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Production of SMR 5 CV from High Viscosity Rubbers by Chemical Peptisation

The paper describes a simple and inexpensive process for the production of SMR 5 CV from high viscosity rubbers by xylyl mercaptan treatment and discusses the vulcanisate properties of such treated rubbers. It also discusses the influence of various chemical peptisers on raw rubber properties under prevailing conditions of block rubber manufacture.

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SMR 5CV with a Mooney viscosity of 55—65 units accounts for over 95% of constant viscosity (CV) rubbers. It normally commands a premium and is the most saleable latex grade. In commercial production, it is necessary to blend clonal latices judiciously to obtain the required viscosity range of SMR 5CV[1-3]. Occasionally, latices with high Mooney rubber cannot be used for CV production. For this reason there is commercial interest for an inexpensive and simple way to reduce the viscosity of rubber. The need for this will be greater in the future as the present trend in Malaysia is towards centralised processing and SMR 5CV is the most economically attractive latex grade for processing in the factories.

Chemical peptisers have been used in the mastication process to lower the viscosity of NR in consumer factories. However, these have not been exploited in the processing of NR by the new process because the following features are generally accepted:

- The high drying temperatures (100°C—110°C) used in the process of preparing block rubbers will result in difficulties in controlling the viscosity reduction of the rubber.
- The plasticity retention index (PRI), a specified property of the raw rubber in the SMR scheme, may be adversely affected.

A systematic investigation was initiated to reassess the feasibility of using chemical peptisers to reduce the viscosity of CV rubbers. This paper discusses the results obtained from this investigation. The peptisers studied included Renacit IV, Renacit VII, Pepton 22, Pepton 65, xylyl mercaptan (active component of RPA 3) and phenylhydrazine hydrochloride.

EXPERIMENTAL

Preparation of Rubbers

Field latex was sieved through a 40 mesh sieve. The required amount of castor oil was stirred into the latex. This was followed by the addition of sodium metabisulphide (0.04% by weight on the rubber, added as 5% aqueous solution), hydroxylamine neutral sulphate (0.15% by weight on the rubber, added as 10% aqueous solution) and the desired amount of chemical peptisers (added either as dispersion, an emulsion or an aqueous solution). The latex was then coagulated with 2% formic acid at pH 5—5.2. The coagulum was matured for 3 h to 10 h before it was reduced to crumbs. The crumbs were then dried by hot air circulated at the desired temperature and time.

Preparation of Compound

All the antioxidants, accelerators and other compounding ingredients were commercial

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materials and were used as supplied. The ACS-1 mix was prepared on 152.4 × 304.3 mm two-roll laboratory mill. The starting roll temperature was 70°C.

The HAF black tread mix was prepared in the 00C Banbury according to the schedule below:

0 min – Add rubber, black oil and powders
2 min – Sweep
3 min – Dump

Sulphur and accelerator were added on the mill at 60°C.

The optimum cure time (at 90% crosslink) of the tread mix was determined by Monsanto Rheometer TM100.

The physical testing of the vulcanisate properties was carried out according to BS 903 Part A2 1971.

RESULTS AND DISCUSSION

Assessment of Chemical Peptisers

An ideal chemical peptiser for reducing the viscosity of CV rubbers should have the following characteristics:

- A high efficiency in reducing the viscosity of rubber.
- Efficiency not sensitive to variation in drying temperature and time.
- No adverse effect on the raw and vulcanisate properties of the rubber.

In the investigation, the suitability of the chemical peptiser for reducing the viscosity of CV rubbers was initially assessed in accordance with the above criteria. Consequently, the influence of the level of peptiser, drying temperature and time on the viscosity and properties of rubber were studied.

Efficiency of Chemical Peptiser

The efficiency of the chemical peptiser in reducing the viscosity of the rubber was assessed by studying the influence of the level of peptiser on the viscosity of the rubber. This is illustrated in *Figure 1.*

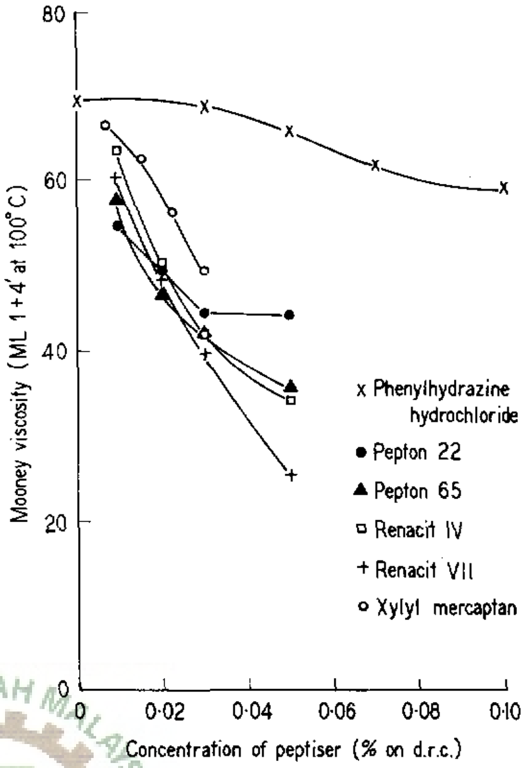

Figure 1. Effect of peptiser on Mooney viscosity.

Renacit IV, Renacit VII, Pepton 22, Pepton 65 and xylyl mercaptan have comparable efficiencies and all are more efficient than phenylhydrazine hydrochloride.

Effect of Drying Temperature

Figure 2 illustrates the sensitivity of the efficiency of chemical peptisers to the variation in drying temperature. Xylyl mercaptan, phenylhydrazine hydrochloride, Renacit IV and Renacit VII are relatively less sensitive to variation in drying temperature than Pepton 22 and 65.

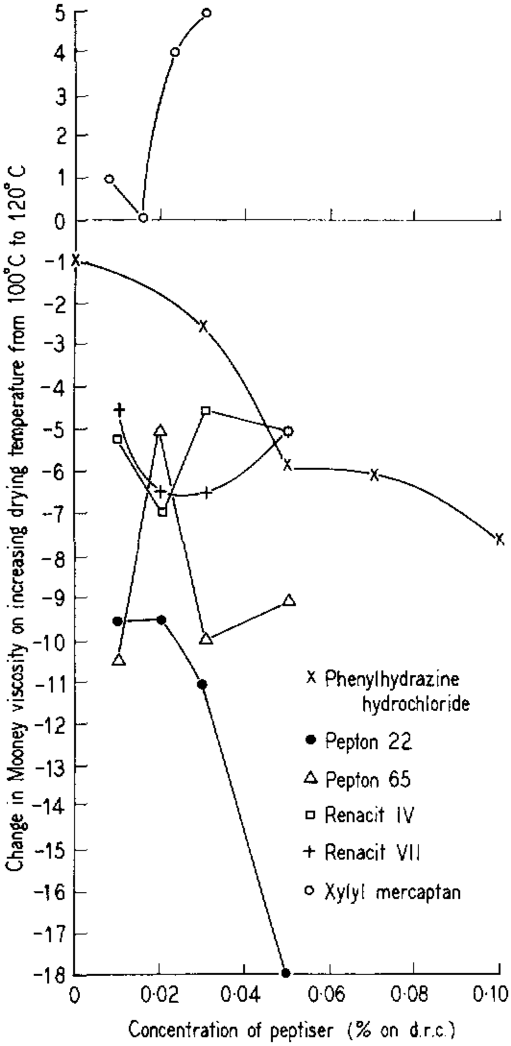

Figure 2. Effect of drying temperature on the sensitivity of efficiency of various peptisers.

Effect of Drying Time

The sensitivity of the efficiency of chemical peptisers and the variation in drying time are illustrated in *Figure 3.* Xylyl mercaptan, phenylhydrazine hydrochloride and Pepton 65 are relatively less sensitive to drying time variation than Pepton 22, Renacit IV and Renacit VII.

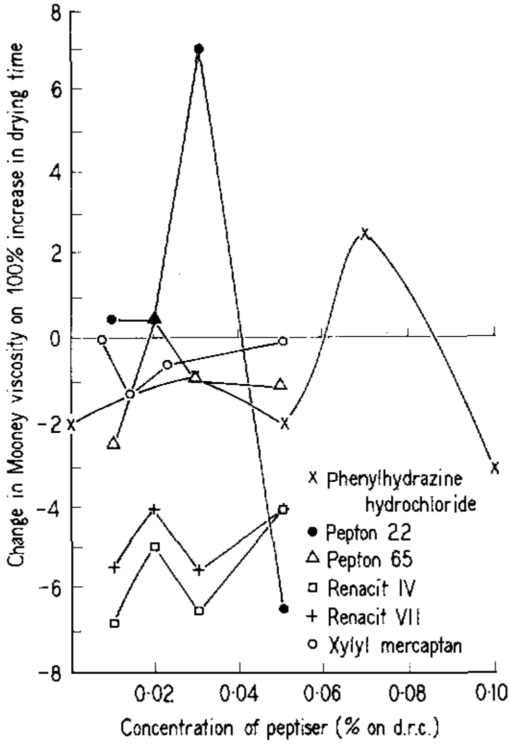

Figure 3. Effect of drying time on the sensitivity of efficiency of various peptisers.

Effect on Plasticity Retention Index (PRI)

The chemical peptiser, besides reducing the viscosity, is generally believed to affect the PRI of the rubber. Consequently, the influence of various chemical peptisers on PRI was investigated and the results are illustrated in *Figure 4.* Pepton 22 and Pepton 65, even at very low levels, adversely affect the PRI. However, Renacit IV, Renacit VII, xylyl mercaptan and phenylhydrazine hydrochloride show little effect on the PRI at levels normally used.

DETAILED EVALUATION OF XYLYL MERCAPTAN

The data given in *Figures 1* to *4* are summarised in *Table 1.* This preliminary study indicates that among the peptisers investigated,

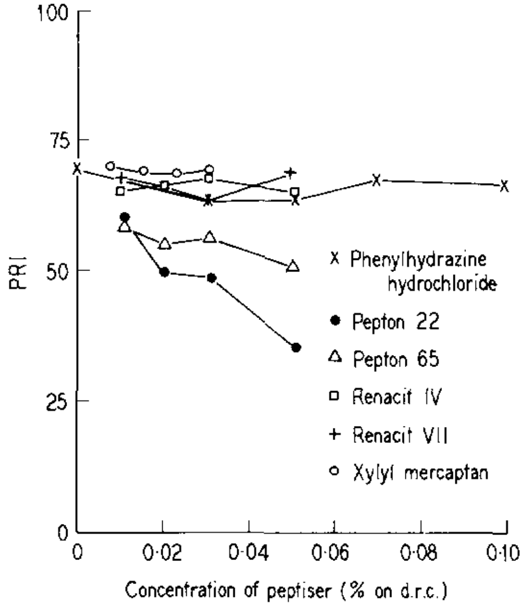

Figure 4. Effect of peptiser on PRI.

xylyl mercaptan is best suited to reduce the viscosity of CV rubbers. As such, only xylyl mercaptan was investigated further.

Clonal Effect

To study the effect of clones on the efficiency of xylyl mercaptan in reducing the viscosity of rubber, two clones, *viz.* PB 86 and GT 1 which produce high viscosity rubber, were chosen.

Figure 5 shows that xylyl mercaptan has different efficiencies in the reduction of viscosity of rubber from different clones. However, the efficiency is consistent for a specific clone. Hence, preliminary investigations must be carried out on a specific bulk of latex before xylyl mercaptan can be used in factory-scale operations for reduction of viscosity in CV rubbers.

Raw Rubber Properties

The raw rubber properties of the rubber treated with different levels of xylyl mercaptan are shown in *Table 2.* Xylyl mercaptan reduces the viscosity of the rubber without any significant adverse effects on the raw rubber properties.

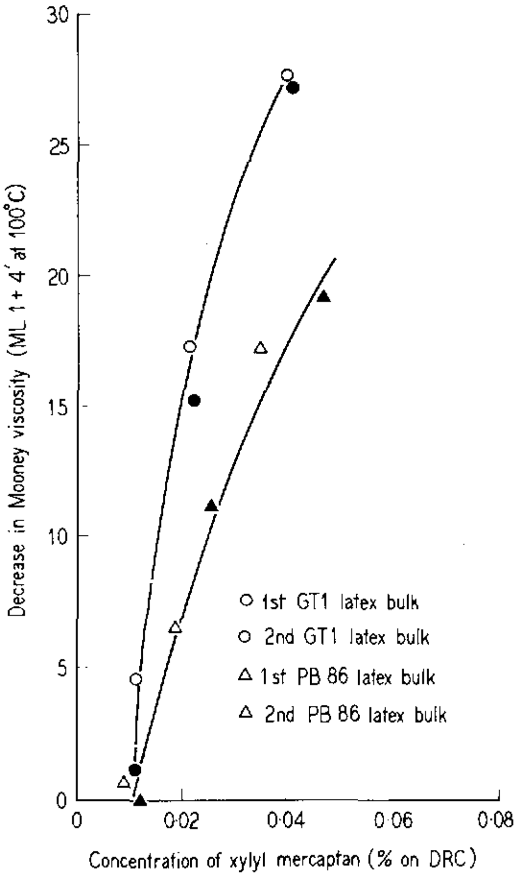

Figure 5. Peptisation of two clonal rubbers by xylyl mercaptan.

Vulcanisate Properties

The rubber treated with different levels of xylyl mercaptan was evaluated in the ACS 1 and HAF tread mix.

To examine the effect of xylyl mercaptan treatment on the technological properties of the rubbers, mixings were carried out on the rubbers, without premastication. All rubbers, despite their differences in viscosity, show comparable physical properties in the ACS-1 mix *(Table 3)*. Evaluation in a black tread mix

TABLE 1. ASSESSMENT OF CHEMICAL PEPTISERS

Chemical peptiser	*Efficiency*	Sensitivity to drying temp.	Sensitivity to drying time	Effect on *PRI*
Xylyl mercaptan	High	Low	Low	No adverse effect
Phenylhydrazine hydrochloride	Low	Low	Low	No adverse effect
Renacit IV	High	Low	Medium	No adverse effect
Renacit VII	High	Low	Medium	No adverse effect
Pepton 22	High	Medium	Medium	Adverse effect
Pepton 65	High	Medium	Low	Adverse effect

TABLE 2. RAW RUBBER PROPERTIES OF RUBBER TREATED WITH DIFFERENT LEVELS OF XYLYL MERCAPTAN

Sample reference	Level of xylyl mercaptan (% d.r.c.)	Dirt (% wt)	Volatile matter (% wt)	P_0	PRI	Ash (% wt)	Nitrogen (% wt)	Mooney viscosity (ML 1 + 4 at 100°C)
1	0	0.009	0.30	48.5	85	0.24	0.39	74
2	0.013	0.004	0.32	34.5	75	0.23	0.38	62
3	0.018	0.004	0.32	31.5	78	0.23	0.38	58
4	0.022	0.006	0.32	26.5	77	0.23	0.38	55

TABLE 3. VULCANISATE PROPERTIES OF ACS-1 MIX

Property (40 min at 140°C cure)	Sample reference 1	2	3	4
Tensile strength ($MN\ m^{-2}$)	17.6 (133)	18.4 (129)	18.4 (127)	19.2 (124)
Elongation at break (%)	880 (89)	870 (90)	900 (84)	900 (84)
Modulus at break (%)	0.94 (152)	0.85 (166)	0.88 (165)	0.91 (155)
Relaxed modulus (MR 100, $MN\ m^{-2}$)	0.54	0.53	0.53	0.52
Dunlop resilience at 20°C (%)	80.9	80.5	80.3	80.1
Hardness (IRHD)	33.2	32.7	33.0	32.7

Figures within brackets indicate percentage retention after seven days of ageing at 70°C.

Compound formulation	Parts by weight
Natural rubber	100.0
Zinc oxide	6.0
Stearic acid	0.5
MBT	0.5
Sulphur	3.5

shows comparable physical properties except for a slight increase of modulus observed with rubbers of lower viscosity *(Table 4)*. Under the ageing condition of seven days at 70°C in an air oven, the xylyl mercaptan treatment does not impart any significant adverse effect on the ageing properties. Further, the reversion property of the rubbers as assessed by the Monsanto rheometer on the HAF tread mix at 150°C was not affected by the xylyl mercaptan treatment *(Figure 6)*.

CONCLUSION

Viscosity of CV rubbers can be reduced by the addition of xylyl mercaptan to latex prior to coagulation. The process has commercial potential since

- The process is simple and inexpensive.
- Xylyl mercaptan is readily available and acceptable to consumers, since it is presently used in the production of pale and sole crepes.

TABLE 4. VULCANISATE PROPERTIES OF HAF BLACK TREAD MIX

Property at 90% optimum cure at 140°C	Sample reference 1	2	3	4
Tensile strength (MN m^{-2})	28.2 (98)	28.1 (108)	28.0 (105)	28.4 (100)
Elongation at break (%)	600 (87)	590 (86)	590 (86)	600 (83)
Modulus 300% (MN m^{-2})	10.3 (156)	10.9 (150)	11.4 (146)	11.3 (140)
Relaxed modulus (MN m^{-2})	1.60	1.66	1.71	1.73
Dunlop resilience at 20°C (%)	62.6	61.4	61.7	63.0
Hardness (IRHD)	63.9	63.2	63.4	64.3
Goodrich flexometer heat build-up (optimum cure time + 5 min)				
Time to give temperature of 50°C (min)	32	30	31	29
Time to give temperature of 100°C (min)	62	48	51	45
Relative resilience (%)	65.9	65.9	63.1	63.1
Compression set 24 h at 70 ± 1°C (%)	10.1	9.2	10.7	10.6

Flexometer starting condition
Stroke—4.45 mm
Applied force—13.6 kg
Figures within brackets indicate percentage retention after seven days of ageing at 70°C.

Compound formulation	Parts by weight
Natural rubber	100
Zinc oxide	4
Stearic acid	2
HAF	50
Dutrex R	5.0
Nonox ZA	1.5
Sulphur	2.5
CBS	0.5

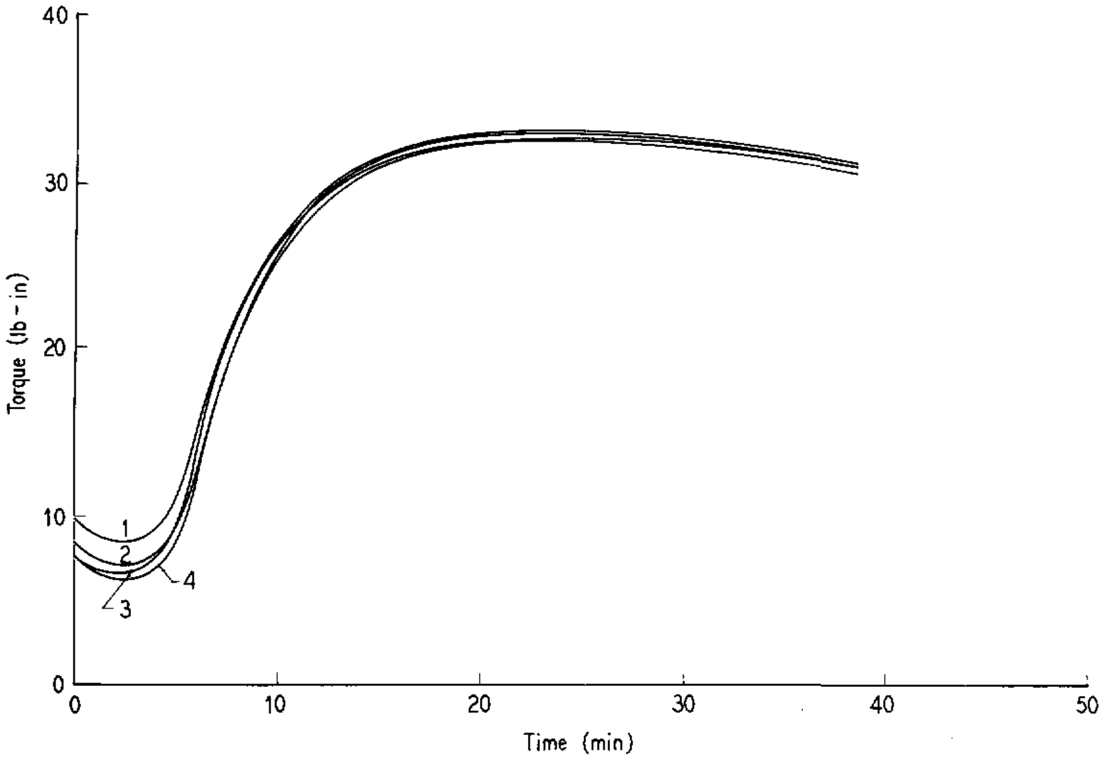

Figure 6. Rheometer curve of the HAF tread mix.

- Preliminary investigations indicate that xylyl mercaptan reduces the viscosity of rubber without any significant adverse effects on the raw rubber and technological properties.

However, further technological investigations and consumer evaluations are required before the process can be recommended for adoption by the industry.

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