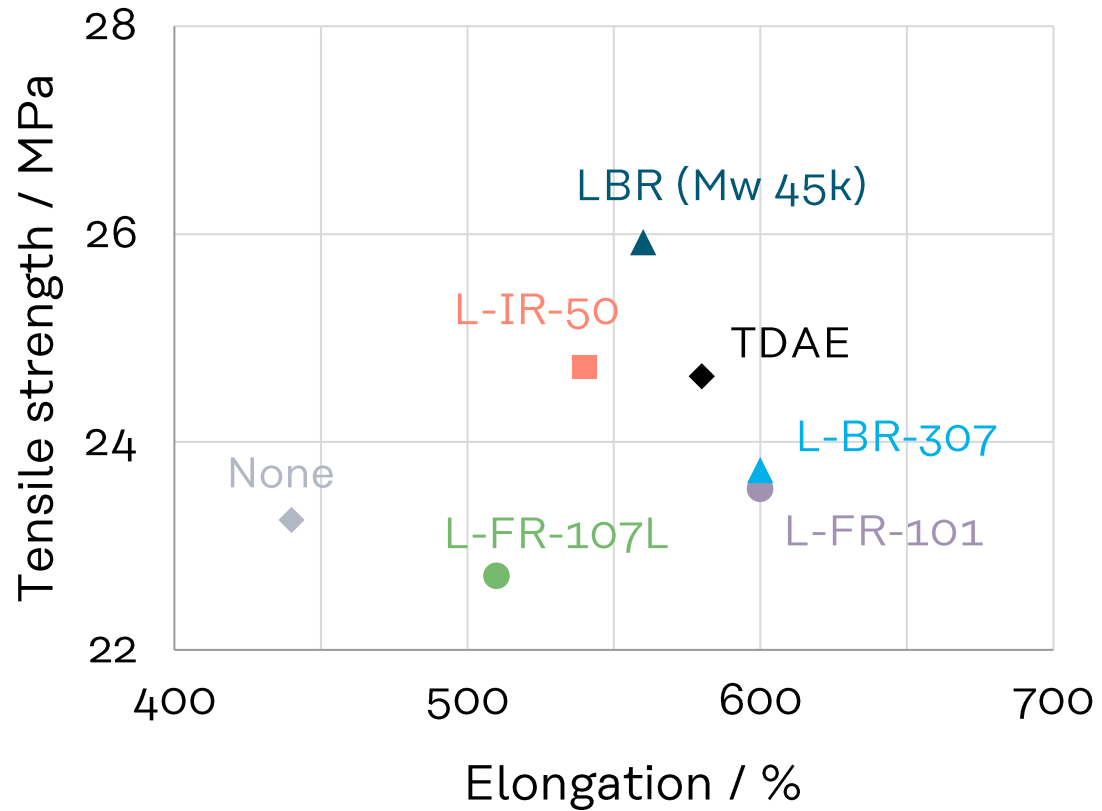


Tensile Properties

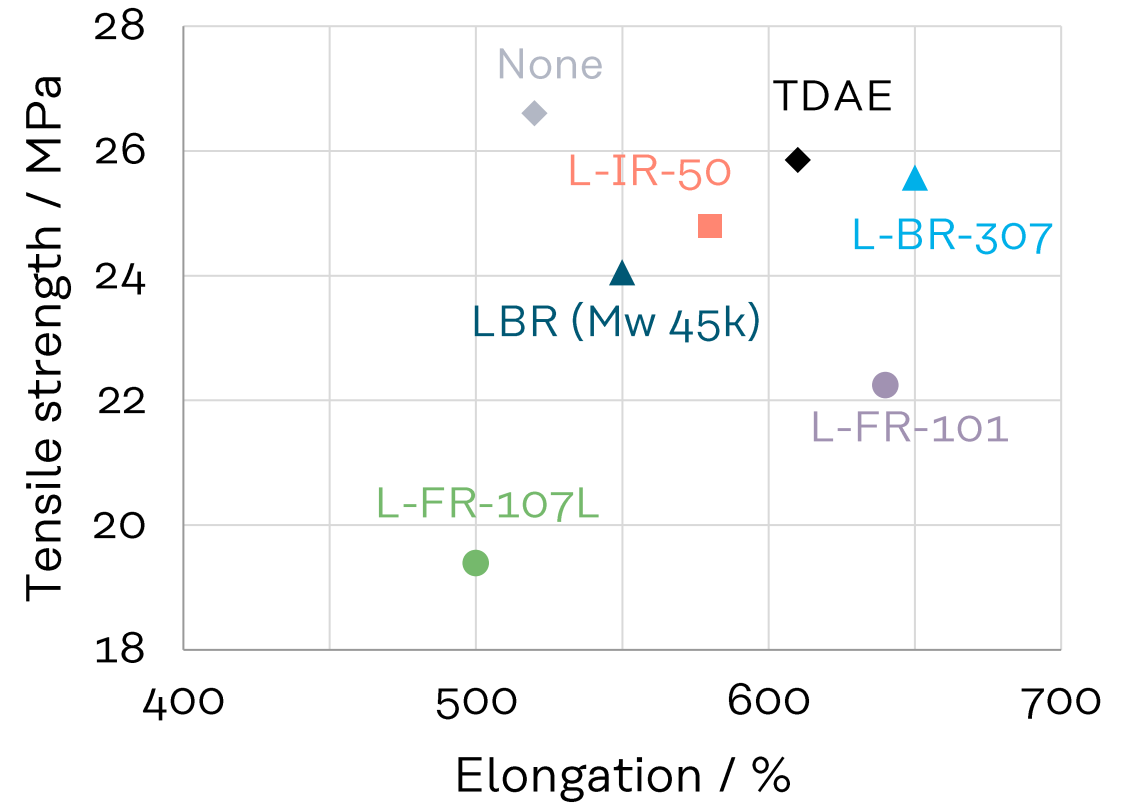


Tensile Properties

SBR/CB/Silica



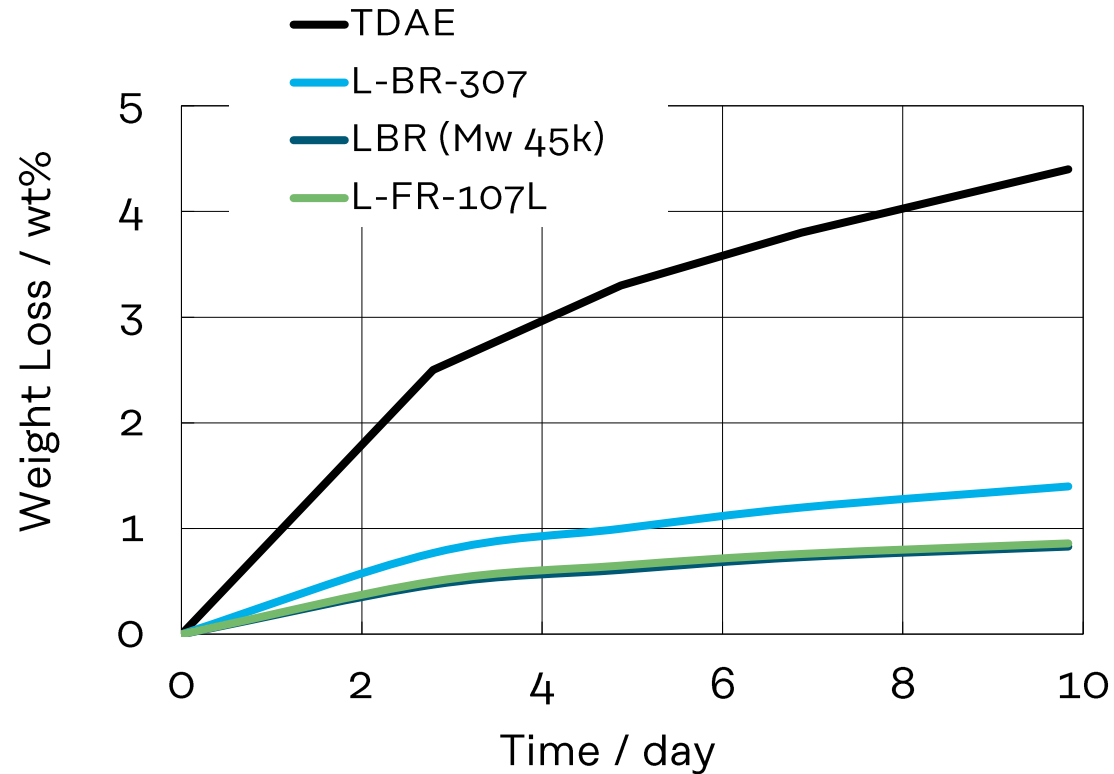
SBR/Silica



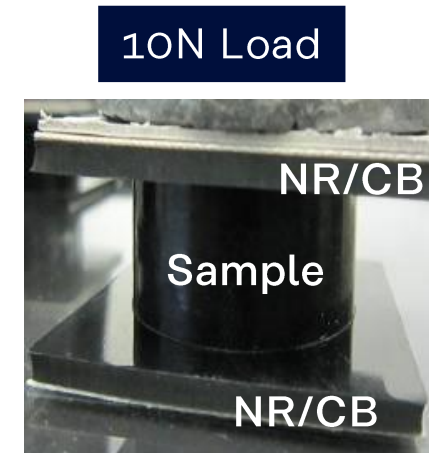
Migration Property



Good



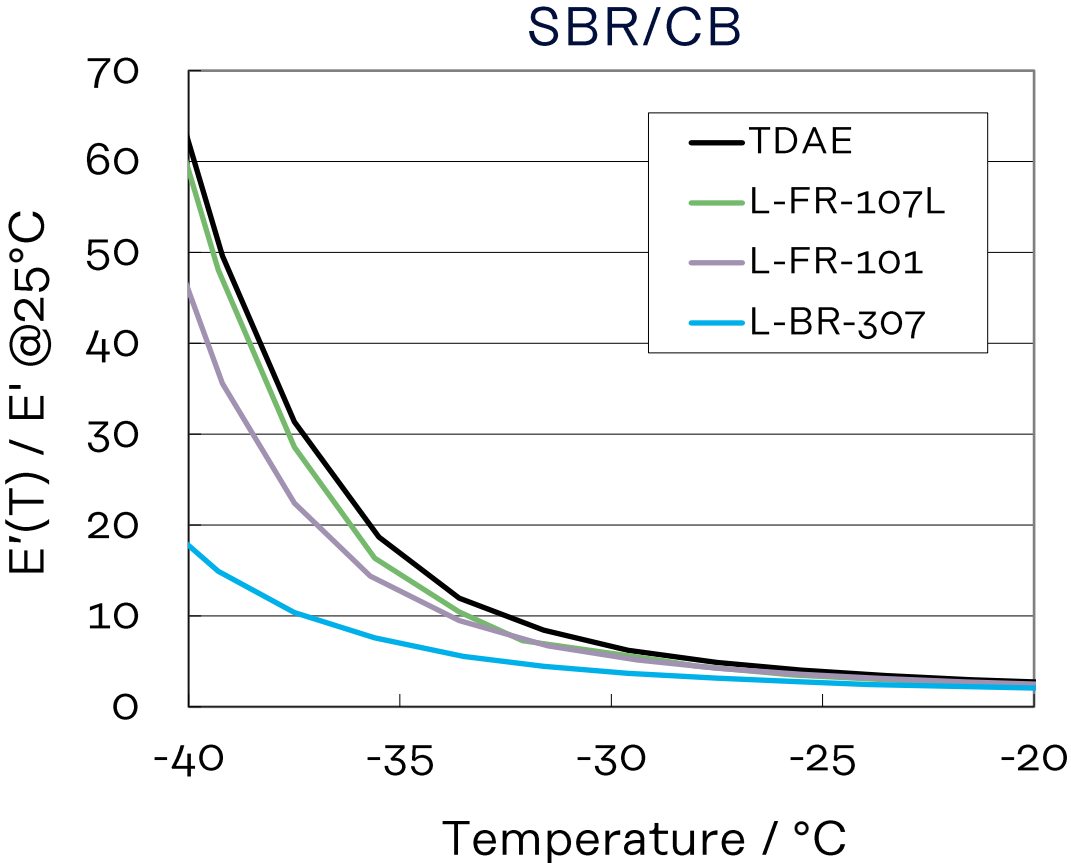
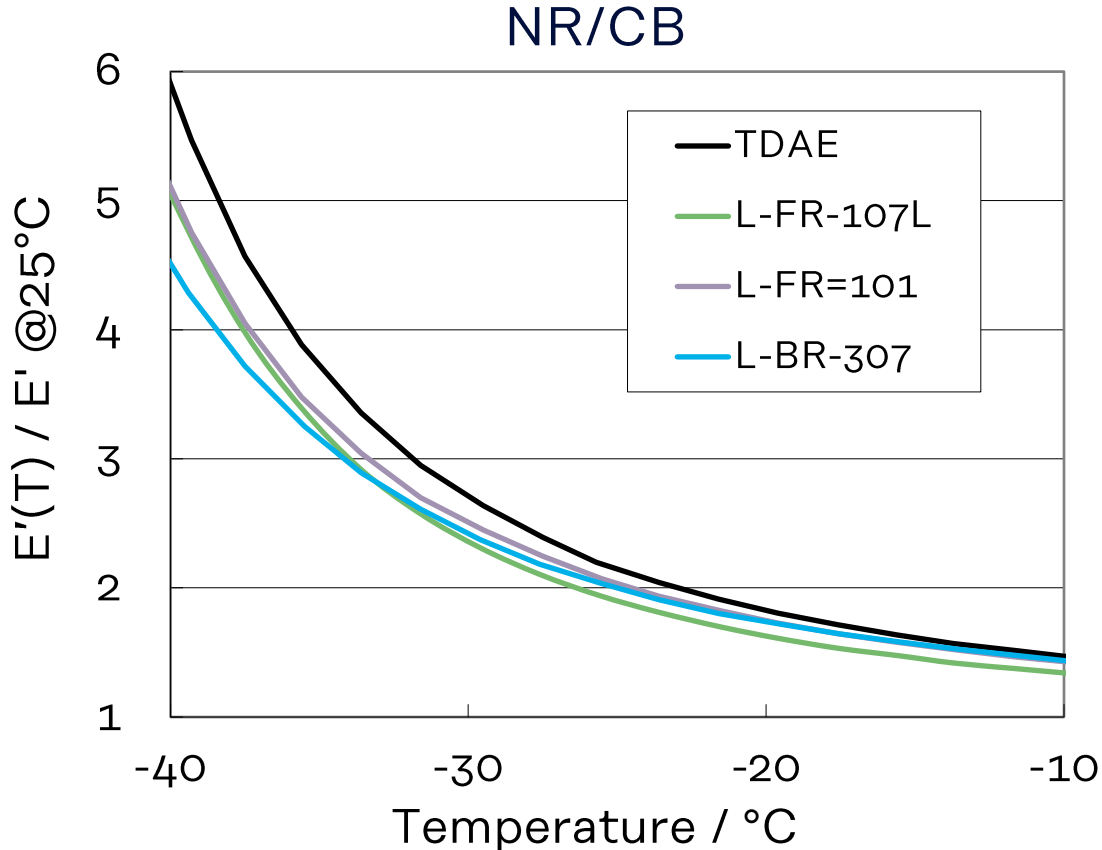
-Formulation-
SBR/CB/Plasticizer
=100/70/40
-Test condition-
Temp.: 70°C



✓ Less migration property => Expected long term performance

Low Temperature Property

-Test condition-
 Static strain:0.5%
 Dynamic Strain:0.1%
 Frequency:10Hz

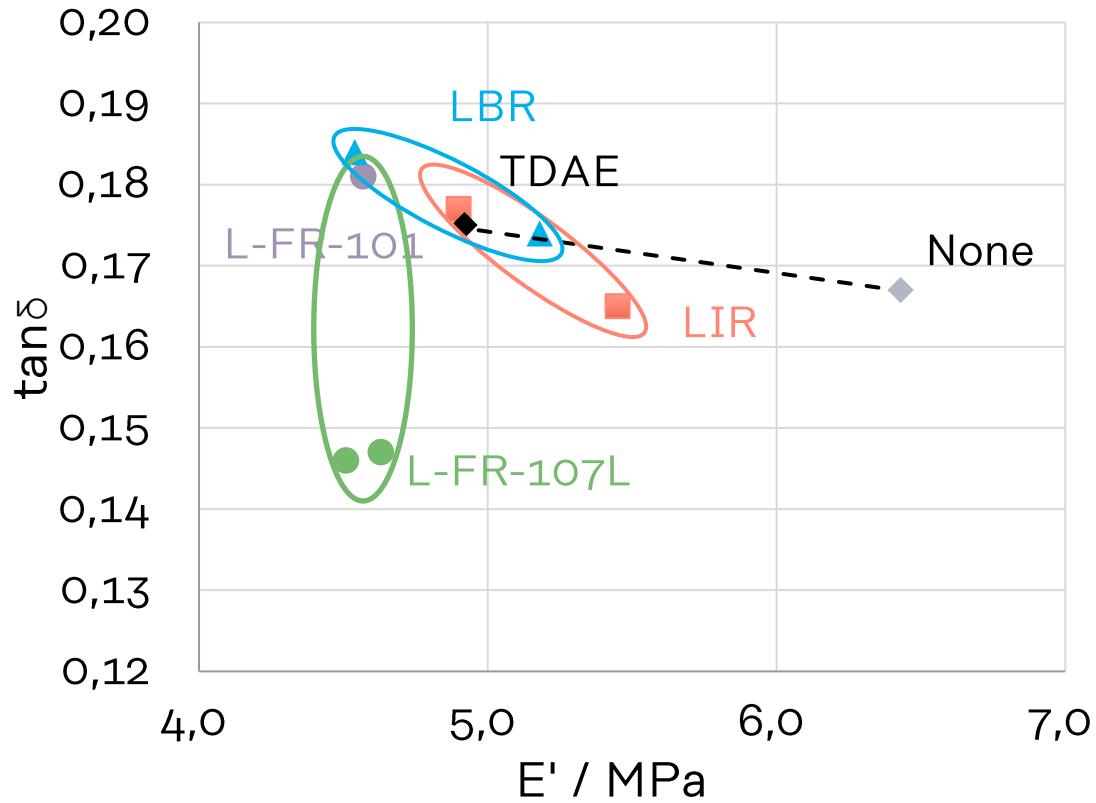


✓ Good low temperature property

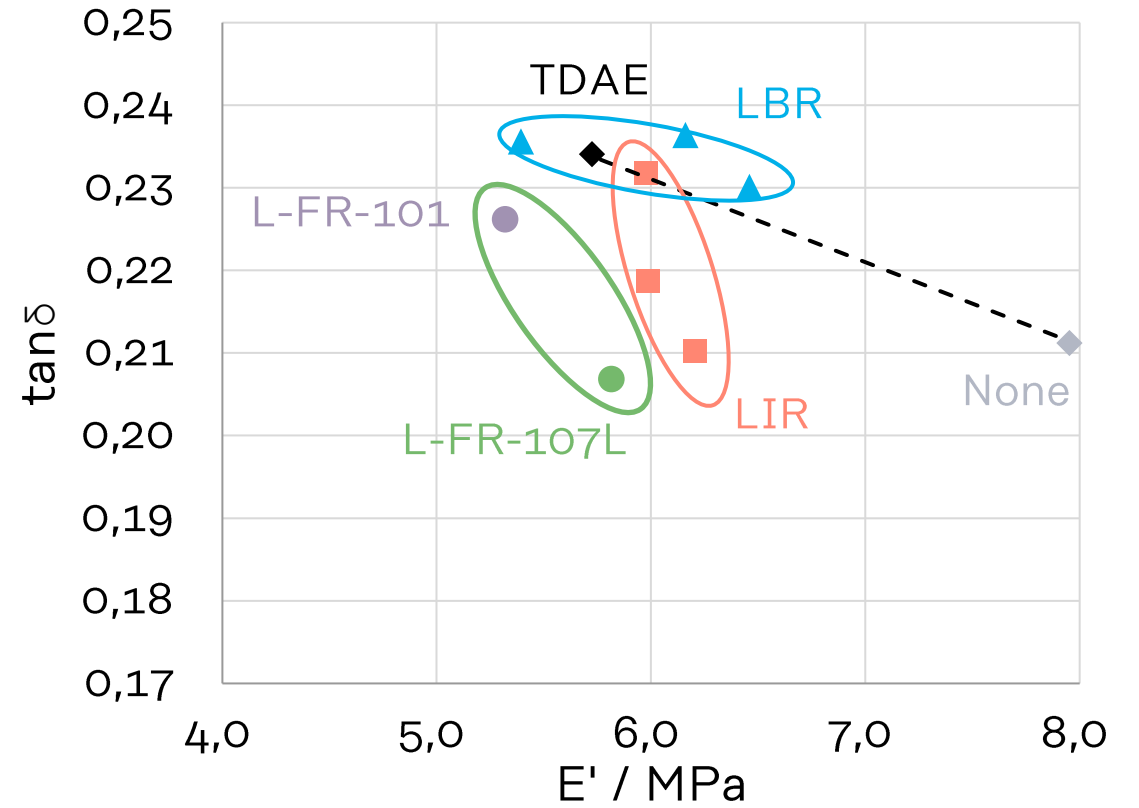
tanδ and E' at 25 °C

-Test Conditions-
 Static strain: 0.5%
 Dynamic Strain: 0.1%
 Frequency: 10Hz

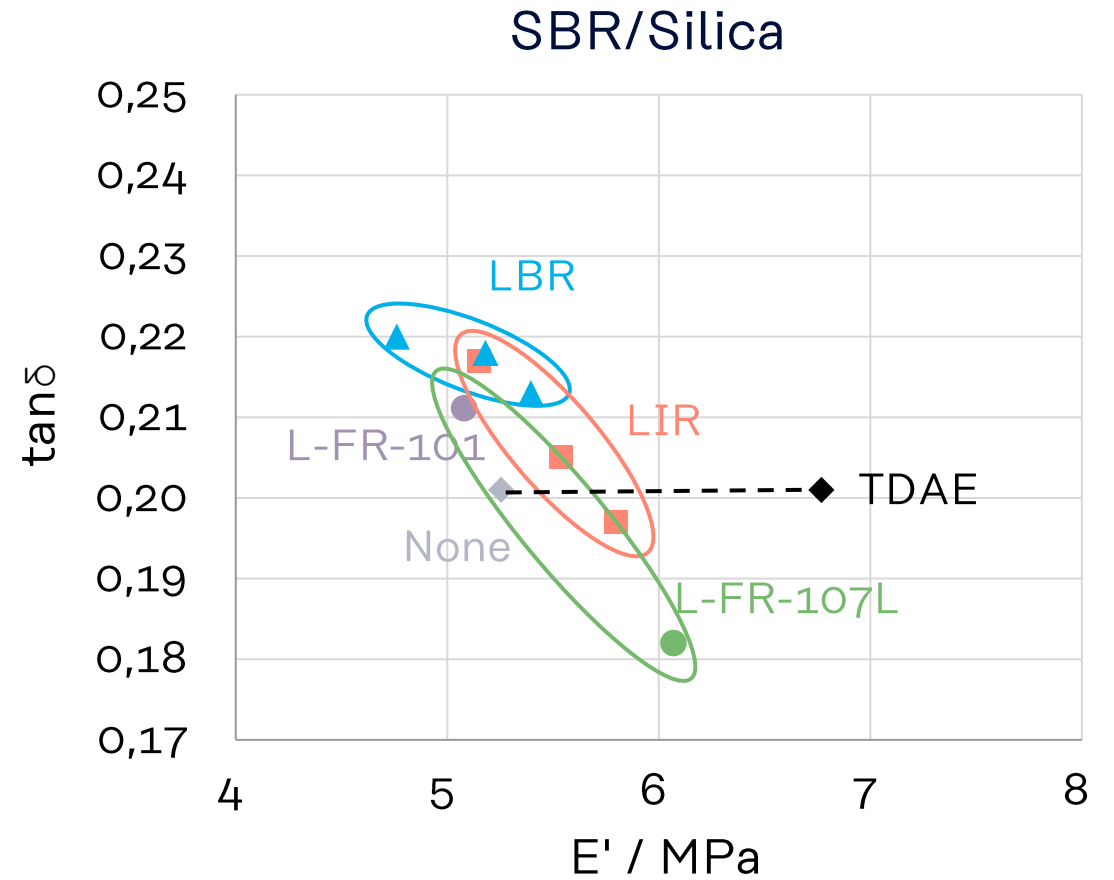
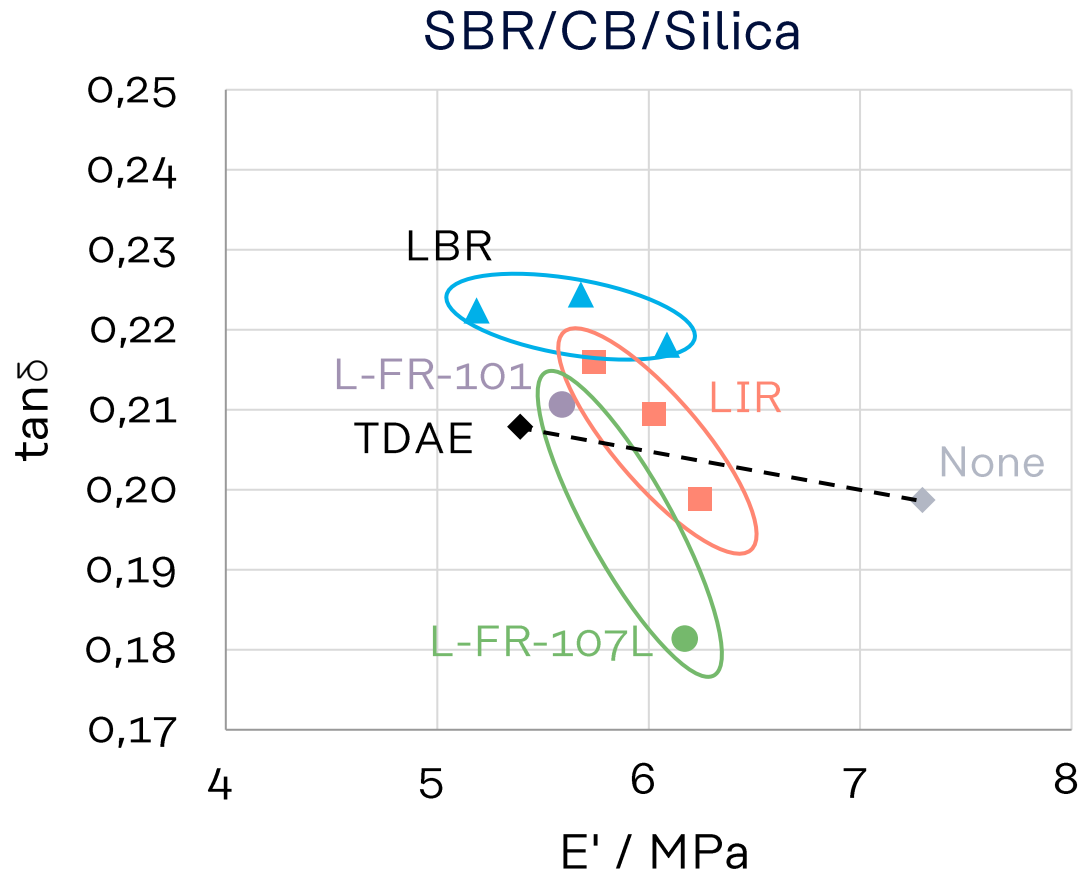
NR/CB



SBR/CB

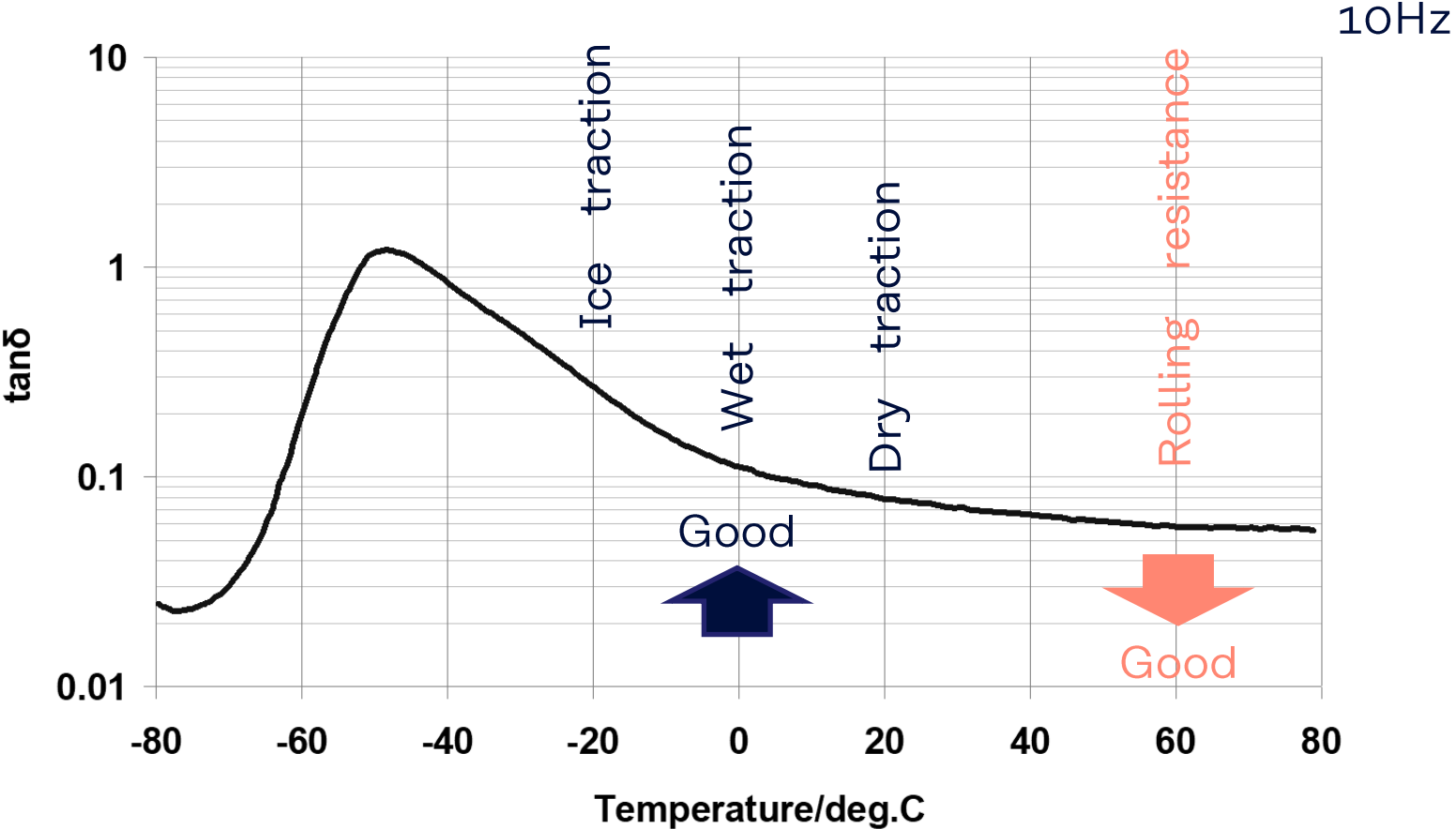


$\tan\delta$ and E' at 25 °C



Better fuel efficiency was suggested by lower $\tan\delta$.

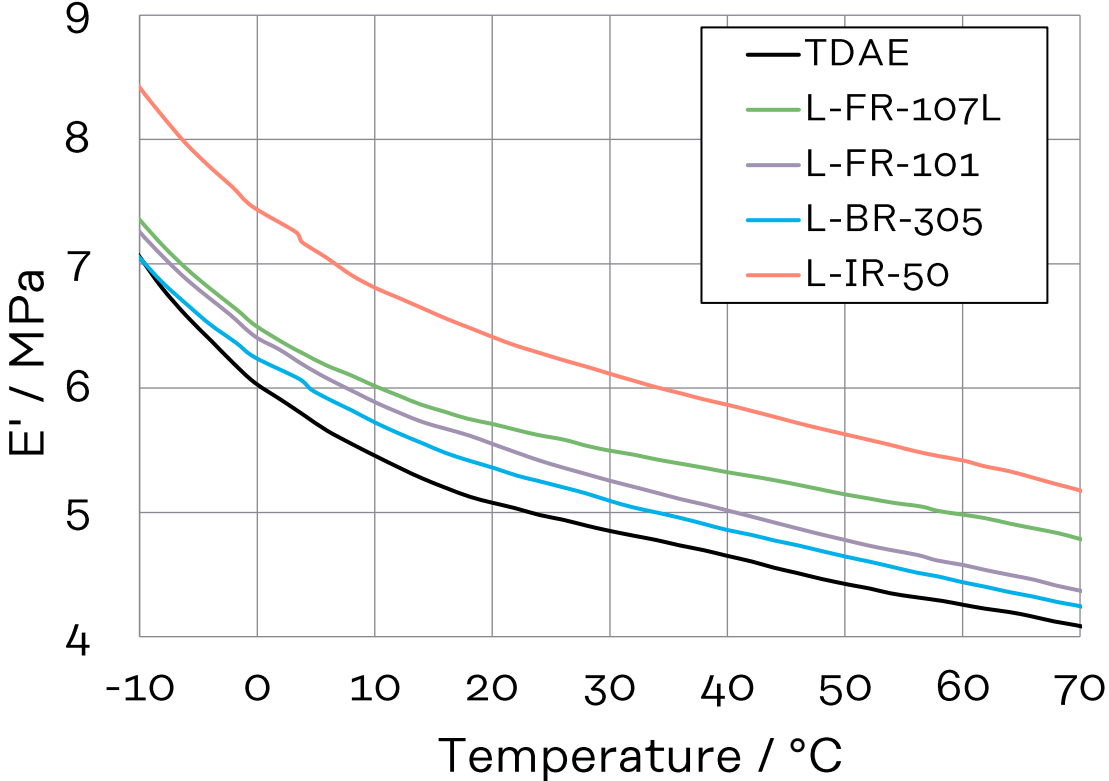
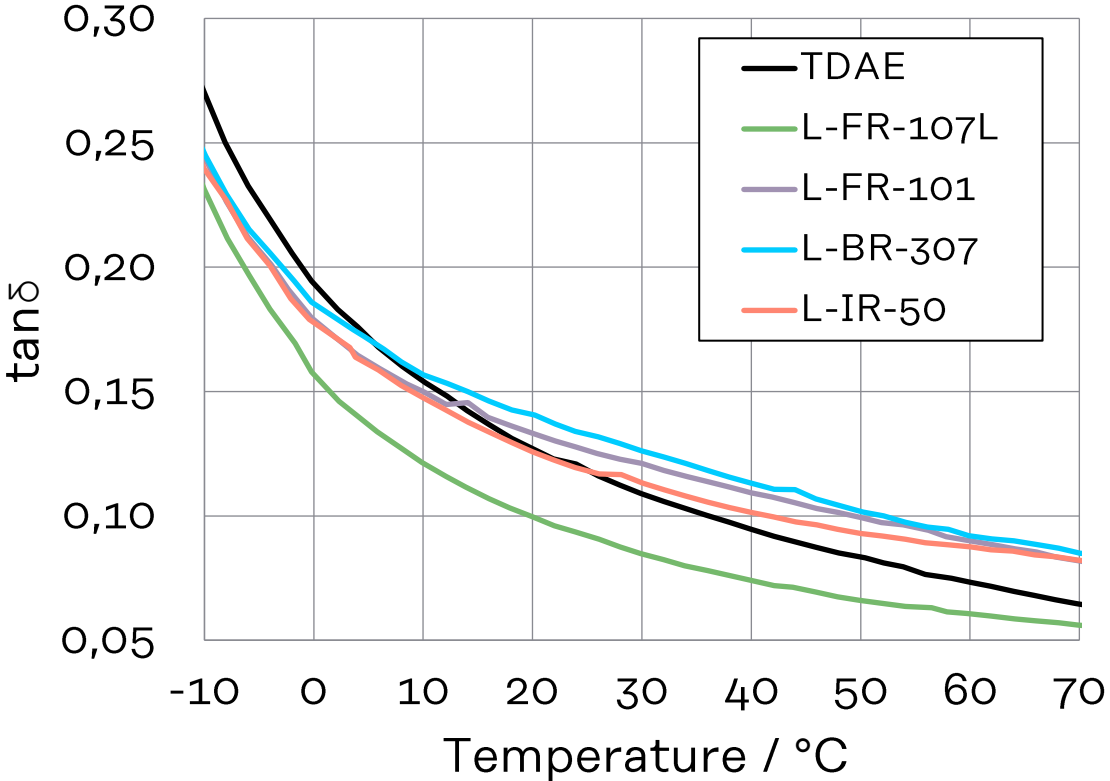
Required Performance and $\tan\delta$



Viscoelastic Property

-Test condition-
 Static strain: 10%
 Dynamic Strain: 2%
 Frequency: 10Hz

NR/CB



Better fuel efficiency was suggested by lower tan δ.

Consideration of Silica-Reinforcement Mechanism

E' of Liquid Farnesene Rubber was increased in Silica formulation compared with CB formulation. Better silica dispersion and reinforcement around silica was suggested.

Moderate Mooney viscosity of Liquid Farnesene Rubber suggest a certain interaction between silica and polymer. (Functionalized polymer increases viscosity dramatically due to too strong interaction.)

Prospected Mechanism in Silica Formulation

- 1) Silane coupling agent reacts with silica surface.
- 2) Silica and coupling agent are covered with Liquid Farnesene Rubber.
- 3) Sulfide bond on coupling agent cleaves and reacts with high branched (reactive) Liquid Farnesene Rubber rather than SBR.
- 4) And then Liquid Farnesene Rubber is co-vulcanized with SBR.
- 5) Molecular mobility of Liquid Farnesene Rubber is inhibited. Accordingly, low $\tan\delta$ was observed.

Summary

Our new polymer provides...

- 1) Bio-based
- 2) Unique structure
- 3) High plasticizing effect
- 4) Low migration property
- 5) Good low temperature traction
- 6) Improved silica dispersion and reinforcement
- 7) Better fuel efficiency

Technical Insight of KURARAY LIQUID RUBBER

High molecular weight Liquid Farnesene Rubber

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Evaluation of high molecular weight LFR

Evaluated effect of molecular weight (more than Mw=100k)

⇒Confirmed improvement of fracture resistance

LFR (Homopolymer)	Molecular weight
L-FR-107L	130k
L-FR-109*	206k
L-FR-100L*	276k
L-FR-100H*	420k
L-FR-100HH*	880k

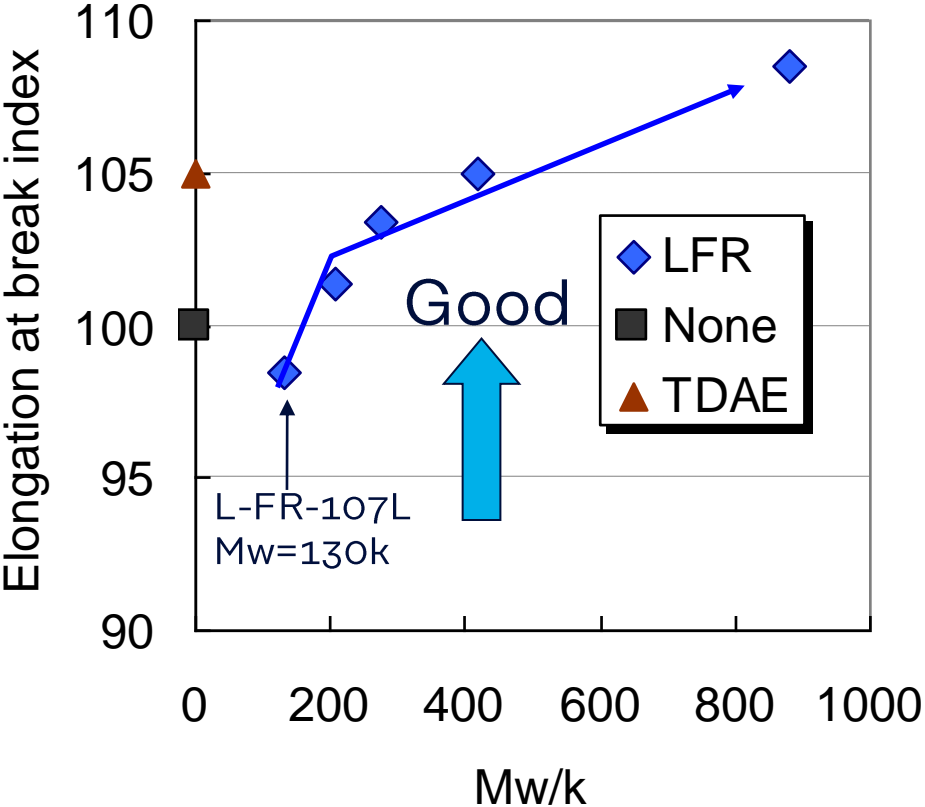
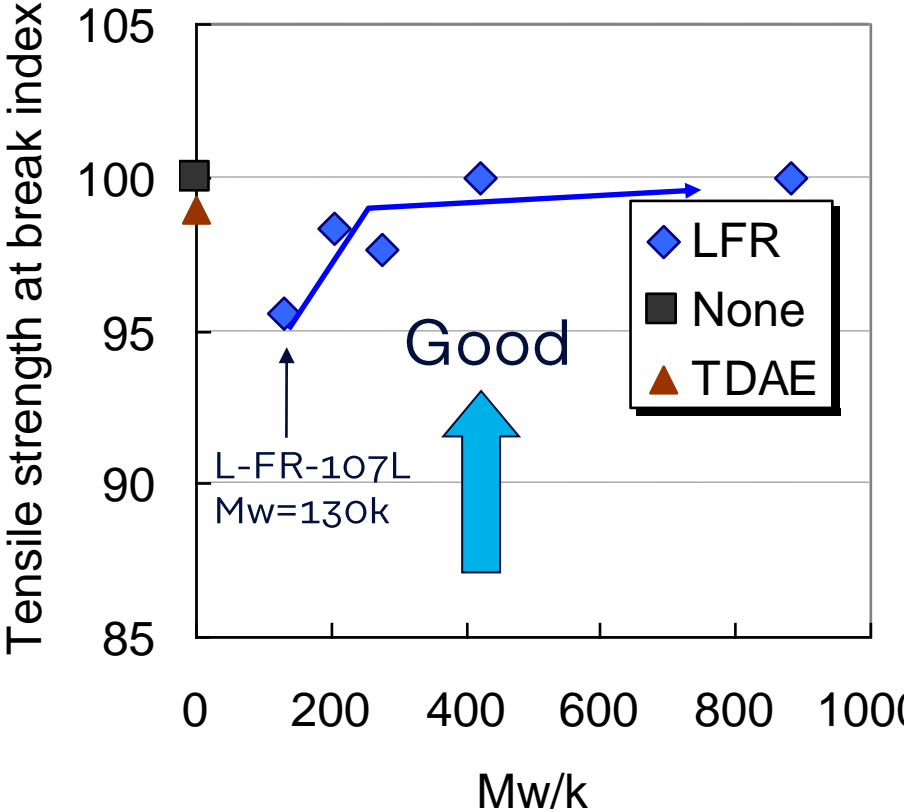
* Development Grade

⇒in NR/CB/plasticizer = 100/40/5 formulation

Evaluation summary

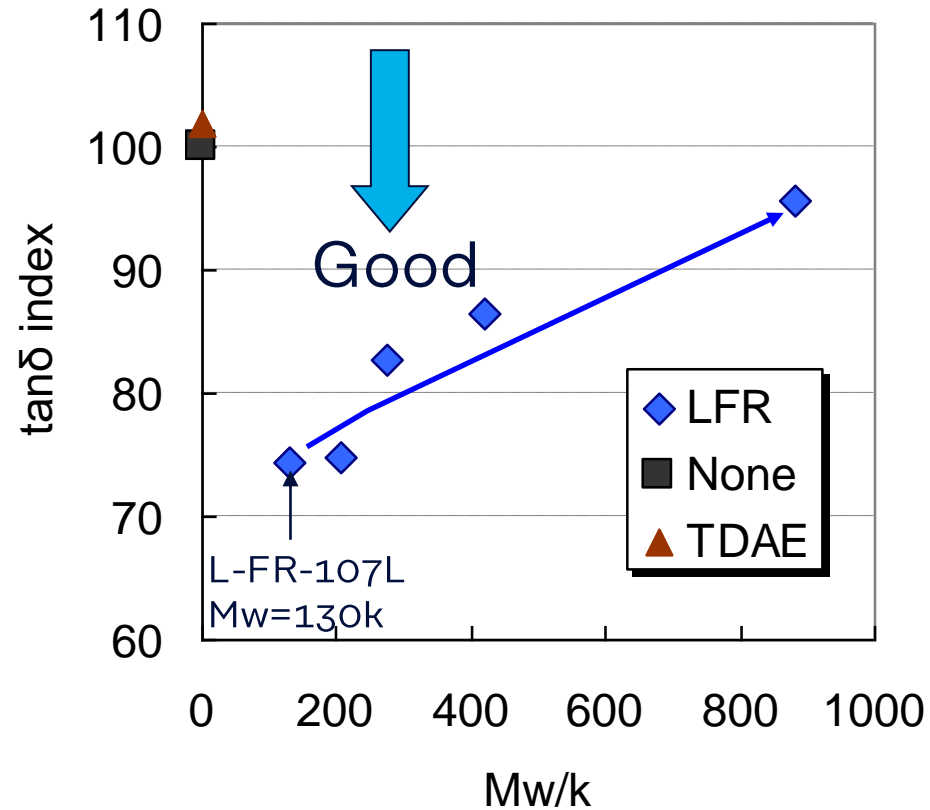
Plasticizer		None	TDAE	L-FR-107L	L-FR-109	L-FR-100L	L-FR-100H	L-FR-100HH
Mooney Vis., ML1+4 (130°C)		43	44	43	43	45	46	46
Mooney Scorch time (130°C)								
t5	(min.)	17	18	16	16	17	17	16
t35	(min.)	21	21	20	19	20	19	20
Curelastometer (145°C)								
t90	(min.)	12.5	11.9	11.9	11.8	12.0	12.0	11.9
Mechanical Properties								
Hardness	Type A	60	56	57	57	58	58	59
EB	(%)	550	530	500	510	520	530	550
TB	(MPa)	29.4	29.1	28.1	28.9	28.7	29.4	29.4
M100	(MPa)	2.5	2.2	2.2	2.2	2.2	2.2	2.2
M200	(MPa)	6.8	6.0	6.4	6.2	6.0	6.0	5.9
M300	(MPa)	13.5	11.8	12.9	12.4	12.1	12.0	11.7
Viscoelasticity (25°C)								
E' (5%)	(MPa)	4.6	4.1	3.8	3.9	4.0	4.1	4.3
tanδ (5%)	(-)	0.14	0.14	0.10	0.10	0.11	0.12	0.13
DIN abrasion	(mm ³)	126	132	134	133	135	131	133

Evaluation result



- ✓ Higher Mw ⇒ Better tensile properties
- ✓ More than Mw=200k showed good reinforcement

Evaluation result



- ✓ Less than Mw=400k ⇒ low tan δ
- ✓ Mw=200k ⇒ Same level as L-FR-107L

Summary

- More than Mw=200k showed improved fracture resistance
- Less than Mw=400k showed $\tan\delta$ reduction

⇒Especially LFR of Mw=206k showed better $\tan \delta$ reduction and fracture resistance.

APPENDIX

NR/CB Formulation

Formulation	phr
Natural Rubber (STR20)	100
Plasticizer	5
Carbon Black (N220)	40
ZnO	3.5
Stearic acid	2
Sulfur	1.5
Accelerator NS ¹⁾	1.2
Antioxidant 6C ²⁾	1
Antioxidant RD ³⁾	1

1) N-tert-Butyl-2-benzothiazolesulfenamide

2) N-(1,3-Dimethylbutyl)-N'-phenyl-p-phenylenediamine

3) Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline

Mixing Conditions

1st stage: Banbury mixer

0'00" NR (75°C)

0'20" CB, TDAE/Liquid Rubber
AO, ZnO, Stearic acid

6'00" Dump out (150-160°C)

2nd stage: Banbury mixer

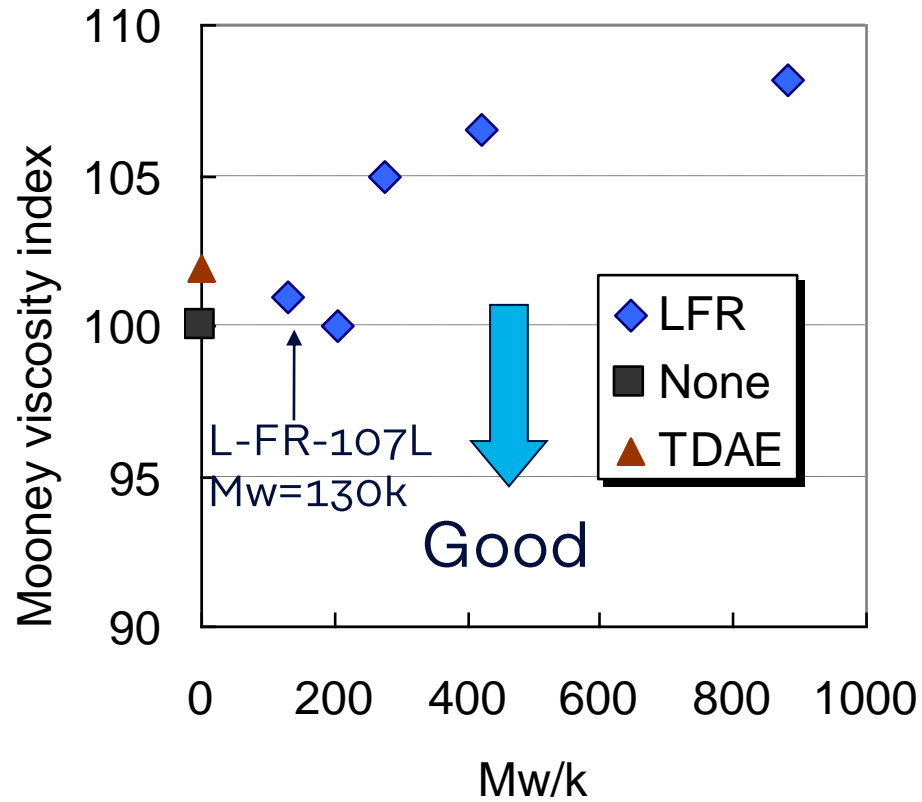
0'00" Compound Sulfur,
Accelerator (50°C)

7'00" Dump out (95-105°C)

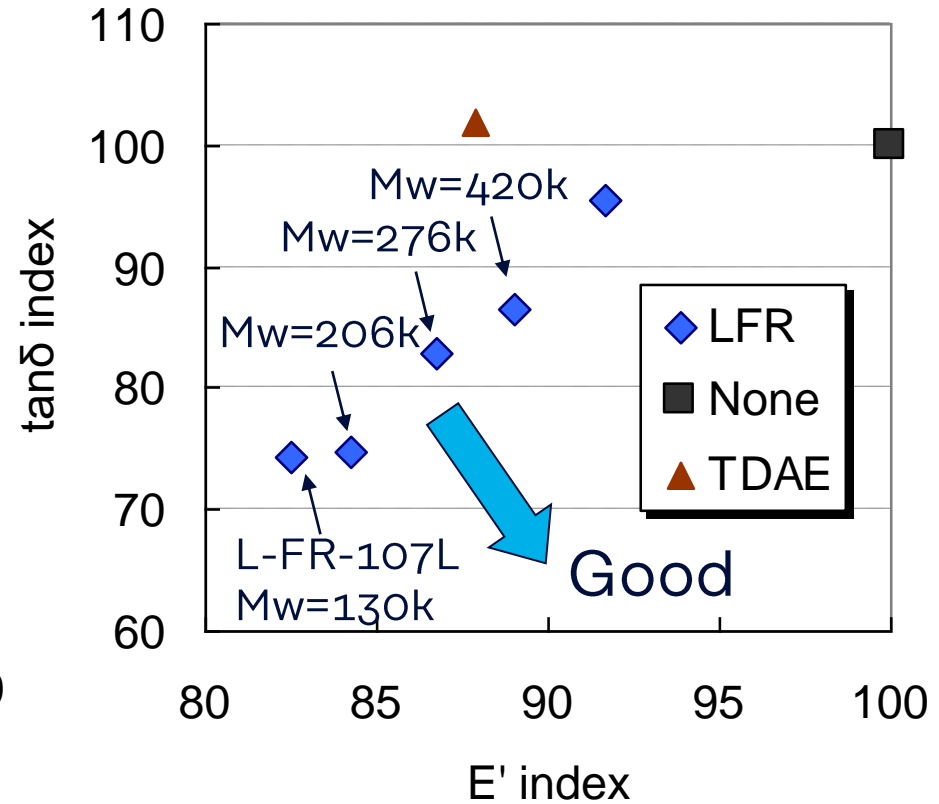
Raw materials

Material	Product Name	Manufacturer	Note
Natural Rubber	STR20	Von Bundit Co., Ltd.	
Carbon black	DIABLACK™ I	Mitsubishi Chemical Corporation	ASTM N220
TDAE	VIVATEC 500	H&R GmbH Co. KGaA	

Evaluation result

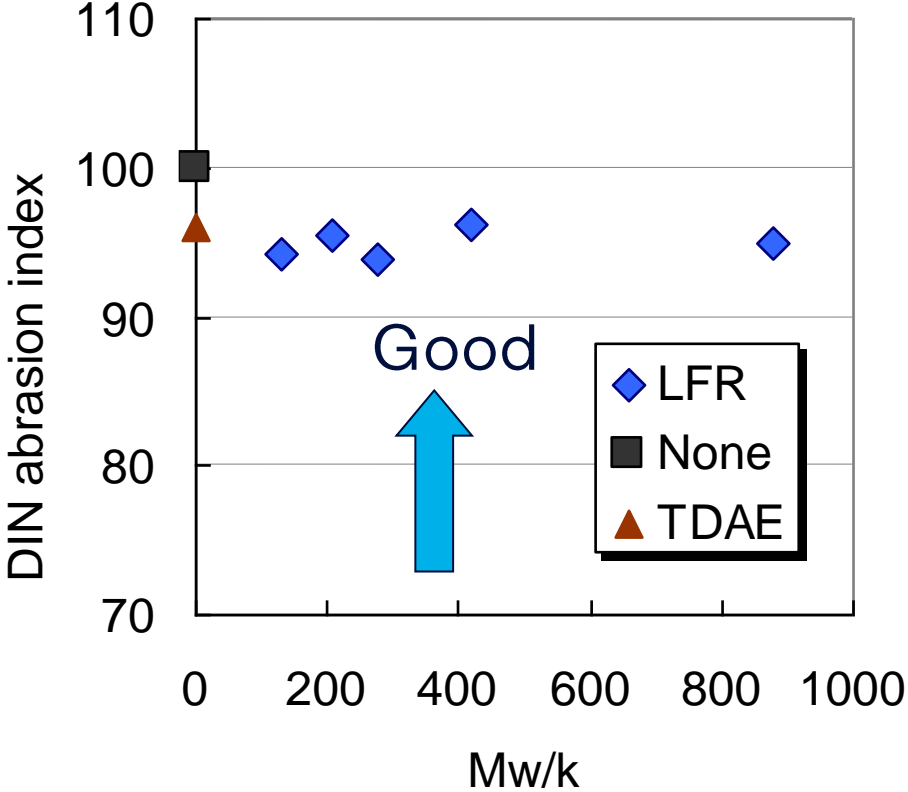


- ✓ Higher Mw ⇒ Higher viscosity
- ✓ Mw=200k ⇒ Same level as L-FR-107L



- ✓ Higher Mw ⇒ Better reinforcement
- ✓ Mw=200k ⇒ Better than L-FR-107L

Evaluation result



Technical Insight of KURARAY LIQUID RUBBER

Influence of molecular weight and addition amount

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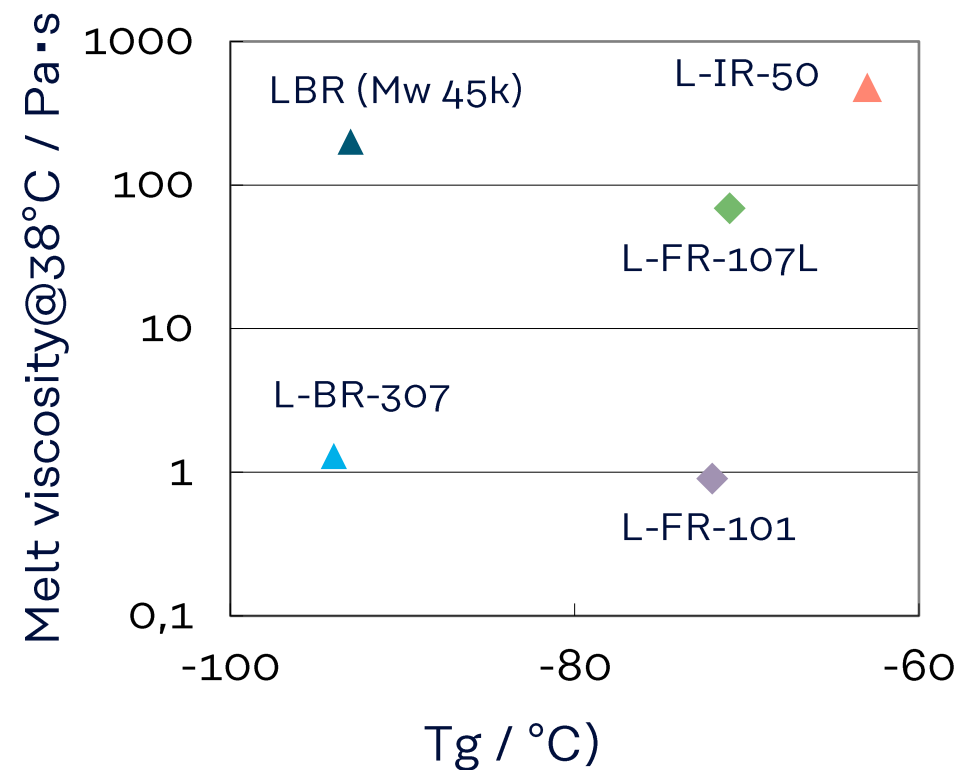
Agenda

- Introduction of Bio-based Liquid Rubber
- Influence of Molecular Weight in NR/CB Formulation
- Influence of Added Amount in NR/CB Formulation

Grade List

Grade	Structure	Tg (°C)	Mw	Visc. @38°C (Pa.s)
L-FR-101*	polyfarnesene	-73	9k	0.4
L-FR-107L	polyfarnesene	-71	135k	69
L-BR-307	polybutadiene	-94	8k	1.3
LIR-50	polyisoprene	-63	54k	480

*Development Grade



NR/CB Formulation

<u>Formulation</u>	<u>phr</u>
Natural Rubber (TSR20)	100
TDAE ¹⁾ or Liquid Rubber	10
Carbon Black (N330)	50
ZnO	3.5
Stearic acid	2
Sulfur	1.5
Accelerator NS ²⁾	1
Anti oxidant 6C ³⁾	1

1) VIVATEC 500 (H&R GmbH Co. KGaA)

2) N-tert-butyl-2-benzothiazolesulfenamide

3) N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine

Mixing Conditions

1st stage: Banbury mixer

0'00" NR (85°C)

0'20" CB, TDAE/Liquid Rubber
AO, ZnO, Stearic acid

6'00" Dump out (150-160°C)

2nd stage: Roll mill

0'00" Compound (55-60°C)

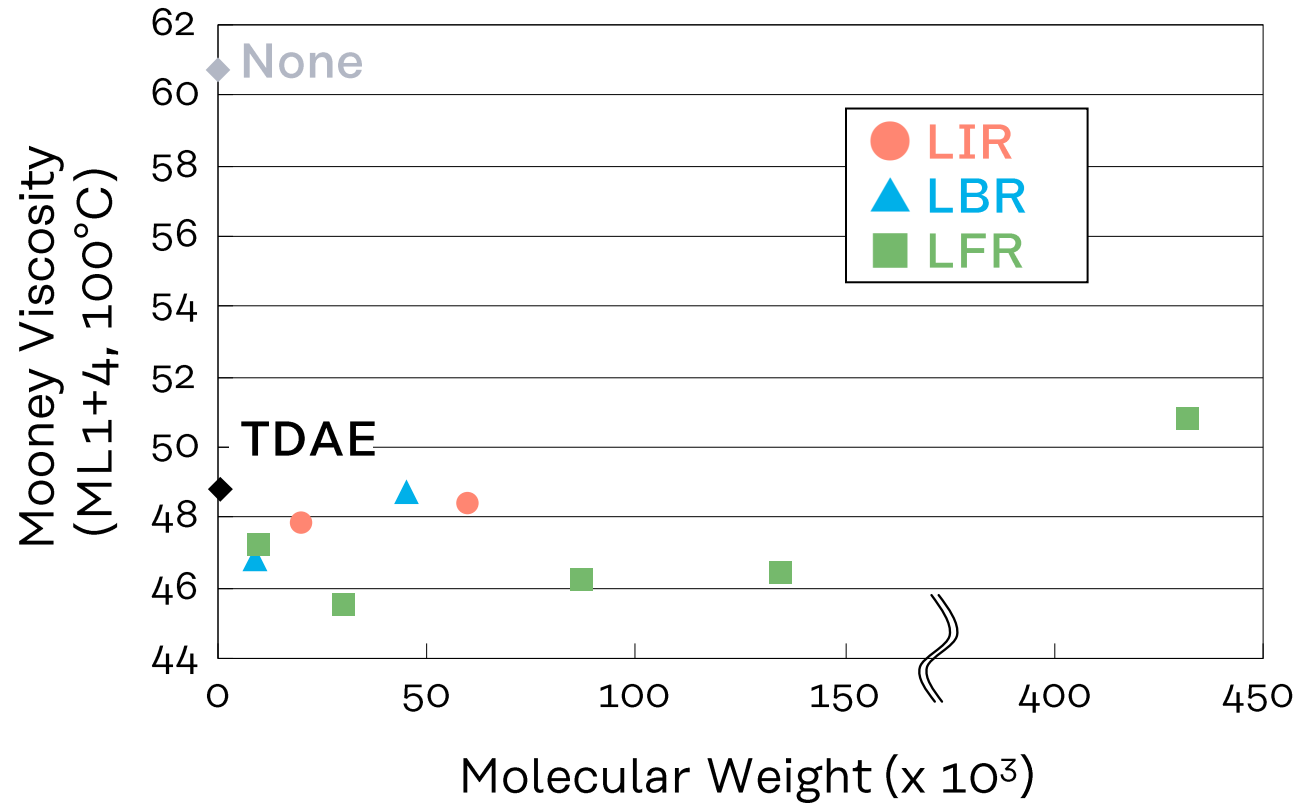
0'30" Sulfur, Accelerator

7'00" Sheeting

NR/CB Formulation Summary

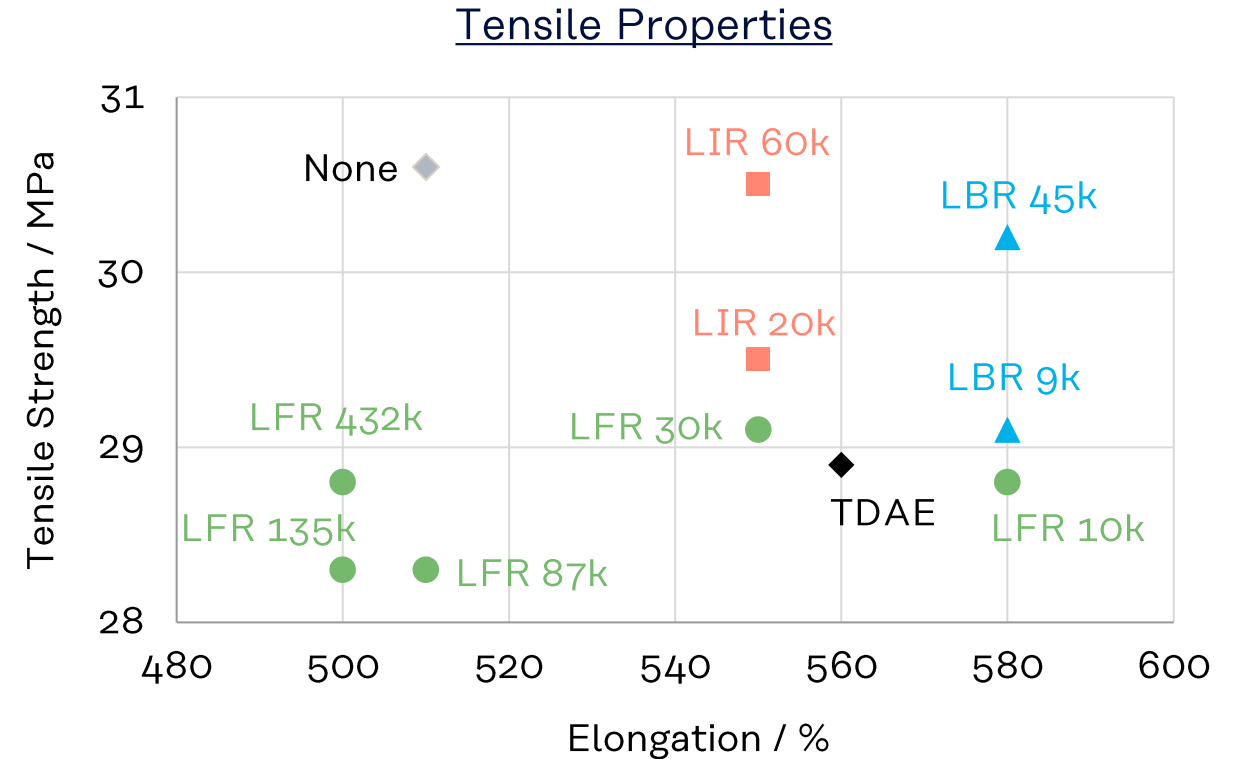
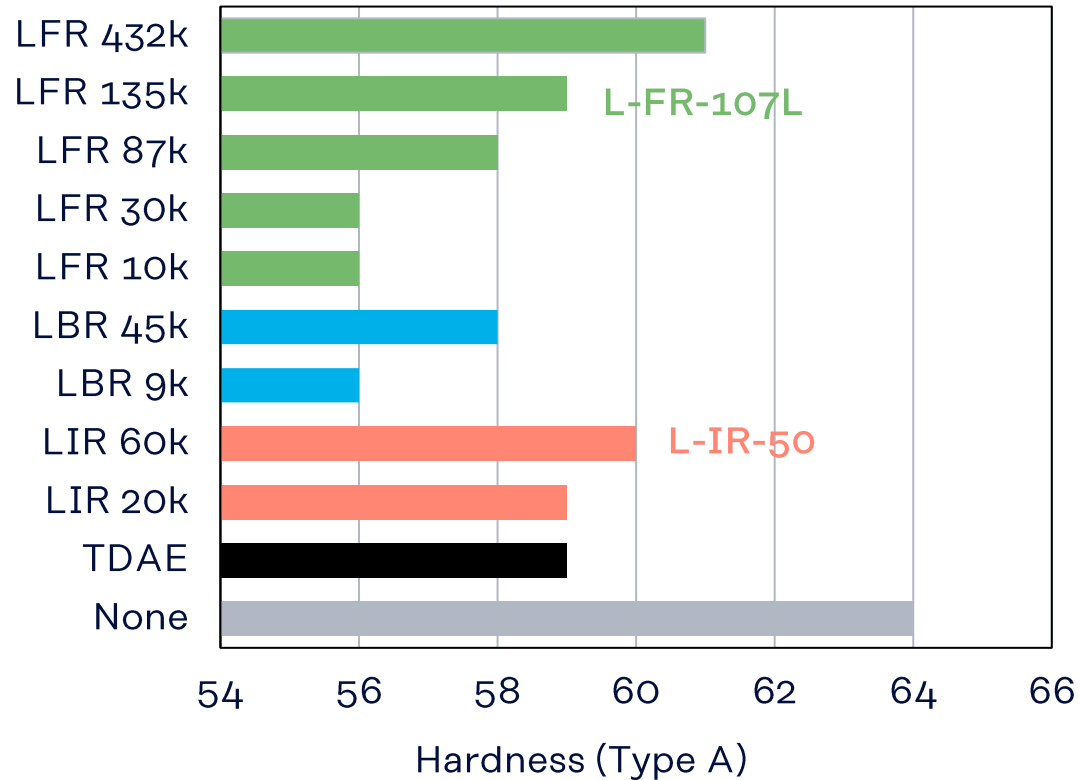
		1	2	3	4	5	6	7	8	9	10	11
Plasticizer		None	TDAE	LIR 20k	LIR 60k (L-IR-50)	LBR 9k (L-BR-307)	LBR 45k	LFR 10k (L-FR-101)	LFR 30k	LFR 87k	LFR 135k (L-FR-107L)	LFR 432k
Mooney Vis. ML1+4 (100°C)		60.7	48.8	47.8	48.4	46.8	48.7	47.2	45.5	46.2	46.4	50.8
Curelastometer (145°C)	t90	11.1	12.1	11.7	11.4	11.4	11.3	10.4	10.2	11.5	10.3	10.6
Bound Rubber	%	35.4	35.9	35.4	36.8	35.8	37.6	37.7	37.7	38.7	39.4	38.9
Toluene extraction	%	2.84	8.46	3.99	3.01	6.78	3.04	5.38	3.23	3.07	2.94	2.97
Mechanical Properties												
Hardness	Type A	64	59	59	60	56	58	56	56	58	59	61
EB	(%)	510	560	550	550	580	580	580	550	510	500	500
TB	(MPa)	30.6	28.9	29.5	30.5	29.1	30.2	28.8	29.1	28.3	28.3	28.8
M100	(MPa)	3.11	2.34	2.34	2.31	2.07	2.27	2.16	2.26	2.27	2.32	2.38
M300	(MPa)	16.4	11.3	12.3	12.6	11.1	12.3	11.5	12.1	13.9	13.5	13.5
Viscoelasticity (25°C)												
5% E'		6.43	4.29	4.90	5.45	4.54	5.18	4.57	4.67	4.51	4.63	5.37
5% tanδ		0.167	0.175	0.177	0.165	0.184	0.174	0.181	0.163	0.146	0.147	0.153

Mooney Viscosity



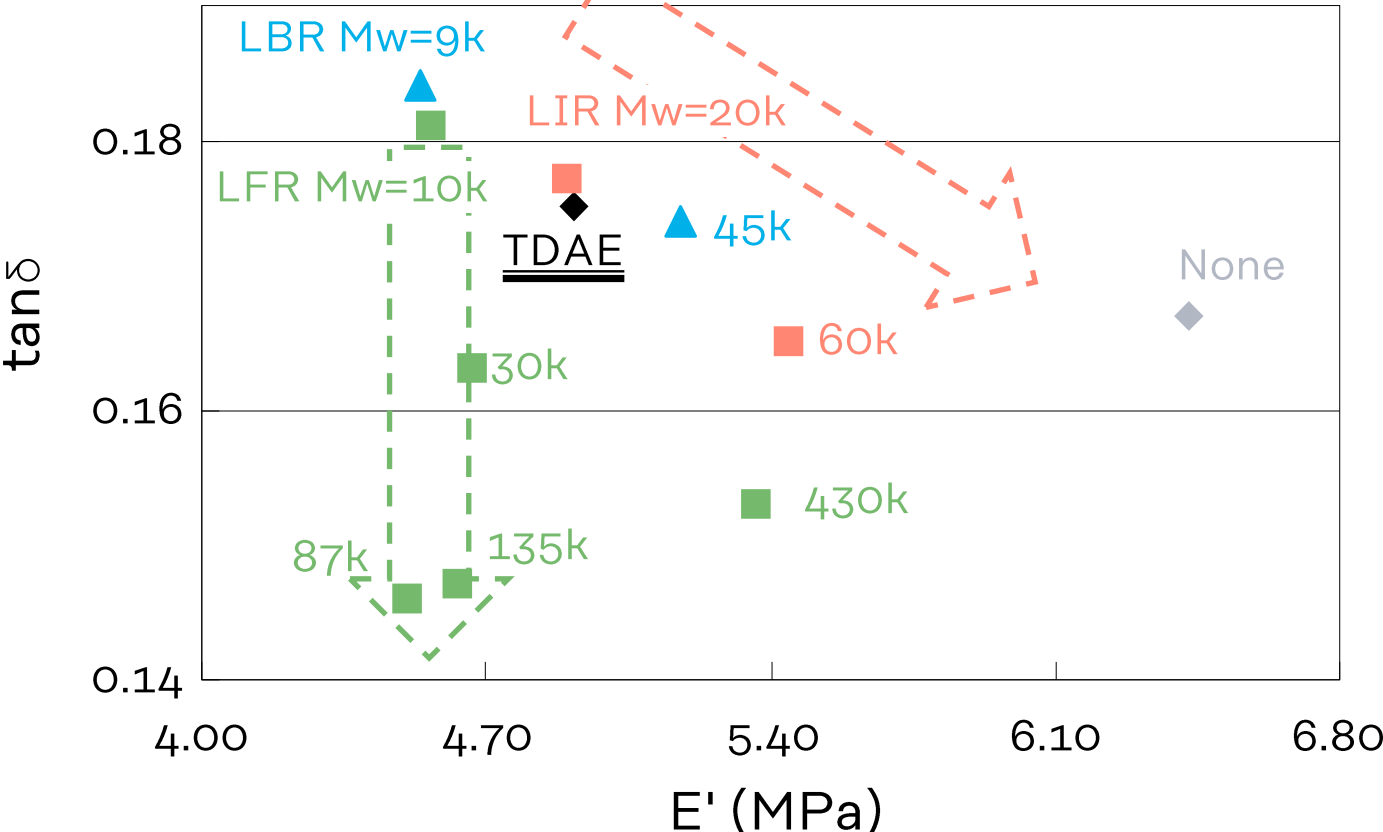
- ✓ Liquid Farnesene Rubber shows better plasticizing effect than TDAE.
- ✓ Too high Mw deteriorates plasticizing effect.

Vulcanized Properties



- Tensile property of low MW liquid rubbers were similar to TDAE.
- High MW LIR and LBR showed higher strength.
- High MW Liquid Farnesene Rubber deteriorated elongation.

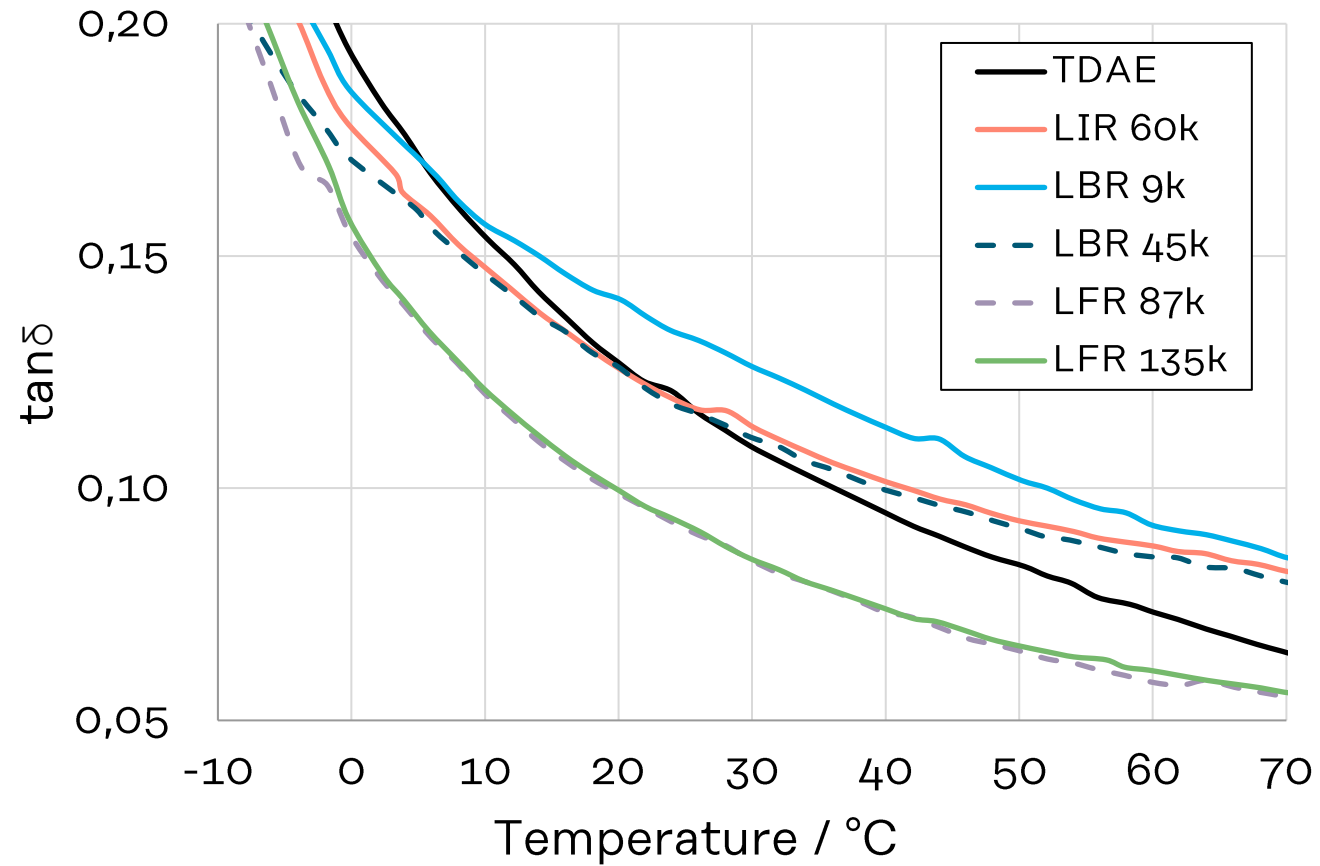
Dynamic Mechanical Analysis



-Test condition-
 Temp: 25°C
 Static strain: 15%
 Dynamic Strain: 5%
 Frequency: 10Hz

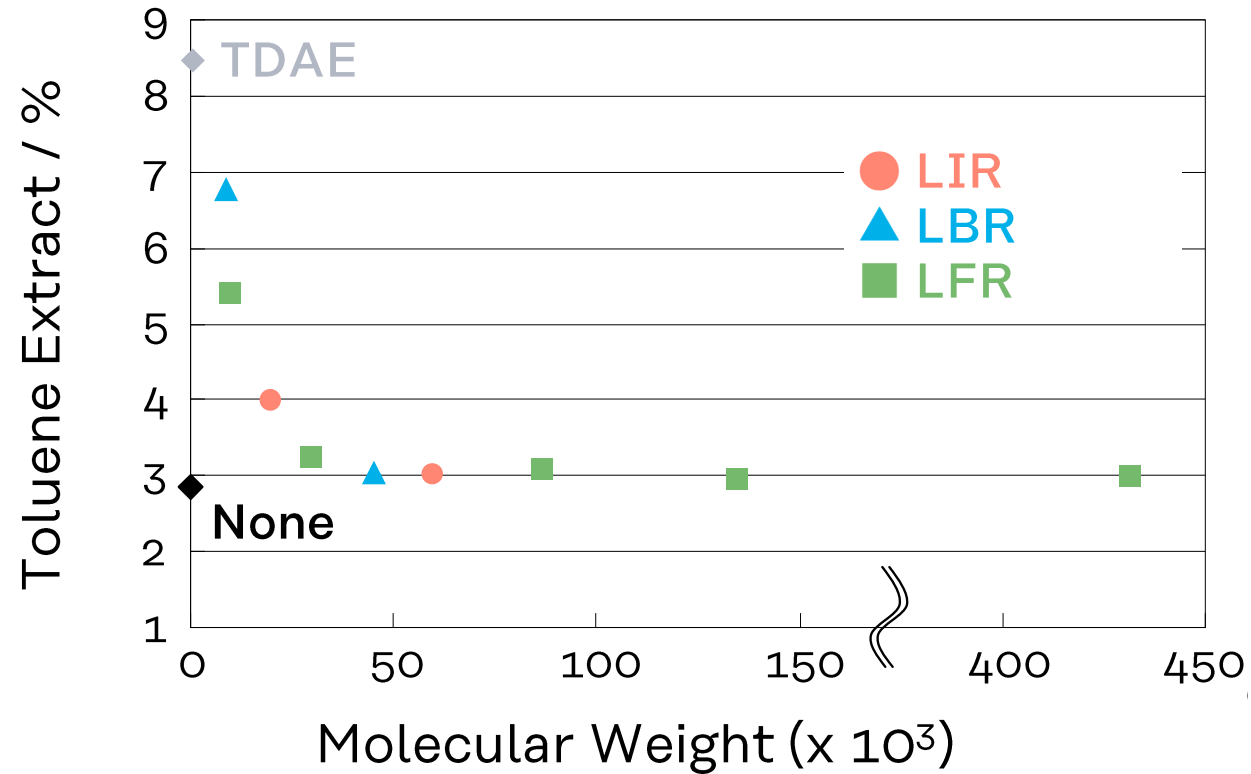
- ✓ Higher Mw Liquid Farnesene Rubber tends to reduce tan δ.
- ✓ The tendency is saturated around Mw=100k.

Viscoelastic Data (Temp Sweep)




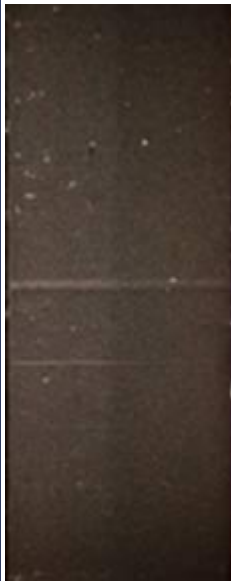


✓ L-FR-107L reduced $\tan\delta$ over wide range of temp.

Toluene Extraction Test



- ✓ Liquid Rubber having Mw of 30,000 or higher was not extracted.
- ✓ Higher Mw Liquid Farnesene Rubber never bleeds out, unlike oil.

CB Macro-Dispersion

Sample	TDAE	LIR 60k LIR-50	LBR 9k	LBR 45k	LFR 10k	LFR 135k L-FR-107L
Cross-section						
Dispersion	Good	Good	Poor	Excellent	Fair	Excellent

- ✓ Cross-section of vulcanized sheet was observed by micro-scope.
- ✓ L-FR-107L gave excellent CB dispersion.

NR/CB Formulation Summary

Features of L-FR-107L

- Good plasticizing effect
- Low $\tan\delta$
- High reactivity
 - => Co-vulcanization with base rubber
 - => Inhibit molecular mobility of L-FR-107L
 - => Not increase $\tan\delta$
- No Bleeding & No Migration
 - => $M_w=30k$ or higher is co-vulcanizable
 - => Long term reliability

Agenda

- Introduction of Bio-based Liquid Rubber
- Influence of Molecular Weight in NR/CB Formulation
- Influence of Added Amount in NR/CB Formulation

Objective

L-FR-107L reduces $\tan\delta$ in NR/CB formulation.

However, too much addition of L-FR-107L may deteriorate physical properties.

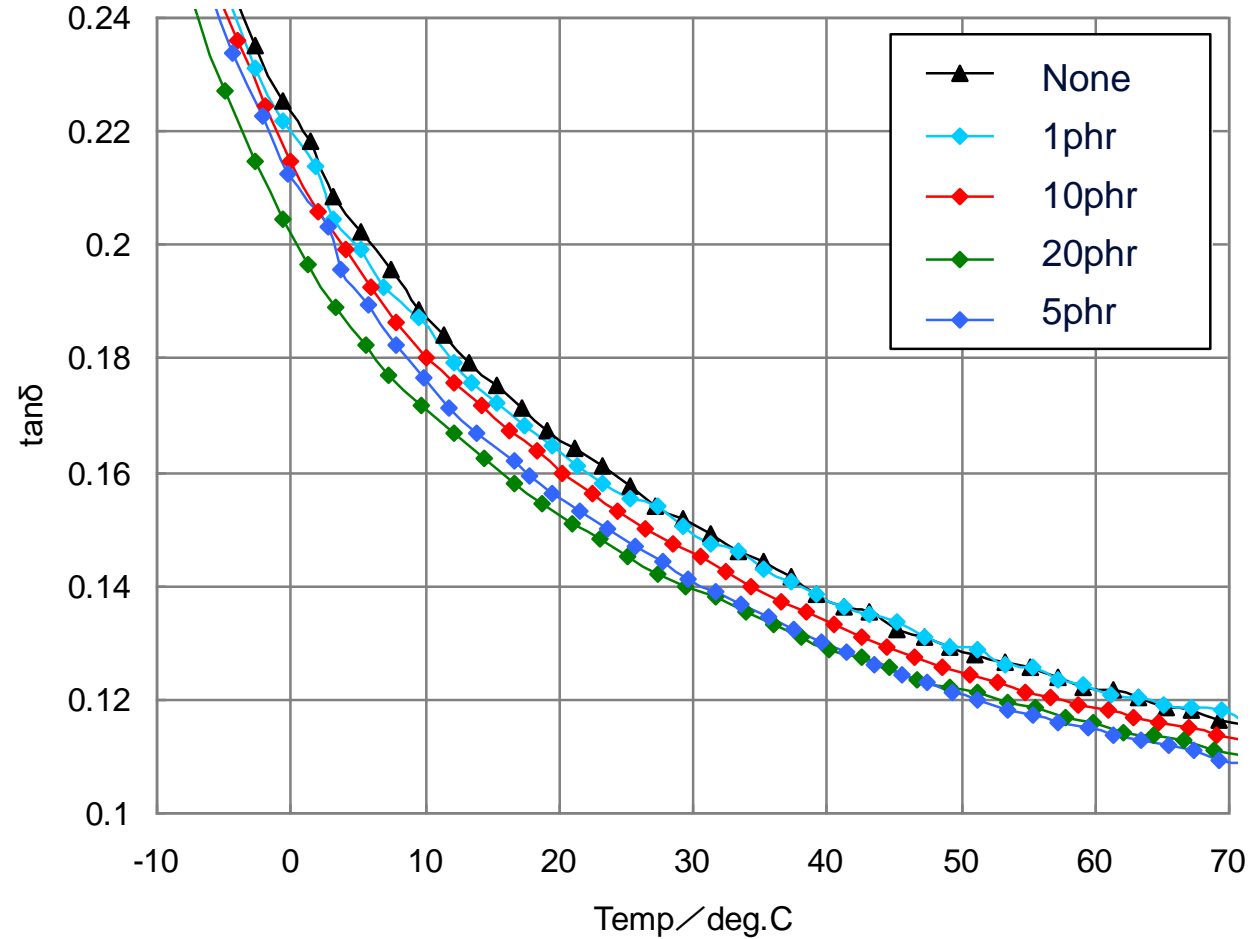
=> Influence of L-FR-107L amount was evaluated.

Influence of L-FR-107L amount

		1	2	3	4	5
NR		100	100	100	100	100
L-FR-107L		0	1	5	10	20
CB		50	50	50	50	50
Mooney Viscosity, ML1+4 (100°C)		62	60	54	47	35
Curelastometer (145°C)						
t10	(min.)	6.9	6.7	6.8	7.0	7.2
t90	(min.)	12.4	12.1	12.3	12.3	12.6
ML	(N.m)	0.2	0.2	0.2	0.2	0.2
MH	(N.m)	1.5	1.5	1.4	1.3	1.1
Mechanical properties						
Hs	Type A	66	64	63	59	53
EB	(%)	509	509	527	516	535
TB	(MPa)	31.6	31.2	30.4	28.2	26.5
M50	(MPa)	1.6	1.6	1.4	1.3	1.0
M100	(MPa)	3.3	3.2	2.8	2.5	1.8
M300	(MPa)	17.5	16.9	15.3	13.8	11.6
DIN abrasion	index	100	115	108	87	79

✓ Tensile and abrasion properties were maintained up to 5phr addition.

Viscoelastic Data

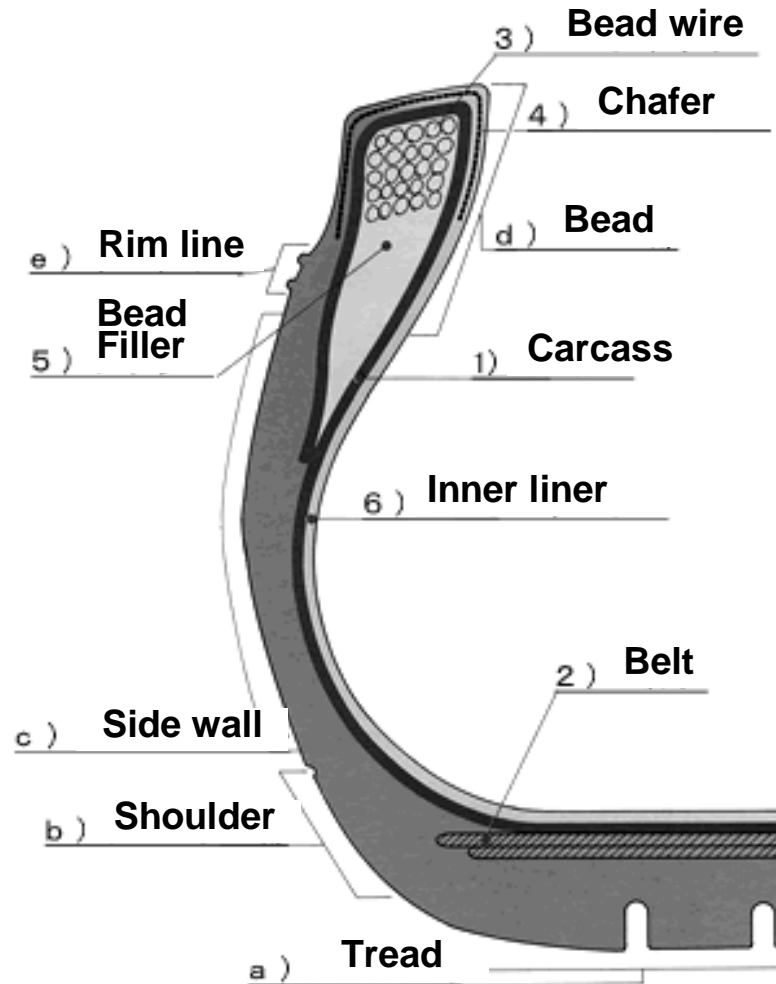


✓ 5 phr addition of L-FR-107L decreased $\tan \delta$ effectively.

Influence of L-FR-107L Summary

5 phr of L-FR-107L achieved well balanced properties, the lowest $\tan\delta$, physical properties and abrasion resistance.

Suitable Application of Liquid Farnesene Rubber



TBR Compound

- ✓ Replacement of resin or oil
- ✓ Good plasticizing effect
- ✓ No migration
- ✓ Low $\tan\delta$

Summary

- Renewable Biomass Material
- Good Plasticizing Effect
- No Bleeding & No Migration
- Decrease $\tan\delta$

Raw materials

Material	Product Name	Manufacturer	Note
Natural Rubber	STR20	Von Bundit Co., Ltd.	
Carbon black	DIABLACK™ I	Mitsubishi Chemical Corporation	ASTM N220
TDAE	VIVATEC 500	H&R GmbH Co. KGaA	

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Comparison with oils & resins

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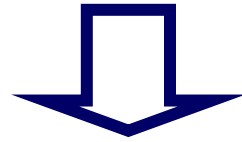
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Agenda

- Introduction of Bio-based Liquid Rubber
- Comparison with various plasticizer
- Influence of Added Amount in NR/CB Formulation

Replacement of Hydrocarbon Resin

- In TBR, hydrocarbon resin is used as plasticizer rather than process oil to reduce migration.
- Resin increases $\tan\delta$ higher than process oil.



- L-FR-107L achieves good plasticizing effect without migration.
Besides, it gives tremendously low $\tan\delta$.

List of Plasticizers

Category	Product Description	Tg (°C)	Supplier
Liquid Rubber	Polyfarnesene (L-FR-107L)	-71	Kuraray
Resins	C5 Aliphatic Hydrocarbon (Escorez™ 1102)	46	Exxon Mobil Corporation
	Alicyclic Hydrocarbon (Quintone 1105)	49	Zeon Corporation
	C9 Coumarone-Indene (G90)	49	NITTO CHEMICAL CO., LTD.
Oil	Aromatic (JSO AROMA 790)	-44	JAPAN SUN OIL COMPANY, LTD.
	Naphthenic (SUNTHENE 450)	-63	JAPAN SUN OIL COMPANY, LTD.
	Paraffinic (DIANA Process Oil PW 380)	-59	Idemitsu Kosan Co., Ltd.
	TDAE (VIVATEC 500)	-50	H&R GmbH Co. KGaA

NR/CB Formulation

<u>Formulation</u>	<u>phr</u>
Natural Rubber (STR20)	100
Plasticizer	4
Carbon Black (N220)	45
ZnO	3.5
Stearic acid	2
Sulfur	1.5
Accelerator NS ¹⁾	1.2
Antioxidant 6C ²⁾	1
Antioxidant RD ³⁾	1

1) N-tert-butyl-2-benzothiazolesulfenamide

2) N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylenediamine

3) Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline

Mixing Conditions

1st stage: Banbury mixer

0'00" NR (75°C)

0'20" CB, TDAE/Liquid Rubber
AO, ZnO, Stearic acid

6'00" Dump out (150-160°C)

2nd stage: Roll mill

0'00" Compound (55-60°C)

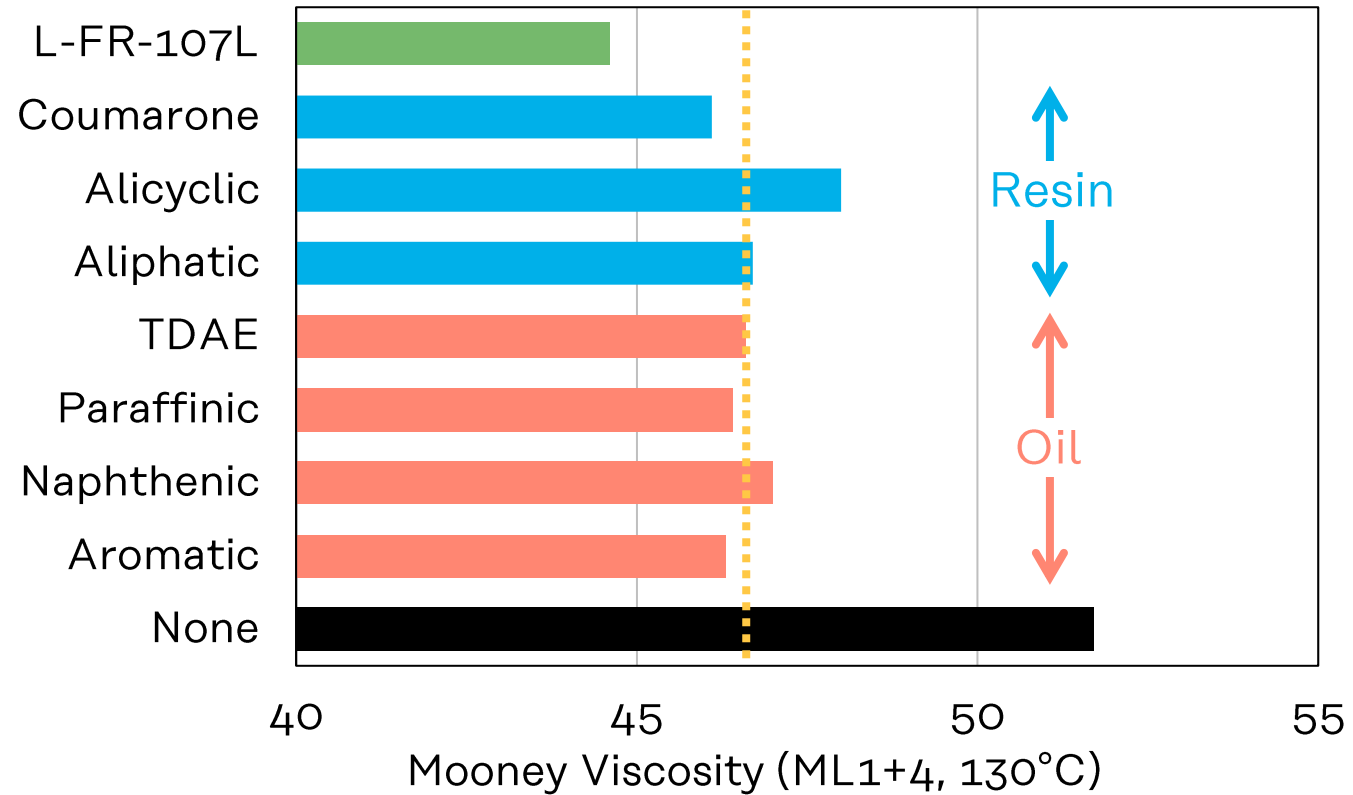
0'30" Sulfur, Accelerator

7'00" Sheeting

NR/CB Formulation Summary

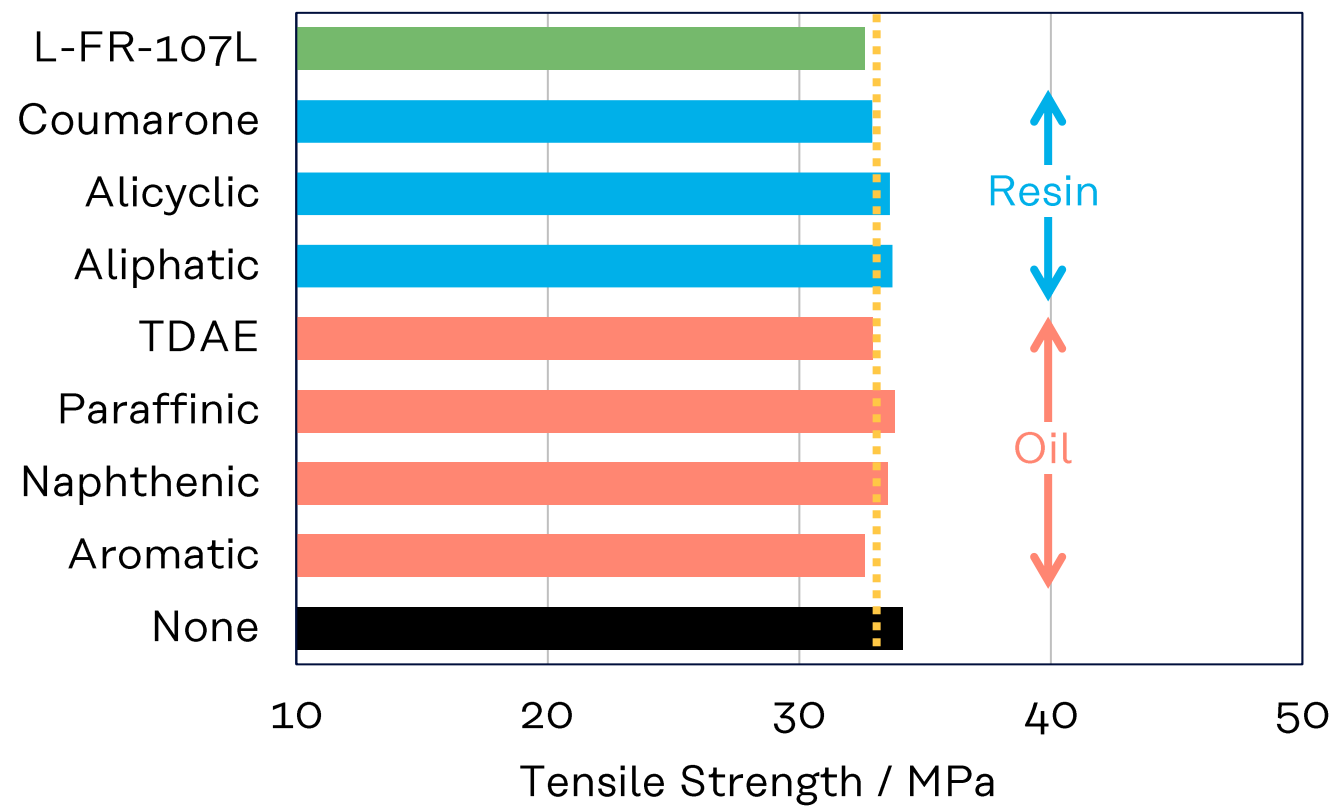
	1	2	3	4	5	6	7	8	9	
Category			Oil				Resin			
Plasticizer	None	Aromatic	Naphthenic	Paraffinic	TDAE	Aliphatic	Alicyclic	Coumarone	L-FR-107L	
Mooney Vis., ML1+4 (130°C)	51.7	46.3	47.0	46.4	46.6	46.7	48.0	46.1	44.6	
Mooney Scorch time (130°C)										
t5 (min.)	18.6	19.8	21.3	21.4	20.6	20.6	24.5	18.3	19.9	
t35 (min.)	21.0	22.3	23.8	23.9	23.1	23.0	27.3	20.5	22.4	
Curelastometer (155°C)										
t10 (min.)	3.5	3.5	3.8	3.9	3.8	3.7	4.3	3.2	3.5	
t90 (min.)	5.8	5.7	6.3	6.5	6.3	6.2	6.5	5.4	5.8	
ML (N.m)	0.25	0.23	0.22	0.22	0.23	0.23	0.23	0.23	0.22	
MH (N.m)	1.48	1.39	1.38	1.36	1.38	1.34	1.23	1.35	1.37	
Mechanical Properties										
Hardness Type A	66	64	64	63	64	64	63	66	65	
EB (%)	540	572	574	575	560	563	607	565	547	
TB (MPa)	34.1	32.6	33.5	33.8	32.9	33.7	33.6	32.9	32.6	
M50 (MPa)	1.61	1.41	1.44	1.40	1.43	1.47	1.34	1.51	1.45	
M100 (MPa)	3.16	2.63	2.72	2.66	2.71	2.80	2.43	2.82	2.77	
M300 (MPa)	16.3	13.7	14.1	14.2	14.3	14.6	12.6	14.3	14.7	
Viscoelasticity (25°C)										
E' (MPa)	5.81	5.24	5.28	5.28	5.27	5.49	5.37	5.42	5.11	
tanδ (-)	0.171	0.189	0.177	0.176	0.176	0.185	0.200	0.191	0.155	

Mooney Viscosity



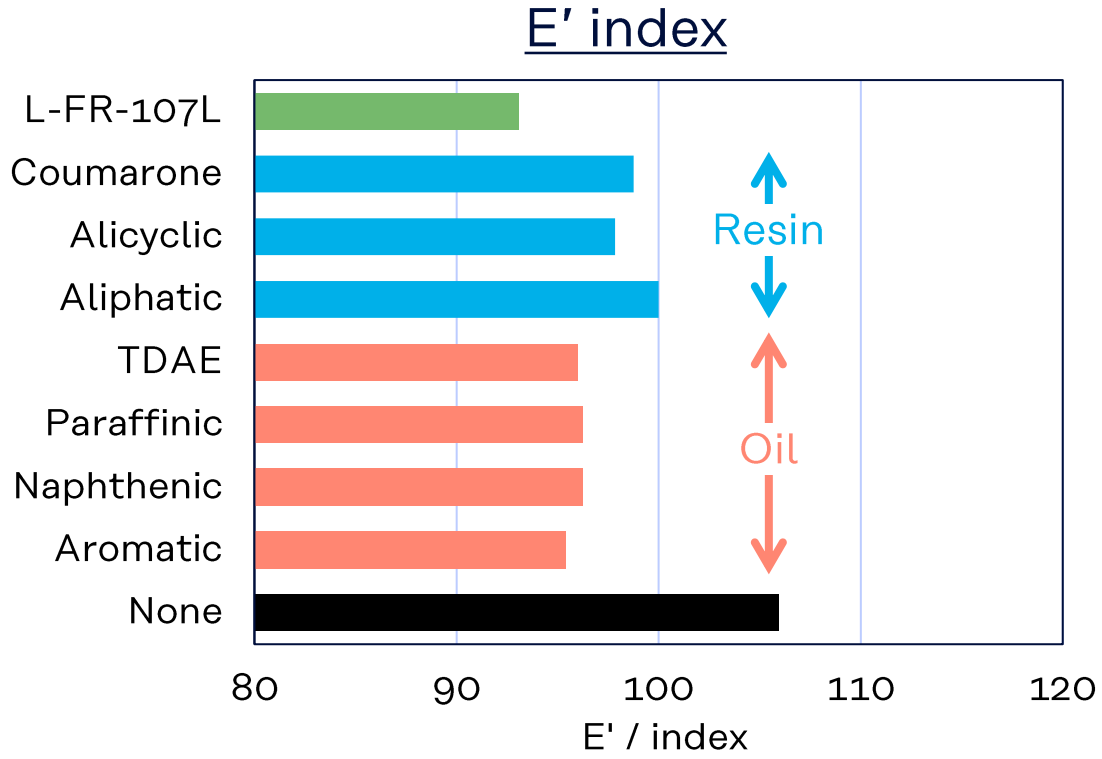
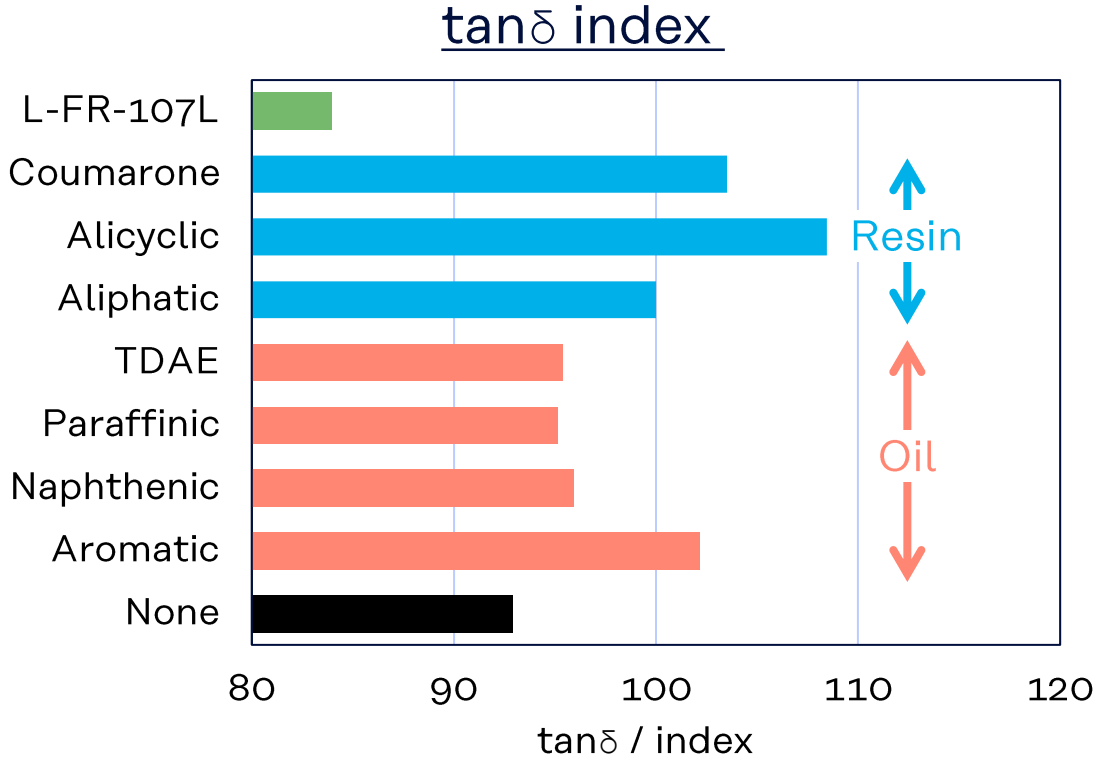
✓ Good plasticizing effect

Tensile Strength



✓ Equivalent tensile strength

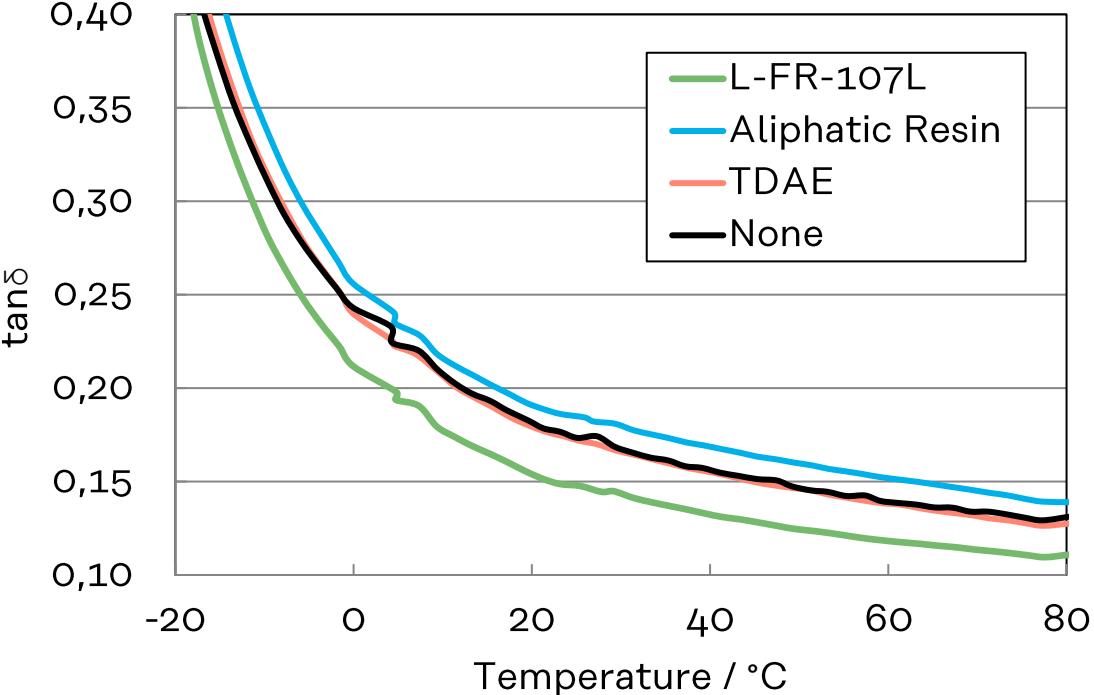
Viscoelastic Data (25°C)



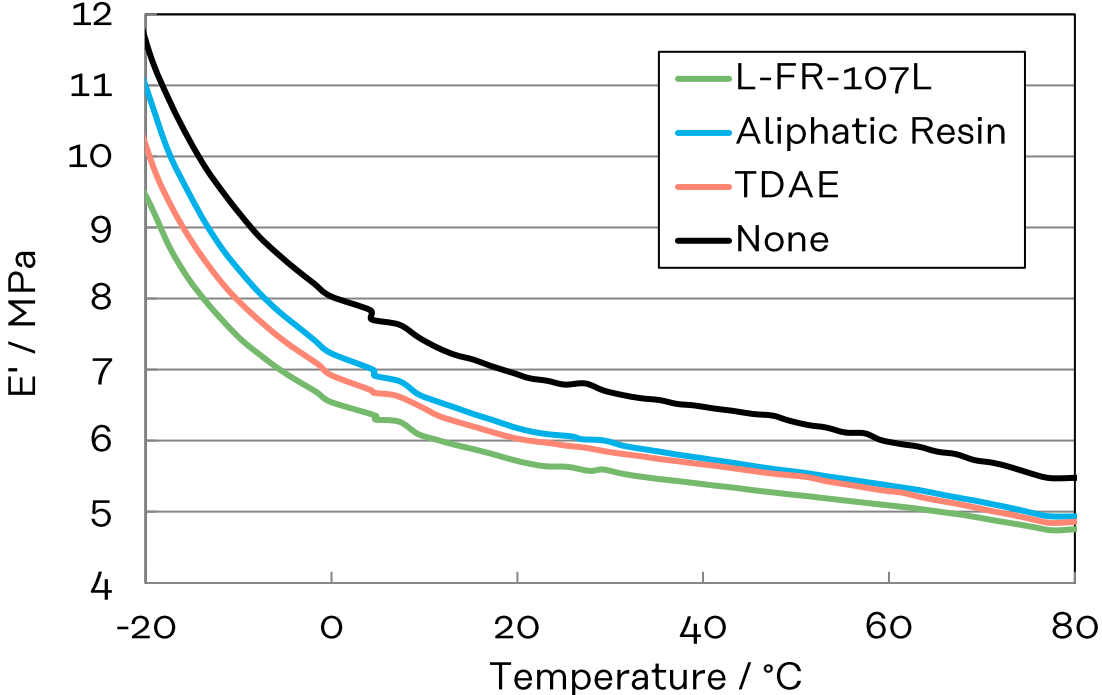
- ✓ 20% tanδ reduction compared with resins
- ✓ Equivalent E' to oils

Viscoelastic Data (Temp Sweep)

tanδ (temp. sweep)



E' (temp sweep)



✓ Lower tanδ over wide range of temp

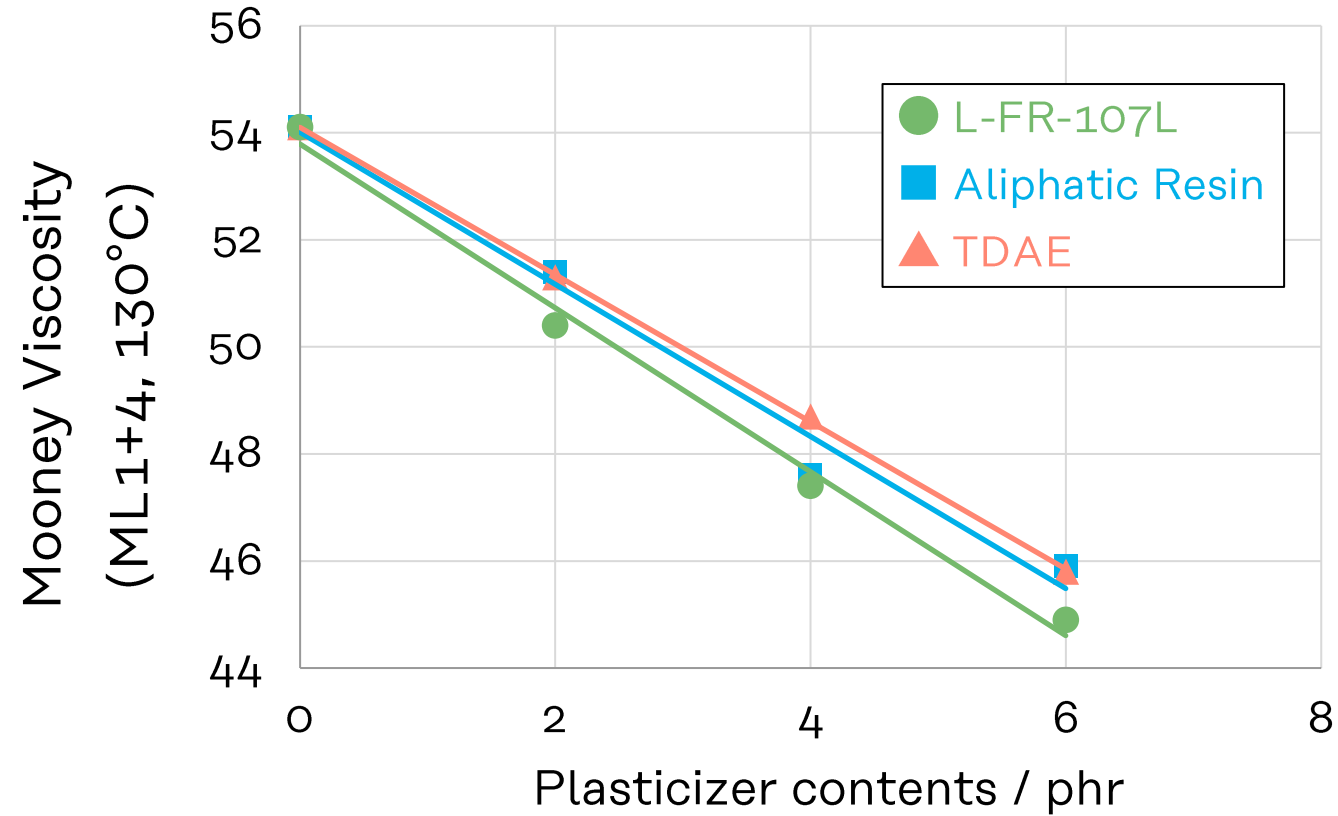
Agenda

- Introduction of Bio-based Liquid Rubber
- Comparison with various plasticizer
- Influence of Added Amount in NR/CB Formulation

NR/CB Formulation Summary

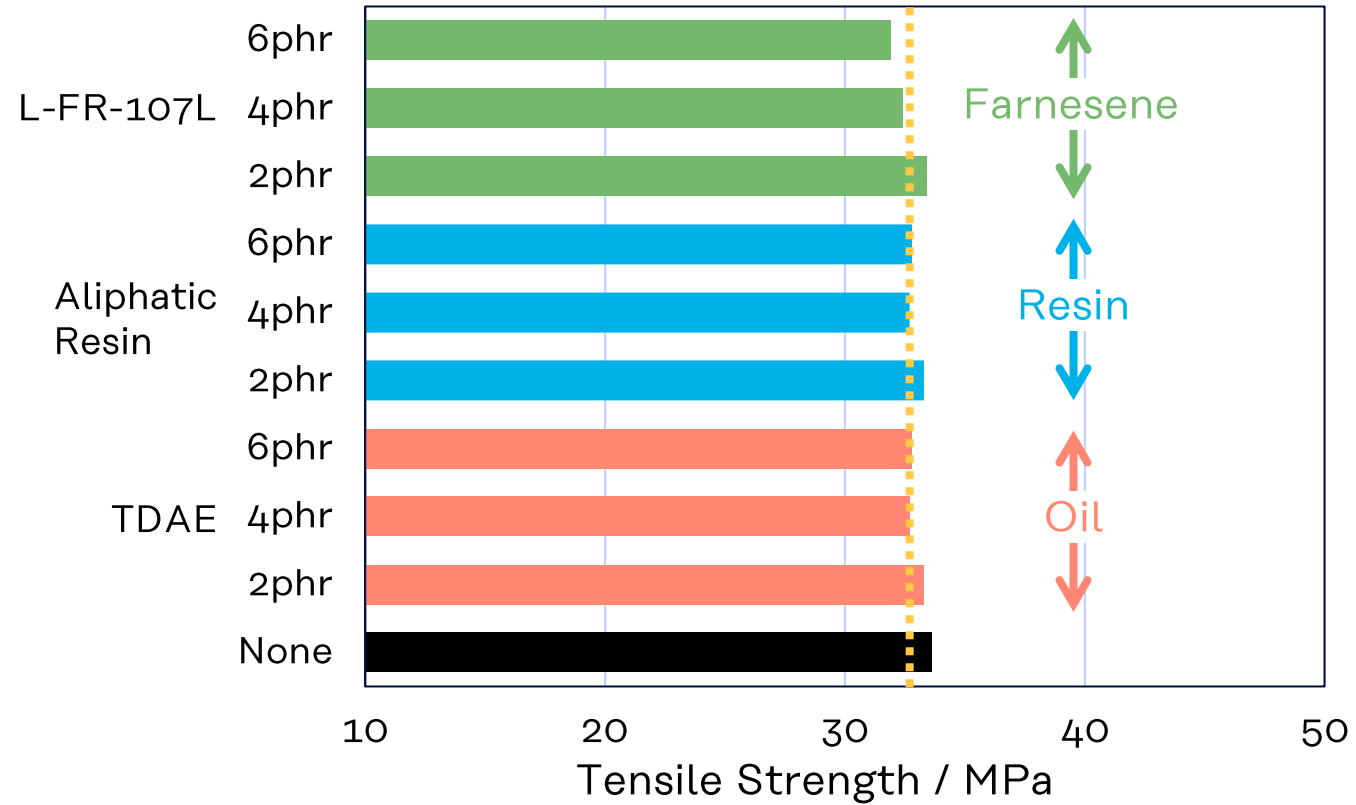
		1	2	3	4	5	6	7	8	9	10
Plasticizer		None	TDAE			Aliphatic Resin			L-FR-107L		
Amount	(phr)	0	2	4	6	2	4	6	2	4	6
Mooney Vis., ML1+4 (130°C)		51.4	51.4	47.6	45.9	51.3	48.7	45.8	50.4	47.4	44.9
Mooney Scorch time (130°C)											
t5	(min.)	20.3	20.3	21.1	22.0	21.1	21.5	22.6	21.1	20.1	20.4
t35	(min.)	22.8	22.9	23.8	24.9	23.9	24.1	25.3	23.9	23.0	23.3
Curelastometer (155°C)											
t10	(min.)	3.9	3.9	4.1	4.2	4.1	4.2	4.3	4.1	4.1	4.1
t90	(min.)	6.4	6.5	6.7	6.8	6.8	6.9	7.0	6.7	6.7	6.7
ML	(N.m)	0.25	0.24	0.23	0.22	0.24	0.22	0.21	0.24	0.23	0.22
MH	(N.m)	1.51	1.45	1.39	1.33	1.44	1.37	1.30	1.46	1.41	1.37
Mechanical Properties											
Hardness	Type A	65	65	64	63	65	64	63	65	64	63
EB	(%)	538	556	560	577	548	553	581	552	545	541
TB	(MPa)	33.6	33.3	32.7	32.8	33.3	32.7	32.8	33.4	32.4	31.9
M50	(MPa)	1.58	1.49	1.40	1.33	1.51	1.42	1.39	1.50	1.42	1.37
M100	(MPa)	3.03	2.85	2.63	2.50	2.89	2.68	2.61	2.83	2.67	2.58
M300	(MPa)	16.0	15.0	13.9	13.1	15.3	14.3	13.6	15.0	14.3	14.0
Viscoelasticity (25°C)											
E'	(MPa)	5.67	5.43	5.12	4.97	5.55	5.40	5.23	5.45	5.25	5.03
tanδ	(-)	0.171	0.169	0.173	0.181	0.182	0.190	0.197	0.169	0.161	0.161

Mooney Viscosity



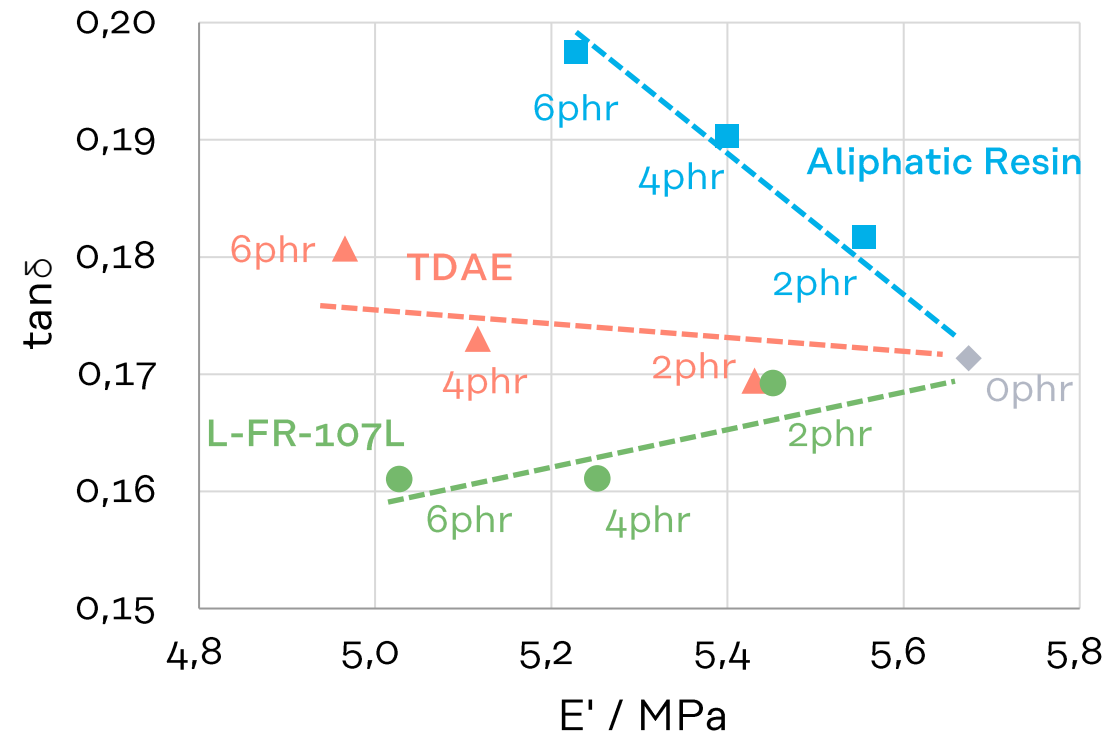
✓ Good Plasticizing Effect

Tensile Strength



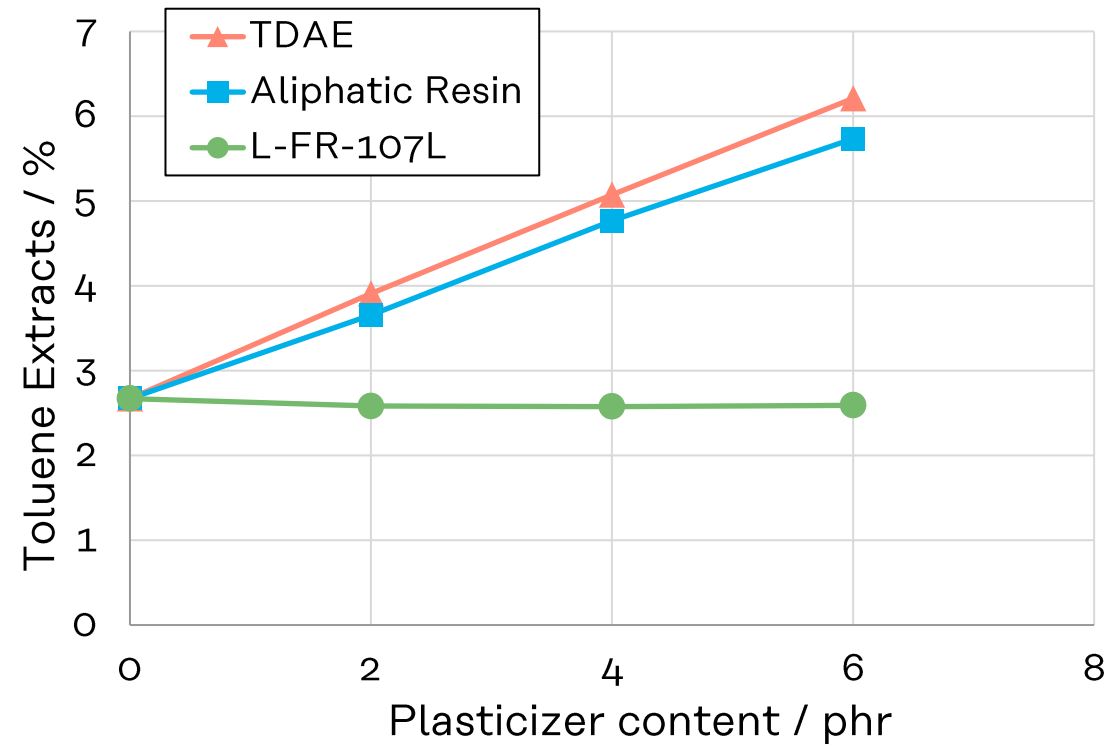
✓ Similar tensile strength

Viscoelastic Data (25°C)



- ✓ The resin increases $\tan \delta$ in proportion to additive amount.
- ✓ 4-6 phr of L-FR-107L reduces $\tan \delta$ the most effectively.
- ✓ Even 2 phr of L-FR-107L is effective compared with the resin.

Toluene Extraction Test



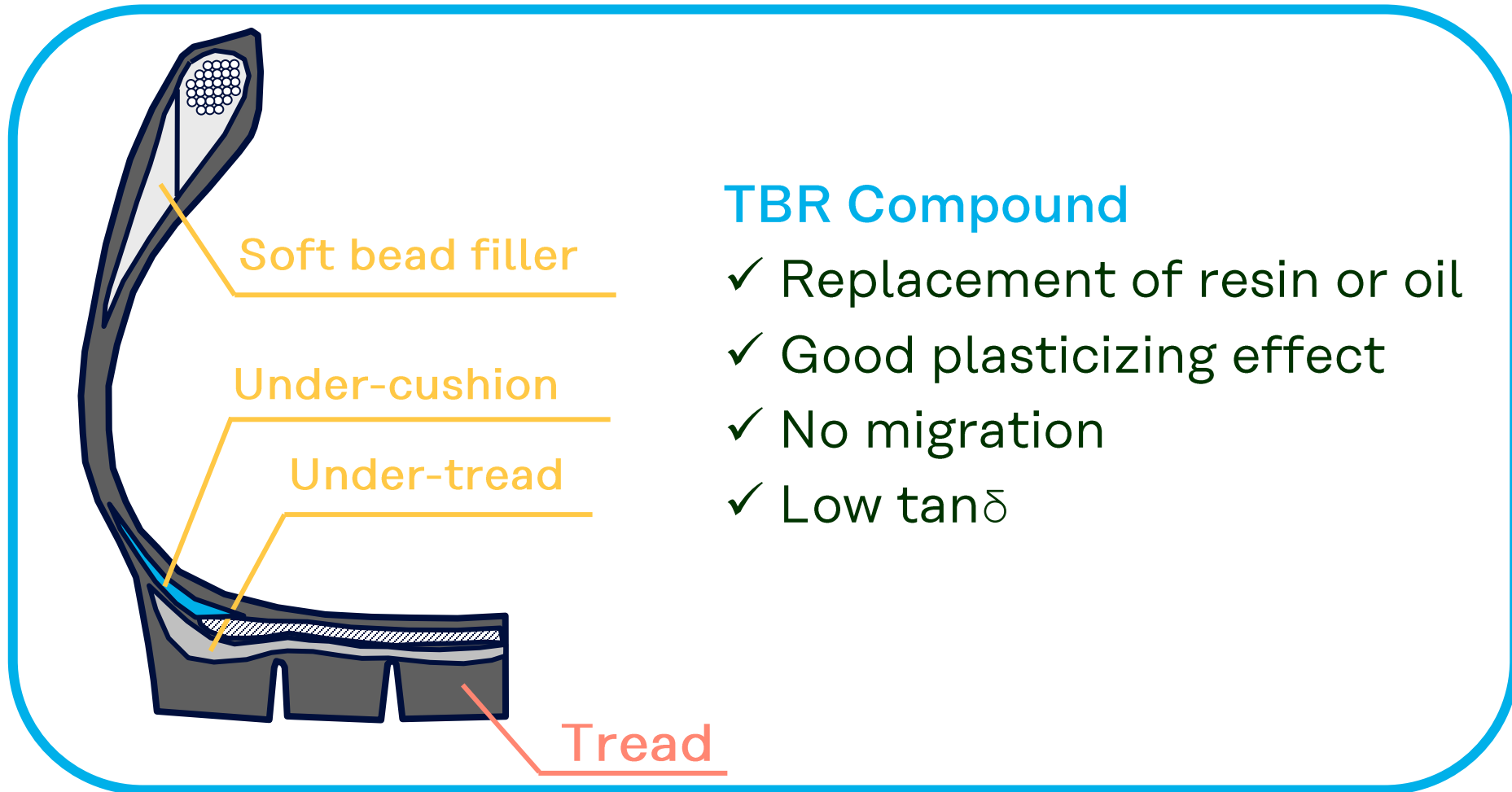
- ✓ L-FR-107L is not extracted due to co-vulcanization.
- ✓ Less or no migration.
- ✓ Long term reliability is expected.

Summary

Features of KB-107 in NR/CB Formulation

- Low $\tan\delta$
- Biomass Material
- Good plasticizing effect
- No Migration

Idea of Application



TBR Compound

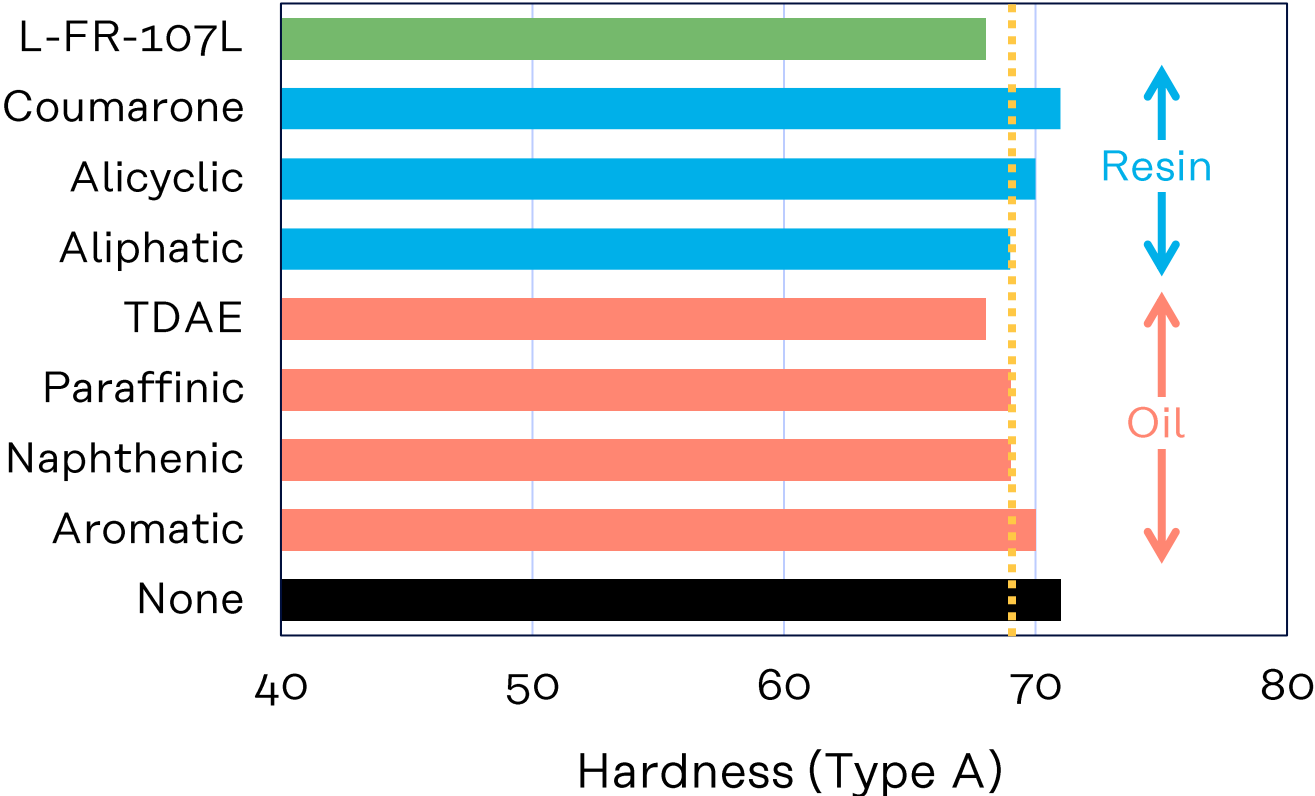
- ✓ Replacement of resin or oil
- ✓ Good plasticizing effect
- ✓ No migration
- ✓ Low $\tan\delta$

APPENDIX

Raw materials

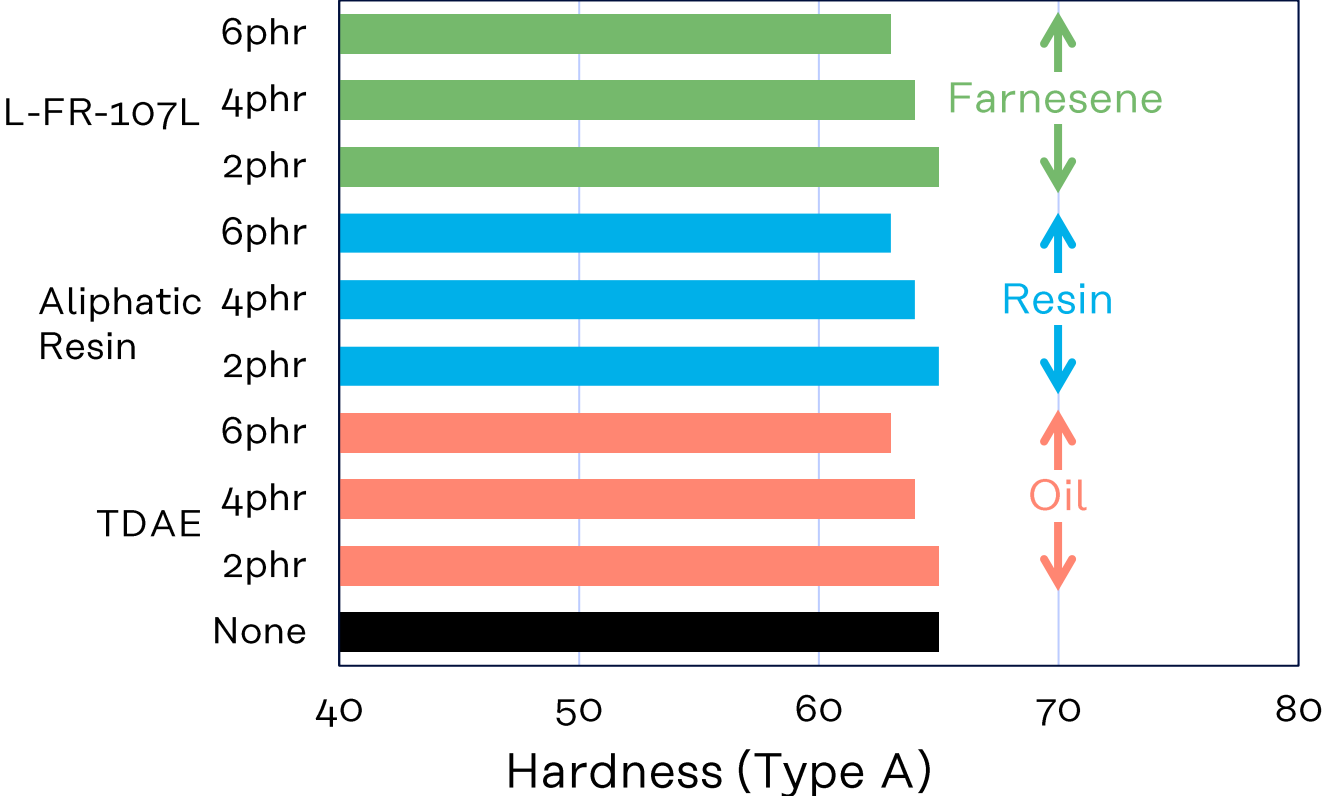
Material	Product Name	Manufacturer	Note
Natural Rubber	STR20	Von Bundit Co., Ltd.	
Carbon black	DIABLACK™ I	Mitsubishi Chemical Corporation	ASTM N220
TDAE	VIVATEC 500	H&R GmbH Co. KGaA	

Hardness (NR/CB/Plasticizer=100/45/4)

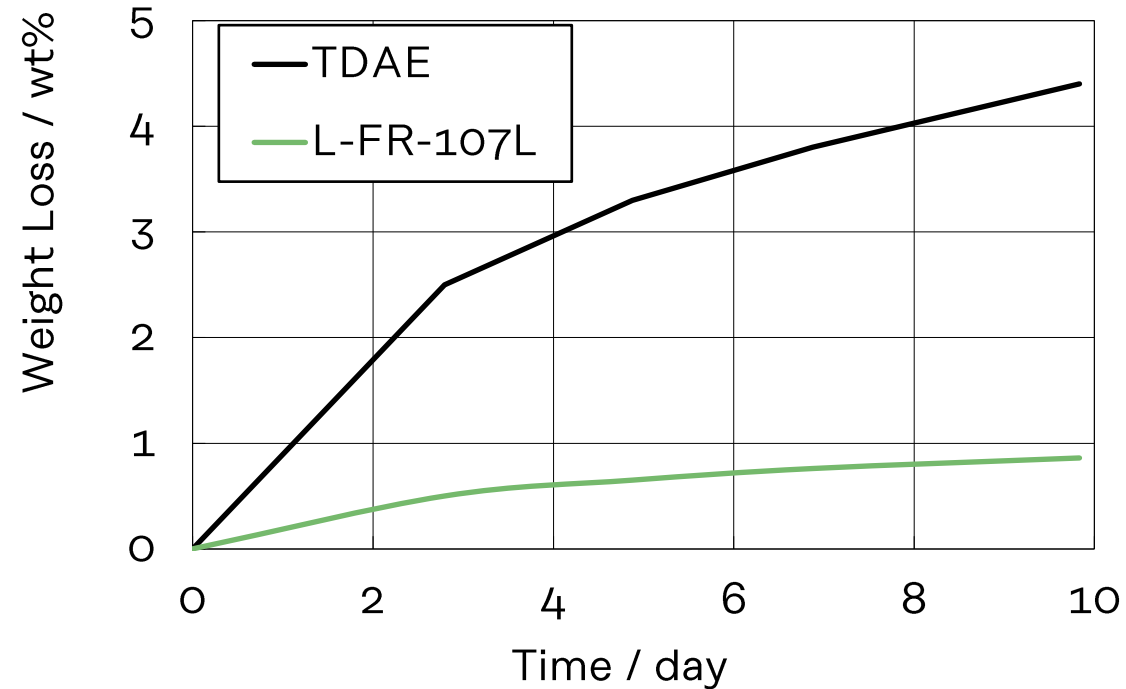


✓ Same range of Hardness

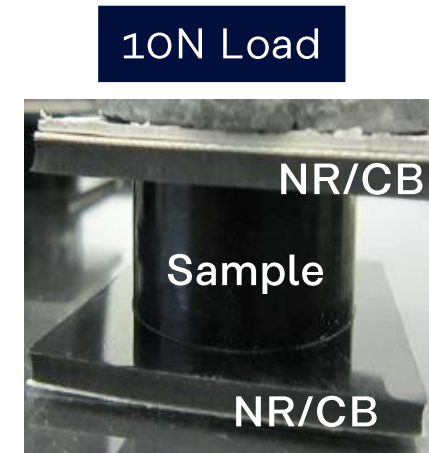
Hardness (NR/CB/Plasticizer=100/45/2-6)



Migration Property



-Formulation-
SBR/CB/Plasticizer
=100/70/40
-Test condition-
Temp.: 70°C



✓ Less migration property => Expected long-term reliability

Kuraray Co., Ltd.
Elastomer Division
Tokiwabashi Tower
2-6-4, Otemachi
Chiyoda-ku, Tokyo 100-0004, Japan

✉ elastomer@kuraray.com

→ www.kuraray.com

→ www.elastomer.kuraray.com

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