A New Comparison of Sheet and Crumb Rubber. Part II. Some Aspects of Processing

G.M. BRISTOW* AND A.G. SEARS*

Complementary samples of RSS CV and crumb SMR CV covering the Mooney viscosity range ca. 50-85 were prepared from five lots of monoclonal latex. Similar processability and curing behaviour were observed for typical tread stocks prepared from these materials. Only in the case of the initial formation of bound rubber or carbon gel, a parameter indicative of rubber/black interaction, was there evidence of an obvious effect of raw rubber production procedure, but even here, this difference was eliminated in the inevitable further working during final mixing.

In a previous paper¹, a comparison was presented of various raw rubber properties of samples of monoclonal SMR CV (crumb) and RSS CV (sheet). Here, data characterising some aspects of processing behaviour for these samples are considered. The main theme is a comparison between the sheet and crumb materials derived from a common (monoclonal) latex source. The processing qualities considered are:

- Susceptibility to oxidative or mechanochemical breakdown
- Banbury mixing of fine particle size carbon black (N220, ISAF black) in a typical tread stock
- Capillary extrusion behaviour of materials prepared by Banbury mixing of fine particle size carbon black.

EXPERIMENTAL

Breakdown Properties

Breakdown was assessed in three different ways: by the well-established PRI test, by mastication in a Brabender plasticorder² (Model PLV 151) and by the hot pressing test described by Lim and Lim³. In each case,

rubber blended according to the SMR procedure⁴ was used.

The plasticorder was equipped with an N50H mixing chamber, cam-type rotors and a standard pressure ram. The machine was heated by circulating oil to either 60°C or 100°C, after which 60 g of rubber, corresponding to a fill factor of 0.82, was masticated at 100 r.p.m. for 4 min. At the end of this period, at which time the rubber temperature as indicated by the built-in thermocouple probe was 100°C-115°C or 130°C-145°C depending on the machine temperature, the rubber was rapidly removed and cooled by a single pass through a cold two-roll mill. Mooney viscosity, V_R mast. (ML1+4, 100°C) was measured after a resting period of 18-24 h. Breakdown was expressed either as the simple decrease in viscosity, $\Delta V_{\rm p}$, or by a breakdown index, BI, where:

$$BI = \frac{\Delta V_R 10^4}{\text{Initial } V_R Wu}$$

Wu being the total work per unit volume (MJ/m³), obtained from integration of machine torque as a function of time⁵. Typical within-blend reproducibility, separately determined from five replicate tests on a single sample of blended SMR L, was:

^{*}Malaysian Rubber Producers' Research Association, Brickendonbury, Hertford, SG13 8NL, United Kingdom

	Mean	S.D.	C.V. (%)
V_R mast.	77.9	0.4	0.5
BI	1.25	0.05	3.7
Dump temperature (°C)	151.0	0.7	0.5

The hot pressing test was operated in the manner indicated by Lim and Lim³ using a platen temperature of 200°C and a sample thickness of 0.5 mm. Breakdown was expressed as ΔV_R or $VRP = (\Delta V_R/\text{Initial } V_R) \times 10$.

Typical within-blend reproducibility, separately determined from ten replicate tests on a single sample of blended SMR CV, was:

	Mean	S.D.	C.V. (%)
V_R pressed	48.5	0.47	0.97
VRP	2.42	0.08	3.12

Banbury Mixing

The masterbatch formulation shown in *Table 1* was mixed in a BR Banbury at a batch factor of 8 and under the following conditions:

Masterbatch mixing cycle

0 min : Add rubber

1 min: Add small powders, black and oil

2 min : Sweep

3 min: Discharge

Cool by one pass through cool

two-roll mill

Remill cycle

0 min: Add masterbatch

2 min : Discharge

Cool by one pass through cool

two-roll mill

BR Banbury rotor speed 155 r.p.m., starting temperature 50°C-60°C, cooling water 7.6 litres/min

Weight losses during mixing were ca. 0.5%. The mixing operation was characterised in terms of gross energy and dump temperature. The power-time traces gave no real indication of black incorporation time (BIT), consistent

TABLE 1. FORMULATION FOR TEST COMPOUNDS

Item	Parts by weight
Masterbatch	
Natural rubber, unmasticated	100
N220, ISAF black	45
Process oil ^a	5
Zinc oxide	5
Stearic acid	3
IPPD ^b	2
Curatives ^c	
Sulphur	2.5
TBBS ^d	0.5

^a Aromatic process oil, Dutrex 729 HP (Shell Chemicals)

with previous experience in these laboratories of mixing practical 100% NR stocks in this type of mixer. While BIT phenomena may be readily apparent in the BR Banbury with some synthetic rubbers, they are only obvious with NR at high levels of black and preferably in the absence of plasticiser and zinc soaps⁶.

The mixes (masterbatches) were characterised by values of Mooney viscosity, V_B , Cabot dispersion rating⁷ and bound rubber after 48 h immersion in toluene. In order to avoid testing final mixes of atypically high viscosity, after 24 h the masterbatches from the highest viscosity clones RRIM 628 and PR 261 were remilled for 2 min in the Banbury under the same conditions as before. After resting for a further 24 h, curatives (Table 1) were added on a two-roll mill. Tests on these final mixes comprised viscosity, V_c , bound rubber content and rheometry at 150°C.

Capillary Flow Tests

An Instron 3211 rheometer was used with two capillaries, both 1.27 mm in diameter but

bN-Isopropyl-N'-phenyl-p-phenylenediamine, Permanax IPPD (Vulnax International)

c Added on a two-roll mill after resting for a further 24 h

^d N-t-Butylbenzothiazole-2-sulphenamide, Santocure NS (Monsanto)

having different lengths: 2.54 mm and 25.4 mm. Testing was limited to two piston speeds corresponding to Newtonian shear rates of ca. 95 s⁻¹ and 445 s⁻¹. Values for wall shear stress π_{w} end correction e, flow index n, and extrudate area swell were derived as described previously¹.

RESULTS AND DISCUSSION

The presentation of the test data is the same as that adopted in $Part I^{\perp}$. That is, results are presented where appropriate as overall mean and standard deviations for the sheet and crumb materials and, more importantly, as values of the difference (sheet minus crumb) for the five clones, together with means and standard deviations for these differences.

Data characterising raw rubber breakdown are given in Table 2 and further analysed in Table 3. Rather surprisingly, perhaps, the sheet materials show slightly lower values of PRI than the crumb rubbers and, consistent with this, mean values of ΔV_R in the Brabender and 200°C pressing tests are larger for the sheet than the crumb rubbers. No such correlation exists. however, for the individual materials. Some discrepancy is evident here with the results of Lim and Ong8, who found that, in the Brabender test, value of ΔV_R for samples of standard, unstabilised RSS 1 were much lower than those for a range of SMR crumb grades including SMR CV. The obvious interpretation of this apparent conflict is that the hydroxylamine neutral sulphate treatment used to prepare RSS CV has destroyed, or offset, this unique character of RSS 1. It must be noted, however, that Lim and Ong's values for SMR L were not greatly different from those they obtained for SMR CV. Furthermore, very limited studies of monoclonal SMR L and SMR CV in these laboratories have given lower ΔV_R values for the CV materials⁹.

Despite the limited consistency between low PRI and high ΔV_R for the Brabender and pressing tests noted above, correlations between the three breakdown parameters are very poor. This is shown by the coefficients recorded in Table 4 and is very evident in the plots of Figures 1, 2 and 3. For the Brabender,

breakdown at both 60°C and, especially, 100°C correlates quite well with the *initial* viscosity of the rubber (Figure 4). As shown by the excellent correlations of Figure 5, greater breakdown stems from the higher heat generation achieved with a higher viscosity rubber. Finally, and most importantly in the present context, in all these correlations, good and bad, there is no evidence for specific effects of the sheet or crumb nature of the rubber.

Data for the mixing of the tread stock from unmasticated rubber are given in *Table 5* with further analysis in *Table 6*. The mixing process was characterised by two parameters, mixing energy and dump temperature. The results show that neither of these discriminates between sheet and crumb rubbers. Furthermore, and rather unexpectedly, neither shows any dependence on the initial viscosity of the rubber.

In keeping with the trends noted for raw rubber breakdown, viscosity of the masterbatch is, as shown in *Figure 6*, strongly dependent on that of the raw rubber and the same regression equation:

$$V_B = 8.76 + 1.07 V_R$$

Correlation coefficient = 0.995

fits both the SMR CV and RSS CV materials. The black dispersion rating of the masterbatch also shows a (negative) correlation with rubber viscosity but here there is slight evidence that inferior dispersion is attained with the sheet rubbers (Figure 7). At the final mix stage, viscosity still shows a correlation with raw rubber viscosity though, as shown in Figure 8, the dependence is less marked:

$$V_C = 28.97 + 0.347 V_R$$

Correlation coefficient = 0.905

No differences in black dispersion were evident after final mixing.

While, despite extensive measurements over many years, the technological significance of bound rubber (carbon gel) remains obscure, in the present instance such measurements do in fact differentiate between sheet and crumb, at least at the masterbatch stage. This is clearly apparent in the plots of percent bound rubber

TABLE 2. RAW RUBBER BREAKDOWN PARAMETERS

			SMR CV			RSS CV				
Item	RRIM 600	RRIM 623	RRIM 628	RRIM 701	PR 261	RRIM 600	RRIM 623	RRIM 628	RRIM 701	PR 261
PRI	91	90	81	83	88	89	81	75	78	85
ML1+4, 100°C V _R	52.5	56.5	84	52	67.5	57	62	87	56	72.5
Brabender mastication 4 min at 100 r.p.m.										
Machine temperature, 60°C										
Final temperature (°C)	101	104	114	102	108	102	106	116	103	110
V _R mast.	45	50	76	46	58.5	48	55	78	49	62
ΔV_R	7.5	6.5	8	6	9	9	7	9	7	10.5
ВІ	1.71	1.33	0.94	1.38	1.43	1.83	1.22	1.36	1.50	1.49
Machine temperature, 100°C					j					
Final temperature (°C)	131	134	145	130	137	134	137	147	134	141
V _R mast.	46.5	52	76	46.5	59	49	54	76	49	61
ΔV_R	6	4.5	8	5.5	8.5	8	8	11	7	10.5
ВІ	1.47	0.99	0.99	1.46	1.46	1.78	1.48	0.28	1.56	1.78
200°C pressing test										
V _R after pressing	41	46	80	39	56	43	48	81	40	58
ΔV_R	11.5	10.5	4	13	11.5	14	14	6	16	14.5
VRP	2.19	1.86	0.48	2.50	1.70	2.46	2.26	0.69	2.86	2.00

TABLE 3. FURTHER ANALYSIS OF THE DATA OF TABLE 3

	RSS CV		SMR	SMR CV			Δ (RSS	CV - SMI	R CV)		
Item	Mean	S.D.	Mean	S.D.	RRIM 600	RRIM 623	RRIM 628	RRIM 700	PR 261	Mean	S.D.
PRI	81.6	5.6	86.6	4.4	-2	-9	-6	- 5	-3	-5.0	2.7
Brabender mastication											
Machine temperature, 60°C											
Final temperature (°C)	107.4	5.7	105.8	5.3	+ 1	+2	+2	+ 1	+ 2	+ 1.6	0.5
ΔV_R	8.5	1.5	7.4	1.2	+ 1.5	+0.5	+1	+ 1	+1.5	+ 1.1	0.4
Machine temperature, 100°C				ļ							
Final temperature (°C)	138.6	5.5	135.4	6.0	+ 3	+ 3	+2	+4	+4	+ 3.2	0.8
ΔV_R	8.9	1.7	6.5	1.7	+2	+3.5	+ 3	+ 1.5	+ 2	+ 2.4	0.8
200°C pressing test											
ΔV_R	12.9	3.9	10.1	3.5	+2.5	+3.5	÷ 2	+ 3	+ 3	+ 2.8	0.6

Parameter	Data points	Correlation coefficient	Significance
ΔV_R Brabender, PRI			
60°C	10	0.015	< 90
100°C	10	-0.489	< 90
ΔV_R Brabender, ΔV_R 200°C pressing			
60°C	10	-0.079	< 90
100°C	10	-0.386	< 90
Plasticity Retention Index, ΔV_R 200°C pressing	10	0.245	< 90
ΔV_R Brabender, initial V_R			
60°C	10	0.573	90-95
100°C	10	0.769	99
Final Brabender temperature, ML1 + 4, 100°C			
60°C	10	0.992	> 99.9
100°C	10	0.988	> 99.9

TABLE 4. CORRELATIONS BETWEEN VARIOUS BREAKDOWN PARAMETERS

versus V_B and bound rubber swelling versus V_B of Figures 9 and 10 respectively. As might be anticipated, the discrimination between sheet and crumb is no longer evident if percent bound rubber is plotted against bound rubber swelling (Figure 11). Conversion of masterbatch to final mix (via a remill stage for clones RRIM 628 and PR 261) results in a decrease in bound rubber (Figure 12) together with an increase in swelling (Figure 13) and, more importantly, the disappearance of any consistent difference between sheet and crumb.

The obvious interpretation of these results is that, while initially during the preparation of masterbatch the interaction between carbon black and rubber is greater for RSS, this is levelled out during subsequent reworking and/or preparation of the final mix. The last measure used to characterise the final mixes, rheometric cure behaviour, also shows no difference between batches based on sheet and crumb and will be considered again in *Part III*.

On the basis of the above results, no differences in downstream processability would be expected between sheet and crumb-based materials. Further confirmation of such parity has been sought in capillary extrusion performance at modest (95 s⁻¹) and high (445 s⁻¹) shear rates. The results in terms of wall shear stress, flow index, and correction and extrudate swell, derived as indicated in *Part I*¹ are given in *Tables 7* and 8.

In the first place it is to be noted that, as shown in *Figure 14*, good correlations exist between the Mooney viscosity, V_c , and wall shear stress at both low and high shear rates. However, the 'normalised' slopes:

Slope
$$\frac{\text{Mean } V_c}{\text{Mean } \pi_w}$$

of the regression lines, together with the likely relative precision of the measured values of π_w and V_c , suggest that Mooney viscosity will

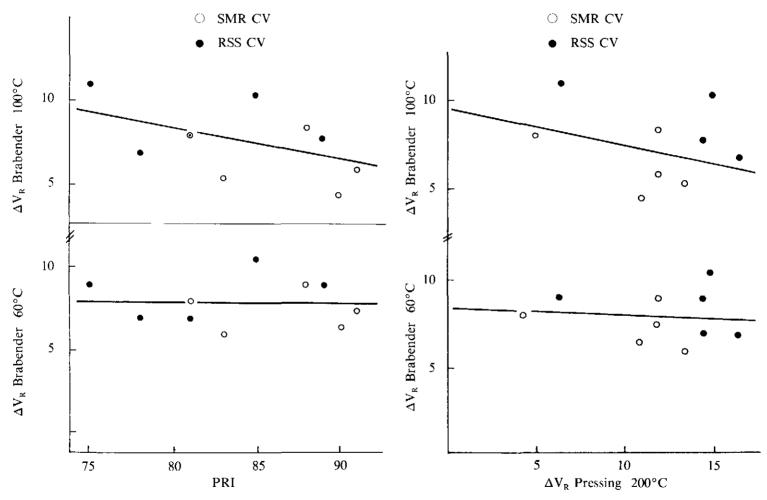


Figure 1. Correlation of Brabender breakdown and PRI.

Figure 2. Correlation of Brabender breakdown and breakdown in pressing at 200°C.

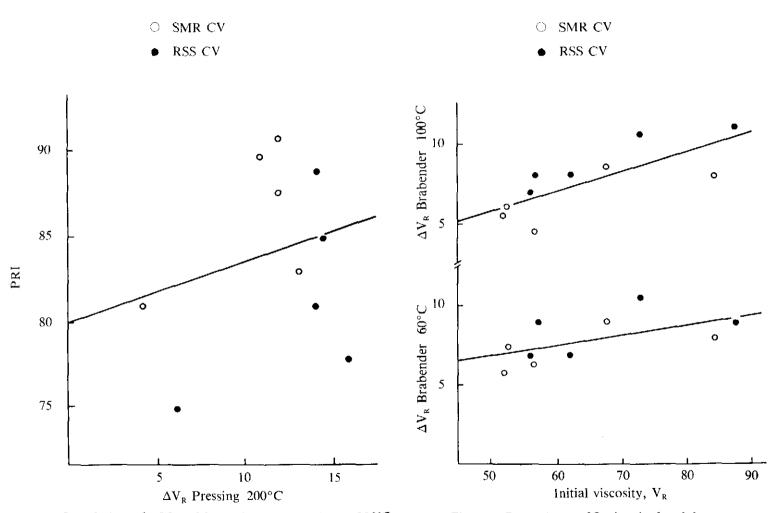


Figure 3. Correlation of PRI and breakdown in pressing at 200°C.

Figure 4. Dependence of Brabender breakdown on initial rubber viscosity.

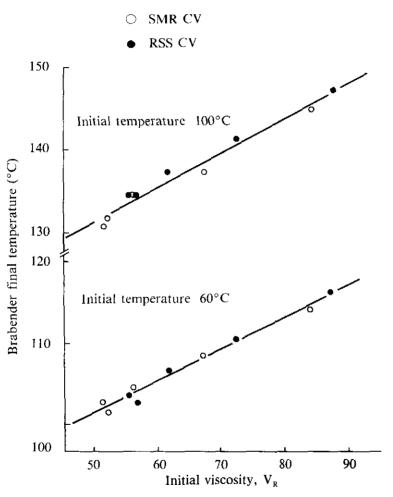


Figure 5. Dependence of Brabender dump temperature on initial rubber viscosity.

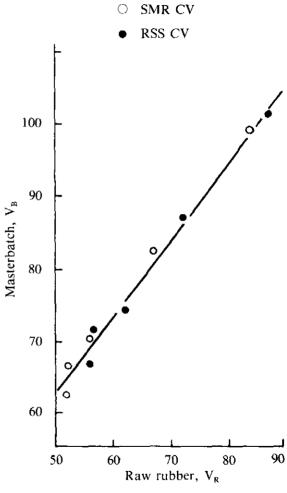


Figure 6. Dependence of tread masterbatch viscosity on raw rubber viscosity.

TABLE 5. BANBURY MIXING OF TREAD COMPOUND

			SMR CV					RSS CV		
Parameter	RRIM 600	RRIM 623	RRIM 628	RRIM 701	PR 261	RRIM 600	RRIM 623	RRIM 628	RRIM 701	PR 261
Masterbatch										
$ML1+4$, 100°C, $V_{B_{3}}$	66.5	70.5	99	62.5	82.5	71.5	74.5	101.5	67	87
Gross energy (MJ/m ³)	1 870	1 730	1 730	1 560	1 730	1 580	1 680	1 730	1 630	1 730
Dump temperature (°C)	129	130	128	127	133	128	130	132	128	133
Cabot black dispersion ^a	C1-3	B1-3	D1-3	C1-3	C1-3	D1-3	C1-3	E1-3	C1-3	D1-3
Bound rubber wt (%)	44.2	45.8	58.3	42.4	50.5	50.8	51.2	62.5	50.2	57.9
Bound rubber swell (wt/wt) ⁶	33.9	35.9	29.6	37.2	32.4	30.5	32.7	26.3	33.3	28.6
$V_{B^-}V_R$	14	14	15	10.5	15	14.5	12.5	14.5	11	14.5
Remill										
ML1 + 4, 100°C, V _{BR}	j _	_	75	_	66	_	79	_		71
Gross energy (MJ/m ³)	_	_	1 060	_	960	<u> </u>	1 030	_	_	1 060
Dump temperature (°C)	_	_	125	_	133	_	130	_		125
Cabot black dispersion	_	_	A1		A2	l —	A2	_		A2
Bound rubber wt (%)	_	_	39.2	_	38.6	_	45.3			37.5
Bound rubber swell (wt/wt)	l –	_	39.2	_	42.8	· –	36.3	_		42.9
V_{BR} - V_{R}		_	-9	_	-1.5	_	-8	-	-	~1.5
Final mix										
ML1+4, 100°C, V	48.5	45	56.5	46	52	47	52	59	51	57
Bound rubber wt (%)	30.6	28.8	35.1	30.3	35.8	33.1	35.4	35.5	33.0	37.0
Bound rubber swell (wt/wt)	51.0	49.3	51.1	52.6	50.2	42.2	41.1	47.1	46.0	50.0
$V_{c}-V_{R}$	-4	- 11.5	-27.5	-6	-15.5	- 10	-10	-28	- 5	- 15.5
Monsanto rheometer, 150°C, 1° are										
$M_{HR} - M_L (torque units)^c$	28.3	29.4	28.5	27.7	26.7	28.9	29.3	28.6	27.4	27.4
Scorch time, t _{st} (min)	4.3	3.9	3.9	4.2	4.6	3.2 ^d	3.7	4.2	4.0	4.6
Cure time, t'_{st} (min)	14.2	14.0	14.7	15.1	15.7	12.9 ^d	14.0	15.0	14.3	15.3
Cure time, t_c (95) (min)	16.3	16.3	17.1	17.5	18.1	15.1 ^d	16.4	17.4	16.6	17. 6
t _{Ri} ^e	34.5	34.0	36.0	36.5	38.0	33.0	36.0	37.5	35.5	38.0

^a Proportion of undispersed black: A (low) — H (high), particle size: 1 (small) — 6 (large)

^bWeight solvent/weight bound rubber

c 1 torque unit = 0.11 Nm

^dOmitted from analysis of Table 6

^e Time to 0.11 Nm reversion (min)

TABLE 6. FURTHER ANALYSIS OF THE DATA OF TABLE 5

	RSS	CV	SMR	CV			Δ (RSS	S CV – SM	R CV)		
Parameter	Mean	S.D.	Mean	S.D.	RRIM 600	RRIM 623	RRIM 628	RRIM 700	PR 261	Mean	S.D.
Masterbatch								<u>-</u>			
Gross energy (MJ/m ³)	1 670	65	1 720	110	-290	-50	0	+70	0	-	_
Dump temperature (°C)	130	2	129	2	1	0	+ 4	+ 1	0	1	2
ML1+4, 100°C, V _B	-	_	*	_	+ 5	+ 4	+2.5	+ 4.5	+ 5.5	+ 4.3	1.2
V_B - V_R	13.4	1.6	13.9	1.9	+ 0.5	-1.5	-0.5	+0.5	-0.5	-0.3	0.8
Bound rubber wt (%)	54.5	5.4	48.2	6.4	+6.6	+ 5.4	+4.2	+7.8	+ 7.4	+6.3	1.5
Bound rubber swell (wt/wt)	30.3	2.9	33.8	3.0	-3.4	-3.2	-3.3	- 3.9	-3.8	-3.5	0.3
Final mix											
ML1+4, 100°C, V _C	53.2	4.8	49.6	4.7	1.5	+7	+2.5	+ 5	+ 5	+3.6	3.3
$V_C - V_R$	- 13.7	8.8	-12.9	9.3	-6	+ 1.5	-0.5	+ 1	0	-0.8	3.0
Bound rubber wt (%)	34.8	1.7	32.1	3.1	+2.5	+6.6	+0.4	+ 2.7	11.2	+2.7	2.4
Bound rubber swell (wt/wt)	45.3	3.6	50.8	1.2	-8.8	8.2	-4.0	6.6	-0.2	-5.6	3.5
Monsanto rheometer, 150°C, 1° arc	1										
M _{HR} - M _L (torque units)	28.3	0.9	28.1	1.0	0.6	-0.1	0.1	-0.3	0.7	0.2	0.4
Scorch time, t _{si} (min)	3.9	0.5	4.2	0.3	-1.1ª	-0.2	0.3	-0.2	0	0	0.2
Cure time, t _c ' (90) (min)	14.7	0.6	14.7	0.7	_	0	0.3	-0.8	- 0.4	-0.2	0.5
Cure time, t_e' (95) (min)	17.0	0.6	17.1	0.8	_	0.1	0.3	-0.9	-0.5	-0.3	0.6
t _{RI}	36.0	2.0	35.8	1.6	-1.5	2	1.5	-1	o	0.2	1.5

^aOmitted from analysis of *Table 5*

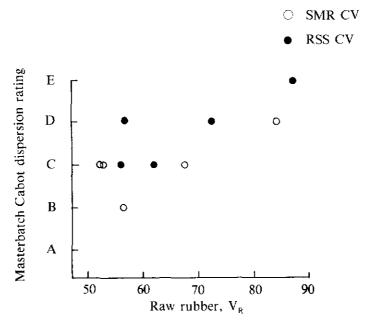


Figure 7. Dependence of masterbatch Cabot dispersion rating on raw rubber viscosity.

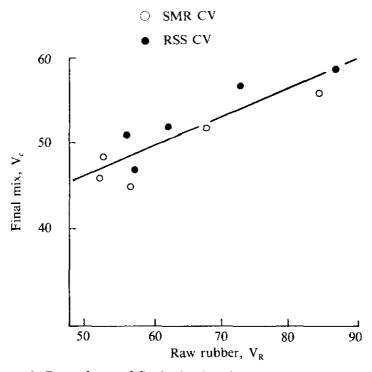


Figure 8. Dependence of final mix viscosity on raw rubber viscosity.

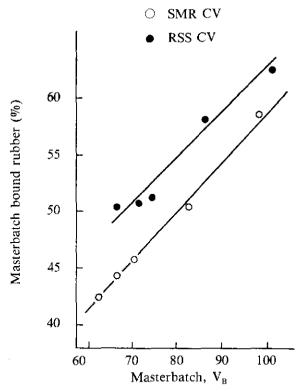


Figure 9. Dependence of masterbatch bound rubber on masterbatch viscosity.

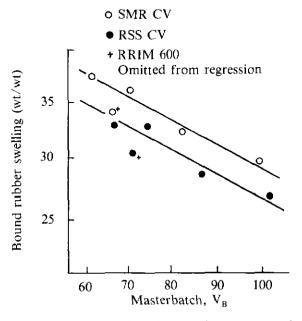


Figure 10. Dependence of bound rubber swelling on masterbatch viscosity.

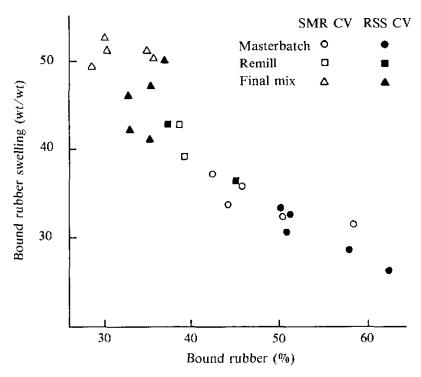


Figure 11. Correlation between bound rubber swelling and percent bound rubber for masterbatch, remill and final mixes.

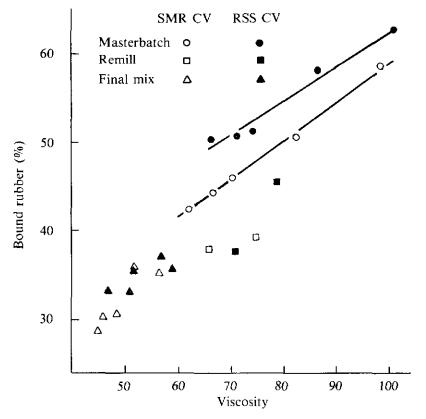


Figure 12. Dependence of percent bound rubber for masterbatch remill and final mixes on viscosity.

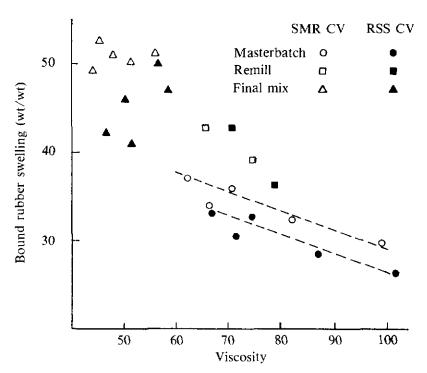


Figure 13. Dependence of bound rubber swelling for masterbatch, remill and final mixes on viscosity.

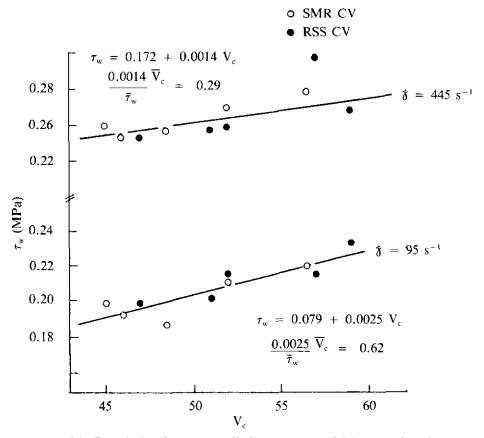


Figure 14. Correlation between wall shear stress and Mooney viscosity.

TABLE 7. CAPILLARY FLOW PARAMETERS FOR BANBURY-MIXED TREAD COMPOUND

Shear rate,	D			SMR CV					RSS CV		
τ (s ¹)	Parameter	RRIM 600	RRIM 623	RRIM 628	RRIM 701	PR 261	RRIM 600	RRIM 623	RRIM 628	RRIM 701	PR 261
95	τ ₁ (MPa)	0.240	0.244	0.271	0.236	0.256	0.250	0.264	0.282	0.250	0.267
	τ ₂ (MPa)	0.718	0.647	0.736	0.621	0.674	0.709	0.709	0.727	0.691	0.736
	τ _w (MPa)	0.187	0.199	0.219	0.193	0.210	0.199	0.215	0.233	0.201	0.215
	e	22.7	18.0	18.8	17.7	17.7	20.5	18.4	17.0	19.5	19.4
445	τ ₁ (MPa)	0.316	0.306	0.347	0.309	0.330	0.309	0.323	0.340	0.326	0.367
	τ ₂ (MPa)	1.02	0.90	1.14	0.98	1.05	0.98	1.08	1.16	1.12	1.17
	$\tau_{\rm w}$ (MPa)	0.238	0.240	0.259	0.234	0.250	0.234	0.239	0.249	0.238	0.278
	c	26.3	22.0	27.2	25.4	25.6	25.4	28.2	29.3	29.7	25.7
95	n	0.156	0.121	0.109	0.125	0.113	0.105	0.068	0.043	0.109	0.166
	S ₁ (%)	42.2	39.5	40.5	42.4	41.6	39.6	43.9	39.8	43.5	41.9
	S ₂ (%)	67.9	66.2	68.8	74,4	71.3	66.7	68.8	71.4	74.5	74.0
445	S ₁ (%)	61.8	52.2	49.0	60.3	57.8	52.1	52.9	48.1	62.7	59.5
	S ₂ (%)	134.5	99.2	102.6	133.1	124.0	101.9	111.1	104.2	143.1	140.1
	ML1+4, 100°C, V _C	48.5	45	56.5	46	52	47	52	59	51	57

 τ_1 = Total shear stress L/D = 20

 τ_2 = Total shear stress L/D = 2

 $\tau_{\rm w}$ Wall shear stress

e = End correction

n = Flow index between $\gamma = 95 \text{ s}^{-1}$ and $\gamma = 445 \text{ s}^{-1}$

 S_1 = Extrudate swell area percentage, L/D = 20

 S_2 = Extrudate swell area percentage, L/D = 2

TABLE 8. FURTHER ANALYSIS OF THE DATA OF TABLE 7

	***			· · ·	***	Ī						
Parameter	Shear rate	RSS	CV	SMR	CV		Δ (RS	SS CV — SMI	R CV)			
$\gamma_{s^{-1}}$	Mean	S.D.	Mean	S.D.	RRIM 600	RRIM 623	RRIM 628	RRIM 700	PR 261			
τ _w (MPa)	95	0.213	0.014	0.202	0.013	+ 0.012	+ 0.016	+ 0.014	+ 0.008	+ 0.005		
τ _w (MPa)	445	0.248	0.018	0.244	0.010	- 0.004	- 0.001	- 0.010	+ 0.004	+ 0.028		
e	95	19.0	1.3	19.0	2.1	- 2.2	+ 0.4	- 1.8	+ 1.8	+ 1.7		
e ·	445	27.7	2.0	25.3	2.0	- 0.9	+ 6.2	+ 2.1	+ 4.3	+ 0.1		
S_1	95	41.7	2.0	41.2	1.2	-2.6	+ 4.4	- 0.7	+ 1.1	+ 0.3		
S ₂	95	71.1	3.3	69.7	3.2	- 1.2	+ 2.6	+ 2.6	+ 0.1	+ 2.7		
S_2-S_1	95	29.3	3.2	28.5	2.5	+ 1.4	- 1.8	+ 3.3	- 1.0	+ 2.4		
S ₁	445	55.1	5.9	56.3	5,4	- 9.7	+ 0.4	. 0.9	+ 2.4	+ 1.7		
S_2	445	120,1	20.0	118.7	16.8	- 32.6	+11.9	+ 1.6	+ 10.0	+ 16.1		
$S_2 - S_1$	445	65.0	14.5	62.4	11.8	- 22.9	+11.5	+ 2.5	+ 7.6	+ 14.4		
		i .		1								

show greater discrimination. Once again, sheet and crumb-based materials conform to the same regressions. Values of the flow index, n, for a power law flow curve, have been calculated from the two data points only, and, while the precision of these values must therefore be limited, n is larger for SMR CV than for RSS CV for four of the five clones (the data for RSS CV — PR 261 appear anomolous in several respects). For RSS CV — RRIM 623 and RRIM 628, n is exceptionally low. Values of e, again estimated from only two data points at each of the two shear rates, are far more selfconsistent. Sheet and crumb materials do not differ significantly in e, and hence in the equivalent 'end pressure' $P_0 = e\pi_{ic}$

Extrudate swell follows the expected pattern, in that swell is greater at higher shear rate and the dependence on shear rate is greater for the die of low L/D ratio (Figures 15 and 16). However, over the very limited range of shear stress covered by the ten samples, there are no obvious trends of swell with stress even though the precision of the data would be expected to

be greater than the observed scatter. In these circumstances it is perhaps not surprising that systematic differences in swell between sheet and crumb-based materials are not apparent. However, the grade mean values of S_1 , S_2 and S_1 - S_2 at each of the two shear rates indicate that differences in swell due to raw rubber production procedure cannot be large.

CONCLUSIONS

The parity in properties and performance between viscosity stabilised sheet and crumb materials established for raw rubber is in large measure also found in the mixing and processing behaviour of a typical tread stock. Only in the case of the initial formation of bound rubber or carbon gel, a parameter indicative of rubber black interaction, is there evidence for an obvious effect of raw rubber production procedure, but even here this difference is eliminated in the inevitable further working during final mixing.

July 1988

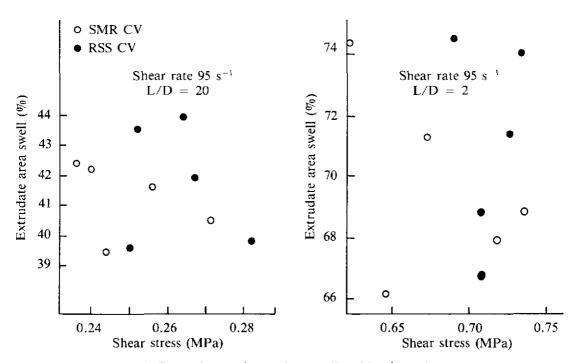


Figure 15. Dependence of extrudate swell at 95 s⁻¹ on shear stress.

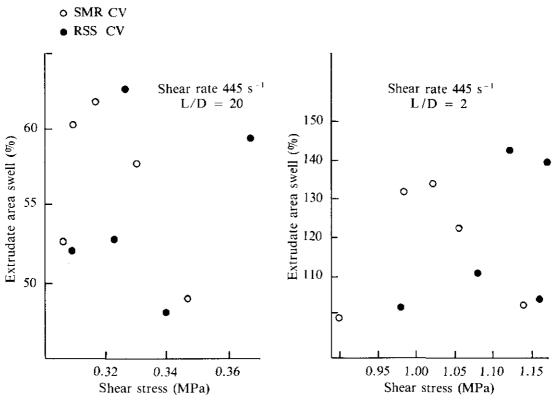


Figure 16. Dependence of extrudate swell at 445 s⁻¹ on shear stress.

REFERENCES

- BRISTOW, G.M. AND SEARS, A.G. (1988) A New Comparison of Sheet and Crumb Rubber. Part I. Raw Rubber Composition and Rheology. J. nat. Rubb. Res., 3(4), 223.
- BRISTOW, G.M. AND SEARS, A.G. (1984) Correlation between Raw Rubber Breakdown and Mixing Performance in an ISAF Tread Stock. NR Technol., 15(1), 1.
- LIM, H.S. AND LIM, C.L. (1985) A Test for Assessing the Breakdown and Mixing Behaviour of Viscosity-stabilized Grades of Natural Rubber. Proc. Int. Rubb. Conf. 1985 Kuala Lumpur, 2, 225.
- RUBBER RESEARCH INSTITUTE OF MALAYSIA (1970) Test Methods for Standard Malaysian Rubbers. SMR Bull. No. 7.

- VAN BUSKIRK, P.R., TURETZKY, S.B. AND GUNBERG, P.F. (1975) Practical Parameters for Mixing. Rubb. Chem. Technol., 48, 577.
- SEARS, A.G. (1987) Unpublished data. Malaysian Rubber Producers' Research Association.
- MEDALIA, A.I. AND WALKER, D.P. Evaluating Dispersion of Carbon Black in Rubber. Technical Report RC-124, Revision No. 2. USA: Cabot Corporation.
- LIM, C.L. AND ONG, E.L. (1985) Breakdown Behaviour of Natural Rubber and Its Influence on Processability. Proc. Int. Rubb. Conf. 1985 Kuala Lumpur, 2, 557.
- SEARS, A.G. (1987) Unpublished data. Malaysian Rubber Producers' Research Association.