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Storage Behaviour of Crated Tyre Rubber: Effect of Baling Temperature and Viscosity Stabilisers

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The storage behaviour of Tyre Rubber in a 1-tonne crate is studied. The viscosity of Tyre Rubber can be stabilised effectively by the use of semicarbazide and hydroxylamine but not formaldehyde. The increase in viscosity is normally less than 5 Mooney units Most of the increase occurs during the first two months of storage in Malaysia. The baling temperature has a marked influence on the rate and the absolute level of increase in viscosity. A baling temperature of less than 60°C is satisfactory for checking this increase in viscosity.

The storage behaviour of natural rubber has been studied extensively by many workers. The increase in viscosity on storage has been attributed to the crosslinking of aldehydic groups present on the main chain with aldehyde condensing groups in the non-rubbers or on an adjacent molecule[1,2,3]. This process of storage hardening goes on for many years and can be accelerated by storage at elevated temperature and low humidity. Sekhar[4] has found numerous chemicals which are effective in the inhibition of this storage hardening process.

Much of the work done has been based on small samples or single bales. The storage behaviour of small samples or single bales may not reflect that of rubber stored in 1 tonne SMR crates. Under Malaysian ambient conditions, the dominant influence on the storage hardening behaviour of rubber stored in crates, is expected to be the temperature of the baled rubber. When rubber is baled hot at about 100°C and placed in a crate, the rubber at about the centre of the crate takes about two months to cool to room temperature; rubber baled at 55°C takes about one month to cool*. The conservation of heat in the rubber would be expected to have a dominant influence in the rate of storage hardening.

*The drop in temperature was monitored by using a Rotatherm thermocouple with range 0°C to 120°C.

In 1972, Tyre Rubber was introduced to consumers as a blend of latex (30%), unsmoked sheets (30%), cuplump (30%) and process oil (10%). It was necessary to stabilise the viscosity of Tyre Rubber in order to confer the required easy processing characteristics[5]. Previous work had shown that hydroxylamine salts, semicarbazide and formaldehyde can stabilise the viscosity of latex rubber[4]. Information on their effect on a blend such as Tyre Rubber was, however, lacking.

This paper reports on studies made on the efficiency of the mentioned chemicals on the storage behaviour of Tyre Rubber. It also highlights the influence of baling temperature on the efficiency of such chemical treatments. Storage behaviour is assessed by observing the change in Mooney viscosity, Wallace plasticity and PRI of the rubber.

MATERIALS AND METHODS

Five series of trials were made. These are detailed below.

Series I. Tyre Rubber A was treated by dipping the wet crumbs in semicarbazide solution. The concentration of the semicarbazide solution varied from 0% to 0.9% weight. One bale per treatment was used in this trial.

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Series II. Tyre Rubber A was treated by dipping the wet crumbs in semicarbazide solution. Tyre Rubber A was treated with semicarbazide solutions ranging from 0.8% to 1.5% weight. Two bales per treatment of Tyre Rubber were used.

Series III. Tyre Rubber and its components were treated by dipping the wet crumbs in 1.2% semicarbazide solution. Two bales per treatment of Tyre Rubber and one bale per treatment of the components were used.

Series IV. Tyre Rubber A was treated by adding hydroxylamine neutral sulphate to the latex component. The hydroxylamine concentration ranged from 0.08% to 0.5% wt on total rubber plus oil. Three bales per treatment were used.

Series V. Tyre Rubber A was treated by dipping the crumbs in formaldehyde solution. The strength of the formaldehyde solution varied from 1% to 5% weight.

In experiments involving the dipping technique, 50 gallons of semicarbazide solution were used to treat one to three bales of Tyre Rubber crumbs. To prevent dilution of the dipping solution, the wet crumbs were allowed to drip until the crumb surfaces were not wet to the touch of hand. As a precautionary measure, the strength of the solution was checked after dipping every bale-equivalent of crumbs and topped up if necessary. In practice, no significant reduction in strength was observed. Where formaldehyde was used, the dipping solution was obtained by diluting the BDH reagent to the required level.

The crumbs were dried in the usual manner to achieve a volatile matter content of below 0.4% weight. The temperature of the dry crumbs was adjusted by blowing air through the bed of dry crumbs. It took less than half an hour to reach 35°C. After baling, the bales were stored in the third and fourth layers of the 1-tonne ISO crate. The rest of the crate was made up of rubber from normal production but baled at the temperature appropriate to the storage test. Rubbers baled at three temperatures, 100°C, 55°C and 35°C were studied. A laboratory mercury-in-glass thermometer (0°C to 150°C) was used to measure the temperature of the rubber biscuits.

In preparing the experimental samples, the raw materials were thoroughly blended to ensure uniformity between the rubbers.

The baled rubbers were sampled after different periods of storage at ambient conditions. The trials were terminated after four months because the rate of change in viscosity of the rubber had leveled out. Test samples were removed from bale corners kept at the inner (central) portion of the pallet and from corners kept at the outer portion of the pallets; one inner and two outer corners were sampled. After sampling, which normally took half an hour, the bales were placed back in the same position that they previously occupied in the pallet. The same inner and outer corners were used on each sampling occasion.

All samples were blended by using the ISO procedure and tested for Mooney viscosity, Wallace plasticity and PRI. The storage behaviour of Tyre Rubber was assessed principally by the change in Mooney viscosity over time. Data on Wallace plasticity and PRI were also tabulated in the *Appendix* for completeness.

Viscosity Stabilisation by Semicarbazide Treatment

In these series of trials, the objectives were to study the effect of temperature, to determine the most cost-effective method of achieving viscosity stabilisation and to assess the contributions of the various components towards the increase in viscosity.

Effect of temperature. In the *Series I* trial, the crumbs were dipped in semicarbazide solutions of strength varying between 0.3% to 0.9% weight. *Table 1* shows that a solution of 0.3% to 0.5% strength is inadequate to stabilise the viscosity of Tyre

TABLE 1. EFFECT OF BALING TEMPERATURE AND SEMICARBAZIDE TREATMENT ON INCREASE OF MOONEY VISCOSITY

Semicarbazide solution (% wt)	Baling temperature (°C)	Storage-hardening test (△ P)	Initial viscosity	Viscosity increase after storage (month) One	Two	Three	Four
0	35	11.5	64	3	3	4	6
	55	14.0	65	5	5	8	8
	100	12.5	67	5	5	7	8
0.3	35	10.0	60	5	2	3	5
	55	7.5	59	5	6	6	7
	100	8.0	60	5	4	5	7
0.5	35	7.5	56	2	1	3	4
	55	8.0	57	4	4	5	5
	100	7.0	58	4	3	5	5
0.9	35	1.5	57	0	0	2	3
	55	2.5	57	3	1	3	3
	100	2.0	58	3	2	4	4

Viscosity data is average of the inner and the outer corners of a single bale.

Rubber at the required level. A dipping solution of 0.9% wt strength is required for adequate stabilisation.

Most of the increase in viscosity occurs during the first two months of storage. During this period the baling temperature has a dominant influence; the lower the baling temperature the lower the initial viscosity and also the lower the rate of increase *(Table 1)*. This influence of baling temperature is also evident in the higher viscosity of the inner corners compared with the outer corners of bales, especially when the baling temperature is 55°C or above *(Table 2)*.

Cost-effective method. In *Series II* trials, the objective was to determine the best method of stabilising the viscosity of Tyre Rubber. *Table 3* shows that at low baling temperature (35°C), a 0.8% wt solution of semicarbazide provides adequate control of the increase in viscosity. If the rubber is baled hot (100°C), the strength of the dipping solution must be nearly doubled to 1.5% wt before adequate stabilisation can be achieved. Therefore, the most cost-effective method of viscosity control is to use the minimum strength of semicarbazide solution and to bale the rubber at 35°C. Since Tyre Rubber contains 10% of oil, it flows better; therefore baling at 35°C presents no difficulty.

Contribution of component rubbers. In *Series III* trials, the relative contribution of the component rubbers towards viscosity increase of Tyre Rubber A was assessed. *Table 4* shows that most of the observed increase in viscosity of Tyre Rubber is contributed by the unsmoked sheet, followed by the cuplump component.

Viscosity Stabilisation by Hydroxylamine.

In *Series IV* trials, the interaction of baling temperature and hydroxylamine on the effective control of the increase in Mooney viscosity was studied. The hydroxylamine levels ranged from 0.08% to 0.50% wt on total rubber plus oil content. *Tables 5, 6* and *7* summarise the test results and indicate that:

- Most of the increase in viscosity occurs during the first two months of storage.

TABLE 2. DIFFERENCE IN MOONEY VISCOSITY BETWEEN INNER AND OUTER BALE CORNERS OF TYRE RUBBER A STABILISED BY SEMICARBAZIDE

Semicarbazide solution (% wt)	Baling temperature (°C)	Storage period (month) One	Two	Three	Four
0	35	+1	+1	+1	+2
	55	+1	+1	0	+3
	100	+5	+2	+2	+1
0.3	35	−1	+1	0	+2
	55	+1	+1	+1	+1
	100	+1	+2	+2	+1
0.5	35	+1	+2	+1	+1
	55	+1	+1	+1	+1
	100	+3	+2	+3	+4
0.9	35	0	0	+1	−1
	55	+2	0	−1	0
	100	+3	0	−1	+3

A positive difference indicates that the viscosity of the inner corner is higher than that of the outer corner.

TABLE 3. EFFECT OF BALING TEMPERATURE AND SEMICARBAZIDE TREATMENT ON INCREASE OF MOONEY VISCOSITY

Semicarbazide solution (% wt)	Baling temperature (°C)	Storage-hardening test (△P)	Initial viscosity	Viscosity increase after storage (month) One	Two	Three	Four
0.8	35	5.0	57	4	4	3	3
	55	5.5	55	3	4	4	4
	100	5.5	56	5	7	6	6
1.0	35	5.0	56	4	4	4	4
	55	2.5	57	3	4	4	3
	100	4.5	57	5	6	6	6
1.5	35	3.0	57	2	3	2	3
	55	3.5	55	4	4	5	5
	100	1.5	56	4	4	3	4

Viscosity data is average of the inner and the outer corners of two bales.

- A low baling temperature effectively controls the rate of increase in viscosity of Tyre Rubber.
- The higher the hydroxylamine content the lower the initial viscosity.
- At the low baling temperature of 35°C, a hydroxylamine content of 0.3% wt is adequate to control the increase in viscosity to about 5 Mooney units; a slightly lower chemical dosage is also satisfactory.

TABLE 4. CONTRIBUTION OF RUBBER COMPONENTS TO INCREASE IN VISCOSITY ON STORAGE (STABILISATION BY SEMICARBAZIDE)

Rubber	Baling temperature (°C)	Storage-hardening test (△P)	Viscosity increase after storage (month) One	Two	Three	Four
Tyre Rubber A	35	4.0	4	4	3	3
	55	3.0	4	4	4	4
	100	4.5	5	6	6	7
Latex rubber	35	6.5	2	3	3	3
	55	7.5	2	3	3	3
	100	7.5	4	5	4	4
Unsmoked sheet rubber	35	6.0	4	5	6	6
	55	6.0	6	6	7	7
	100	4.0	6	8	7	9
Field coagulum rubber	35	3.5	4	5	4	4
	55	4.0	3	3	5	4
	100	7.0	5	6	6	6

Viscosity data is average of the inner and the outer corners of a single bale.

TABLE 5. EFFECT OF BALING TEMPERATURE AND HYDROXYLAMINE TREATMENT ON INCREASE OF MOONEY VISCOSITY

Hydroxylamine level (% w/w)	Baling temperature (°C)	Storage-hardening test (△P)	Initial viscosity	Viscosity increase after storage (month) One	Two	Three	Four
0	35	8.0	66	0	+5	+4	+4
	55	6.5	67	+3	+5	+5	+4
	100	8.0	68	+5	+4	+6	+4
0.08	35	9.5	62	0	3	4	3
	55	4.5	63	1	2	3	1
	100	5.5	63	3	3	6	3
0.15	35	4.5	61	−1	3	3	2
	55	3.0	62	0	2	3	1
	100	3.0	62	4	4	4	3
0.30	35	4.0	56	4	5	6	5
	55	4.5	60	1	1	2	2
	100	4.0	62	3	1	2	4
0.40	35	4.0	56	0	4	5	6
	55	1.5	59	1	1	3	1
	100	2.5	60	1	2	3	1
0.50	35	2.5	55	2	5	3	5
	55	2.0	57	0	1	3	0
	100	3.5	60	0	1	1	−1

Viscosity data are the mean values of the inner and the outer corners of three bales.

TABLE 6. EFFECT OF BALING TEMPERATURE AND HYDROXYLAMINE TREATMENT ON INCREASE OF MOONEY VISCOSITY

Hydroxylamine level (% wt)	Baling temperature (°C)	Storage-hardening test (△P)	Initial viscosity	Viscosity increase after storage (month)			
				One	Two	Three	Four
0.3	35	6.5	56	−1	1	4	1
	55	4.5	56	0	1	2	1
	100	4.0	54	5	5	6	3
0.4	35	6.5	58	2	1	0	3
	55	4.5	59	2	1	2	0
	100	5.0	58	4	4	3	2
0.5	35	3.5	53	3	3	3	5
	55	4.0	53	3	3	4	3
	100	5.0	54	3	3	5	2

Viscosity data are the mean values of the inner and the outer corners of four bales.

TABLE 7. DIFFERENCE IN MOONEY VISCOSITY BETWEEN INNER AND OUTER CORNERS OF BALE OF TYRE RUBBER A STABILISED BY HYDROXYLAMINE

Hydroxylamine level (% wt)	Baling temperature (°C)	Storage period (month)			
		One	Two	Three	Four
0.3	35	0	+1	0	0
	55	+1	0	+1	+1
	100	+1	+1	+1	+1
0.4	35	−1	0	0	0
	55	+1	+1	−1	0
	100	+1	+1	+1	+1
0.5	35	0	0	+1	0
	55	+1	0	0	0
	100	+1	+1	0	+1

A positive difference indicates that the viscosity of the inner corner is higher than that of the outer corner.

- If the baling temperature is not controlled, the hydroxylamine content must be increased to 0.5% wt before effective control of viscosity is possible.

Viscosity Stabilisation by Formaldehyde

Previous work has indicated that formaldehyde could possibly be used to stabilise the viscosity of rubber. This method of stabilisation was tested with Tyre Rubber. Since formaldehyde can flocculate latex, the technique of adding formaldehyde to latex was not suitable. Instead Tyre Rubber crumbs were dipped in formaldehyde solutions of strength up to 5% weight.

Table 8 shows that Tyre Rubber cannot be stabilised adequately by dipping in formal-

TABLE 8. EFFECT OF BALING TEMPERATURE AND FORMALDEHYDE TREATMENT ON INCREASE OF MOONEY VISCOSITY

Formaldehyde solution (% wt)	*Baling temperature (°C)*	*Storage-hardening test (△ P)*	Initial viscosity	Viscosity increase after storage (month)			
				One	Two	Three	Four
1	35	8.5	65	0	4	4	6
	55	4.0	68	1	2	3	1
	100	7.0	66	5	7	8	2
2	35	8.5	65	0	2	4	2
	55	5.0	68	1	2	2	1
	100	8.0	65	4	8	8	7
3	35	10.0	64	1	7	6	5
	55	7.0	68	3	2	4	3
	100	6.5	66	4	6	5	6
4	35	11.0	65	1	4	5	4
	55	8.5	69	0	2	3	1
	100	7.0	68	6	6	6	4
5	35	10.0	66	1	3	3	3
	55	7.5	68	3	3	4	3
	100	9.0	70	4	5	4	3

Viscosity data is the average of the inner and the outer corners of three bales.

dehyde solutions of less than 4% strength; the initial viscosity and the subsequent rate of increase in viscosity on storage are unacceptable. Working with solutions stronger than 5% is difficult because of the pungent fumes of the formaldehyde. Also, the technique would not be as economical as the techniques described earlier.

Wallace Plasticity and PRI

The data on Wallace plasticity (P_0) and PRI for some trials were obtained. These tend to be more variable and consequently less conclusive. P_0 and PRI data of *Series I* and *IV* are given in the *Appendix*. The variability could be due to testing errors since the experiments were carried out over four months.

CONCLUSION

Semicarbazide and hydroxylamine neutral sulphate are effective in inhibiting the increase in viscosity of Tyre Rubber on storage; formaldehyde is unsuitable. The temperature of the rubber during baling and packing plays a critical role in the rate of increase in viscosity on storage; it also determines the level of viscosity-stabilisers needed for effective stabilisation. Lower levels of chemical treatment can be adopted when the baling temperature is kept low (below 40°C).

When using semicarbazide, the dipping solution must be at least 0.8% wt strength and the baling temperature must not exceed 60°C. Of the three components, unsmoked sheets contribute most to the increase in viscosity on storage; the next highest contribution is by cuplump rubbers.

When hydroxylamine neutral sulphate is used, 0.3% wt of the chemical based on total weight of product is adequate for viscosity stabilisation. There are indications that chemical usage at 0.15% strength is acceptable.

When treated adequately with semicarbazide or hydroxylamine, the viscosity of Tyre Rubber stabilises three or four months after storage. The extent of the increase is less than 5 Mooney units if the baling temperature is kept below 60°C. This is confirmed by tests on Tyre Rubber which has been stored in England for more than twelve months[6,7]. This order of increase is similar to that observed with SMR 5CV kept for four years[8].

Tyre Rubber should not be baled at temperature exceeding 60°C; this ensures that the viscosity increase will still be acceptable even if the viscosity-stabiliser used is on the low side. The occurrence of torn polyethylene wrappers is also reduced.

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APPENDIX

TABLE I. CHANGE IN WALLACE PLASTICITY OF TYRE RUBBER TREATED WITH SEMICARBAZIDE

Semicarbazide solution (% wt)	Baling temperature (°C)	Initial P_0	Change in P_0 after storage (month) One	Two	Three	Four
0	35	38	+5	+3	−3	−
	55	37	+9	+5	+2	−
	100	37	+10	+6	+3	−
0.3	35	34	+6	+3	−1	−
	55	35	+6	+3	0	−
	100	33	+9	+6	+1	−
0.5	35	33	+4	+4	−1	−
	55	32	+6	+8	+1	−
	100	33	+7	+5	0	−
0.9	35	33	+2	+1	−1	−
	55	32	+4	+4	−1	−
	100	32	+4	+3	−2	−

Above data relate to same experiment as *Table 1* of text.
The decrease recorded for month three is probably due to testing error.

TABLE II. CHANGE IN PRI OF TYRE RUBBER TREATED WITH SEMICARBAZIDE

Semicarbazide solution (% wt)	Baling temperature (°C)	Initial PRI	Change in PRI after storage (month) One	Two	Three
0	35	83	+2	+7	+7
	55	89	−9	−1	−6
	100	86	−6	−1	−6
0.3	35	88	−3	+3	+1
	55	90	−9	−6	−3
	100	91	−12	−11	−11
0.5	35	89	−5	−11	−4
	55	91	−10	−17	−7
	100	88	−8	−11	−3
0.9	35	85	−6	−4	−6
	55	84	−8	−4	+1
	100	84	−5	−3	+4

Above data relate to same experiment as *Table 1* of text.

TABLE III. CHANGE IN WALLACE PLASTICITY OF TYRE RUBBER TREATED WITH SEMICARBAZIDE

Semicarbazide solution (% wt)	Baling temperature (°C)	Initial P_0	Change in P_0 after storage (month) One	Two	Three	Four
0.8	35	32	+2	+2	+3	+2
	55	32	0	0	+1	+1
	100	33	+1	+2	+3	+1
1.0	35	32	+2	0	+1	0
	55	34	−1	−2	+1	0
	100	34	+1	+1	+2	−1
1.5	35	33	0	+1	+2	+1
	55	32	+2	+1	+3	+1
	100	34	−1	0	0	−1

Above data relate to same experiment as *Table 3* of text.

TABLE IV. CHANGE IN PRI OF TYRE RUBBER TREATED WITH SEMICARBAZIDE

Semicarbazide solution (% wt)	Baling temperature (°C)	Initial PRI	Change in PRI after storage (month) One	Two	Three	Four
0.8	35	79	0	+6	+4	+4
	55	91	−9	−9	−8	−10
	100	88	−7	−5	−13	−8
1.0	35	82	−2	+4	+2	−1
	55	85	−7	−3	−9	−9
	100	90	−6	−5	−10	−8
1.5	35	78	−14	+4	−4	+3
	55	75	−1	+8	+2	+7
	100	85	−4	−10	−13	−6

Above data relate to same experiment as *Table 3* of text.

TABLE V. CHANGE IN WALLACE PLASTICITY OF TYRE RUBBER TREATED WITH HYDROXYLAMINE NEUTRAL SULPHATE

Hydroxylamine neutral sulphate (% wt)	Baling temperature (°C)	Initial P_0	Change in P_0 after storage (month) One	Two	Three	Four
0	35	36	+2	+5	+4	0
	55	40	+1	+2	+2	−2
	100	37	+4	+5	+4	0
0.08	35	31	+3	+10	+3	+2
	55	35	−1	+5	+1	−3
	100	32	+2	+9	+4	+1
0.15	35	31	+2	+6	+3	+1
	55	36	−2	+1	−3	−4
	100	32	0	+5	+2	0
0.3	35	28	+4	+8	+5	+1
	55	33	−1	+4	−2	−4
	100	32	+1	+4	+2	+1
0.4	35	28	+2	+4	+3	+2
	55	32	−1	0	−1	−3
	100	30	+1	+3	+1	−1
0.5	35	28	+1	+4	+3	+1
	55	30	−1	+3	−1	−2
	100	28	+3	+4	+3	0

Above data relate to same experiment as *Table 5* of text.

TABLE VI. CHANGE IN PRI OF TYRE RUBBER TREATED WITH HYDROXYLAMINE NEUTRAL SULPHATE

Hyrdoxylamine neutral sulphate (% wt)	Baling temperature (°C)	Initial PRI	Change in PRI after storage (month) One	Two	Three	Four
0	35	89	−4	+5	−8	−4
	55	79	+1	+12	+5	+3
	100	78	+1	+7	+2	+1
0.08	35	95	−10	−17	−20	−11
	55	81	+4	−3	−6	+3
	100	88	−7	−13	−4	−4
0.15	35	85	0	−6	−7	−4
	55	80	+3	−3	−5	+1
	100	86	−1	−12	−11	−5
0.3	35	89	−4	−10	−12	−1
	55	85	−1	−9	−2	−4
	100	81	+5	−3	−4	−7
0.4	35	91	−3	−6	−15	−1
	55	78	+8	+4	+4	+6
	100	81	+3	−2	+3	+4
0.5	35	86	−1	+2	−8	−2
	55	83	+1	+1	−4	0
	100	86	−3	+2	−10	−5

Above data relate to same experiment as *Table 5* of text.

TABLE VII. CHANGE IN WALLACE PLASTICITY OF TYRE RUBBER TREATED WITH HYDROXYLAMINE NEUTRAL SULPHATE IN STORAGE

Hydroxylamine neutral sulphate (% wt)	Baling temperature (°C)	Initial P_0	Change in P_0 after storage (month) One	Two	Three	Four
0.3	35	29	+2	+1	+3	+5
	55	35	−5	−5	−3	−1
	100	34	−5	−3	−2	−1
0.4	35	29	+4	+3	+3	+8
	55	30	+3	+3	+6	+7
	100	30	+3	+3	+4	+7
0.5	35	31	−1	−1	0	+3
	55	32	−2	−2	+2	+2
	100	29	+2	+1	+4	+5

Above data relate to same experiment as *Table 6* of text.

TABLE VIII. CHANGE IN PRI OF TYRE RUBBER TREATED WITH HYDROXYLAMINE NEUTRAL SULPHATE ON STORAGE

Hydroxylamine neutral sulphate (% wt)	Baling temperature (°C)	Initial PRI	Change in PRI after storage (month) One	Two	Three	Four
0.3	35	82	−3	−1	0	−1
	55	83	−2	−7	−3	−3
	100	80	+1	0	−1	+1
0.4	35	81	−1	+1	+1	−1
	55	80	+2	−2	−5	−1
	100	81	+1	−1	0	−1
0.5	35	78	+1	+2	+3	+1
	55	84	−6	−7	−6	−5
	100	79	0	−1	−2	0

Above data relate to same experiment as *Table 6* of text.