EFFECT OF SUN DRYING ON THE PROPERTIES OF SHEET RUBBER

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Drying is one of the most critical steps in the processing of latex into dry forms of natural rubber. Sheet rubber, which predominates the Indian rubber market, is conventionally dried in smoke. However, owing to various constraints, many small growers resort to at least partial drying in the open sun. In the present study different modes of sun drying are compared with smoke drying. The effect of ultra-violet light on the raw rubber properties has also been studied. It is observed that Po, Mooney viscosity and gel content increase while PRI decreases as a result of sun drying. The results indicated that unless exposed to sun for prolonged periods, the raw rubber and the breakdown properties are not affected significantly. A slight over-exposure of sheet to direct sunlight will not adversely affect the initial vulcanizate properties of the rubber but its ageing properties are found to be inferior.

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Key words: Breakdown behaviour, Sheet rubber, Sun drying, Technological properties

INTRODUCTION

More than 70 per cent of the natural rubber latex produced in India is processed as sheet rubber. A major share of the production is by the small growers. The methods of processing adopted by the individual growers may differ and this could be reflected in the properties of the sheet rubber.

Drying is one of the most critical operations in the production of sheet rubber. Smoke drying is practised conventionally and the creosotic materials present in smoke impart a limited short-term protection against fungal attack. Further, smoke drying is quicker than open sun drying (Thomas, 1971). The consumption of firewood for drying of sheet rubber is reported to be 1 kg per kilogram of rubber (Edgar, 1958). However, the low availability of firewood has recently become a constraint. In order to reduce the cost of drying and also to improve the quality of crepe rubber, solar heating was proposed as an alternative method (Walpita et al., 1984). Experiments with smoke house fitted with solar panels showed that there could be a saving in firewood to the extent of 60 to 70 per cent by using solar-cum-smoke dryer (Nair *et al.*, 1988). But the capital expenditure on installation of solar panels is comparatively high.

It is a usual practice in smallholdings to resort to varying extents of direct sun drying of sheet rubber. Mostly sun drying is limited to the initial one or two days while in some cases it may be extended even to the complete drying period. There have been contradictory reports on the effect of direct exposure of natural rubber to sun. O' Connell (1966) reported that sunlight has a deleterious effect on all grades of natural rubber and even a few hours' exposure can seriously affect the properties of the rubber. It has also been reported that direct exposure of wet sheets to sunlight does not affect the dynamic properties of the rubber (Tan et al., 1977). Tillekeratne et al. (1995) reported that complete sun drying & sheet rubber does not adversely affect its physical or vulcanizate properties. The present study is carried out to assess the effect of sun drying on the raw rubber properties, mastication characteristics and processing properties of sheet rubber.

EXPERIMENTAL

Fresh latex required for the production of sheet rubber was collected from the Experimental Farm of the Rubber Research Institute of India. Sheet rubber was prepared under standard conditions (Kuriakose, and Thomas, 2000). Freshly machined sheets were dried under different conditions (Table 1). Treatments 1 and 2 were the conventional practices adopted for drying sheet rubber. Treatments 3 and 4

Table 1. Modes of drying of sheet rubber

Treatment	Mode of drying
1	Smoke dried for 5 days
2	Sun dried for 40 h*
3	Over-dried in smoke for 10 days
4	Over-dried in sun for 60 h
5	Wet sheets exposed to 3h UV radiation** and then smoke dried for 5 days
6	Wet sheets exposed to 3h UV radiation and then sun dried for 40 h
7	Smoke dried for 2 days and then dried in the sun for 20 h
8	Sun dried for 20 h and then smoke dried for 2 days
9	Smoke dried sheets exposed to 3h UV radiation

^{*} From 10 am to 3 pm for 8 days

were included to evaluate the effect of excessive drying. Wet sheets were exposed to a monochromatic source of UV light, and subsequently exposed to smoke / sun drying, to assess the relative effect of UV light on degradation (Treatments 5 & 6). Treatments 7 and 8 were to compare the extent of degradation occurring in sheets, when combined modes of drying were adopted. Smoke dried sheets were exposed to UV radiation to assess the isolated effect of radiation (Treatment 9). Five replications were maintained and the parameters evaluated included volatile matter (ASTM D 4571 -98), Wallace plasticity and plasticity retention index (ASTM D 3194 – 99), Mooney viscosity (determined using a Mooney viscometer Model Shimadzu SMU 202 as per ASTM D 1646 – 99) and gel content (ASTM D 2765 – 84).

Mastication characteristics were assessed in terms of breakdown index and associated parameters using a Haake Rheocord 90 (Bartha *et al.*, 1983) at 40 RPM for 10 min with an initial temperature of 40°C. Figure 1 gives a typical Haake Rheogram from which various parameters were measured. The measurement made included Torque at the $2^{\rm nd}$ min (TQ_2), $5^{\rm th}$ min (TQ_5), and $10^{\rm th}$ min (TQ_{10}), the rate of breakdown measured from the drop in torque TQ_2 - TQ_5 with time [BI]₁ and [TQ_5 - TQ_{10}] with time [BI]₂, totalized torque at the $5^{\rm th}$ min [TTQ]₁ and at the $10^{\rm th}$ min [TTQ]₂ and the difference in temperature at the $10^{\rm th}$ min (Δt).

Molecular weight determination was carried out by a Waters 510 Gel Permeation Chromatograph (GPC) having a 410 Diffraction Refractometer. HPLC grade toluene was used as solvent with a flow rate set at 1 ml/min. The GPC columns were caliberated with polystyrene standards.

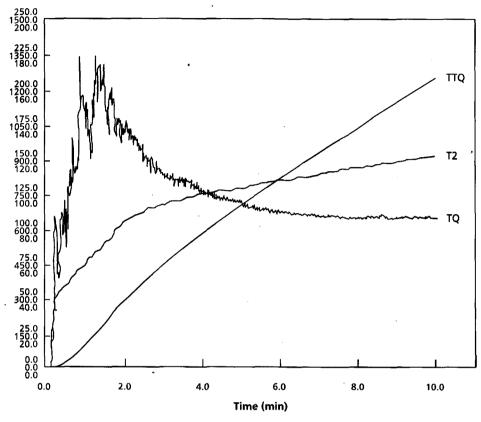
To assess whether sun drying has any effect on the rate of vulcanization and on the properties of vulcanizate, a gum rubber compound prepared as per ACS-1 formulation and a filled compound of selected samples containing HAF black, were prepared using a laboratory model two-roll mixing mill. The formulations of the mixes are given in Table 2. The compounding in-

Table 2. Formulation of compounds

Ingredient	ACS-1	HAF filled
Natural rubber	100	100
Zinc oxide	6	5
Stearic acid	0.5	2 يو
HAF black (N300)	_	40
Naphthenic oil	_	4
Antioxidant HSL*	_	1
MBT	0.5	_
CBS	_	0.6
Sulphur	3.5	2.5

^{* 2,2,4 -}trimethyl 1,2 -dihydroquinoline

^{** 2} UV lamps each of 20 wats kept at a distance of 25 cm from the sample



TQ: 0-250 (Nm) TTQ: 0-1500 (Nm-min) T2: 0-200 (°C)

Fig. 1. Breakdown properties of sheet rubber

gredients used for the study were of commercial grade. Cure characteristics of the compounds were determined using a Monsanto Rheometer R100 at 150°C. Test samples were moulded using an electrically heated hydraulic press to their respective optimum cure time. Physical properties of the vulcanizates namely, stress-strain properties (ASTM D 412-80 Zwick UTM, model 1474), tear strength (ASTM D 624-00 Zwick UTM, model 1474), hardness (ASTM D 2240-00, Shore A), resilience (ASTM D 1054-00, Dunlop tripsometer), heat build-up (ASTM D 623-99, Goodrich flexometer), compression set (ASTM D 395-98, Method B), abrasion resistance (DIN 53516) and ageing resistance (ASTM D 573-99) were tested.

RESULTS AND DISCUSSION

Mean values of the statistically analysed data of the raw rubber properties are given in Table 3. There is no significant difference among the treatments in the case of volatile matter and the values are within the limit of 0.8% fixed for ISNR grades (IS: 4588;1986). In the case of Wallace plasticity, there is significant difference among the different modes of drying. The mean vaues are significantly high for sheets exposed to direct sun, except in the case of those dried for prolonged periods. The high values indicate that sunlight initiated both crosslinking and attachment of the scissioned chains to the original rubber chains to form longer chains. These occur

Table 3. Raw rubber properties

reatment	Volatile matter (%)	Wallace plasticity (P ₀)	Mooney viscosity	Gel content (%)	Plasticity retention index (PRI)
1	0.56	46.60	76.80	9.75	82.00
2	0.45	50.60	85.46	21.42	69.60
3	0.58	44.80	7 8.16	10.64	80.80
4	0.49	42.20	83.78	19.50	53.60
5	0.64	47.20	76.58	11.30	80.80
6	0.48	48.80	86.74	18.60	68.40
7	0.56	47.67	85.67	19.62	76.35
8	0.65	48.67	85.1 <i>7</i>	17.21	77.67
9	0.52	46.00	76.74	9.75	81.80
VR CD (P≤0.05)	0.82 ^{NS}	14.93**	7.58**	8.84**	36.86**
Group I		1.81	4.49	4.56	5.03
Group II		2.34	5.79	5.88	6.50
Group I and I	I	2.09	5.1 <i>7</i>	5.26	5.80
		2.09 5, 6 and 9; Group II = 1			5

^{**} Significant at P≤ 0.01; NS = Not significant

simultaneously but on prolonged exposure, chain scission is predominant (Tan et al., 1977). Po is a qualitative measure of the average molecular weight of the sample. Tillekeratne et al. (1995) reported that an initial sun drying of wet sheets does not cause much degradation. However, it is observed that the effect of sun drying, either during the initial period of drying (Treatment 8), or towards the final period (Treatment 7), does not contribute much, as far as Wallace plasticity is concerned and the values are comparable with other sun dried samples.

Significant differences were also noted in the case of Mooney viscosity and gel content. The mean values are significantly high for all the different modes of sun dried sheets and all were on par. However, the same trend is not obvious as in the case of Wallace Plasticity when the sheets are exposed to sun for longer periods. Reduction in Mooney viscosity and gel content were not observed in the above case. Higher gel content indicates crosslinked fractions and highly branched chains, which could leads to an increase in Mooney viscosity also. The values for smoke dried sheets were low and

comparable.

Plasticity retention index is a measure of the resistance of rubber to oxidation. The mean values were high for all treatments where smoke drying was adopted with a slight reduction when the duration of smoking was reduced. The lower chances for oxidation in the smoke filled environment may have contributed for this. Significantly low PRI values were observed for sheets exposed only to sun and the lowest value was recorded for the sheets dried in sun for longer periods. The decrease in PRI values with exposure to sunlight has already been reported (Bateman et al., 1966). Over exposure to sunlight increases the number of reactive groups such as hydroperoxides, which could cause the rubber chains to degrade further on heating in air at 140°C. This explains the low PRI values for sun dried sheets especially in the context of the higher initial Po values.

The breakdown properties of the rubber obtained under different treatments are presented in Table 4. The torque at the 5th min were higher and comparable for the sheets dried in smoke alone and with com-

Table 4. Breakdown parameters

				-			
Treatment	TQ ₅ (Nm)	TQ ₁₀ (Nm)	[BI] ₁	[BI] ₂	[TTQ] ₁ (Nm/min)	[TTQ] ₂ (Nm/min)	Δt°C
1	122.60	110.20	. 20.26	2.48	705.40	1264	82.0
2	118.60	106.80	1 9.4 6	2.36	706 .60	1261	80.0
3	122.20	110 .60	19.34	2.32	694.00	1260	82.8
4	112.40	100.60	22.72	2.36	684.80	1213	75.6
5	122.40	110.00	19.38	2.48	704.00	1269	83.0
6	116.60	106.60	21.38	2.00	699.20	1253	79.6
7	121.67	108.67	17.70	2.60	704.67	1264	81.0
8	124.67	109.67	19.87	2.00	718.33	1305	83.3
9	119.00	110.10	20.80	2.40	694.40	1248	82.8
VR	3.47**	5.21**	NS	NS	NS	NS	5.95**
Group I	5.58	4.51					3.42
Group II	7.20	5.82					4.41
Group I and	d II 6.43	5.20					3.94

Group I = Treatments 1, 2, 3,4, 5, 6 and 9; Group II = Treatments 7 and 8 ** Significant at P≤0.01; NS = Not significant

bination of smoke and sun. Lower values were obtained for the sun dried sheet and the lowest was recorded for the sheet overexposed to sun. As mastication proceeds, torque reaches a maximum value and then descends. It almost stabilises at the end of the 10th min and this torque is taken as the stabilised torque. The stabilised torque is a measure of the viscosity of the masticated rubber. A higher stabilised torque for the sample dried in smoke is an indication of the higher molecular weight of the same. The stabilised torque at the 10th min was also higher for the sheets where at least partial smoke drying was resorted to. Samples dried in sun recorded lower values, the lowest being for that dried for longer periods (Treatment 4).

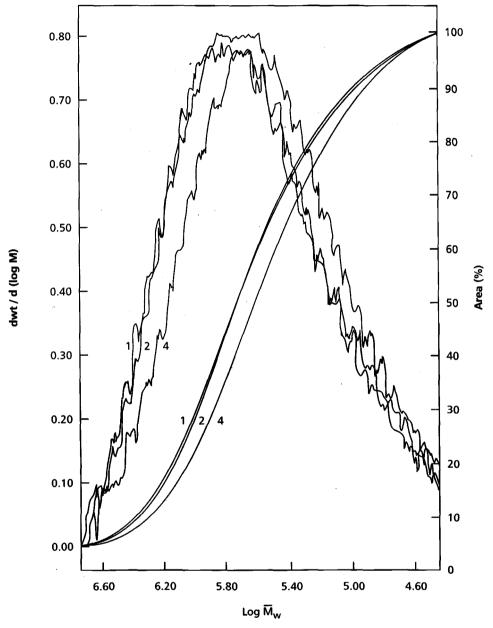
Significant differences among treatments were not observed for the rate of breakdown at the 5th and 10th min. This indicates that there was not much difference in the mastication behaviour for the sheets obtained under the different treatments. The totalised torque, TTQ, is a measure of the work done during mastication. The TTQ measured at the 5th min was not statistically significant among the different treatments but the value at the 10th min was only significant at 5% except for the excess sun dried

sheet (Treatment 4), which showed the lowest value.

The heat generated during mastication is an indication of the state of degradation of the rubber. A highly degraded rubber will generally yield less heat during mastication. (Thomas *et al.*, 1998). The difference in temperature observed during mastication at the 10th min was almost the same for all the treatments except for the excess sun dried sheet (Treatment 4), which recorded the lowest value. This indicates that over exposure of sheet to sun causes molecular degradation of rubber.

The molecular weight distribution curves recorded by GPC for the conventionally dried, sun dried and for the excess sun dried sheets are given in Figure 2. The molecular weight and molecular weight distribution of rubber were not affected up to 40 h of exposure to sunlight (normal drying period of rubber sheets). However, over exposure to sun brings about molecular scission, and as a consequence the low molecular weight fraction increases and the high molecular weight fraction decreases. The number average molecular weight $(\overline{M}n)$, weight average molecular weight $(\overline{M}w)$ and polydispersity of samples from Treatments 1, 2 and 4 are given in Table 5. There was no

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1. Smoke dried 2. Sun dried 4. Excess sun dried

Fig. 2. Molecular weight distribution curves of smoke dried, sun dried and excess sun dried sheets

significant difference in these parameters for treatments 1 & 2 but for the excess sun dried sheet the values were lower.

Cure characteristics and technological

properties of ACS-1 compounds are shown in Table 6. Optimum cure time for the sun dried sheets was slightly higher than those dried by the conventional method and the

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Table 5. Molecular characteristics

Treatment	Mn (Daltons)	Mw (Daltons)	Polydispersity
Smoke dried for 5 days T ₁	2.31 x 10 ⁵	7.31 x 10 ⁵	3.17
Sun dried for 40 h T ₂	2.28×10^{5}	7.62 x 10 ⁵	3.34
Sun dried for 60 h T ₄	2.01×10^{5}	6.21×10^5	3.09

Table 6. Cure characteristics and vulcanizate properties of ACS-1 compounds

Parameter	Treatment number*				
	1	2	4	7	- 8
Optimum cure time (t _{so})					
at 150°C, min.	10.00	11.00	11.50	11.00	11.00
Mooney viscosity,					
ML(1+4) 100°C.	42.40	42.90	41.70	42.50	42.00
Cure rate index $[100/(t_{90}-ts_2)]$	13.30	11.10	11.10	11.00	11.10
Hardness, Shore A	30.00	32.00	29.00	30.00	31.00
Heat build-up, ∆t, °C	15.00	15.00	16.00	16.00	14.00
Compression set, %	25.80	24.10	24.60	26.30	25.90
Resilience, %	63.80	63.80	63.10	63.10	63.10
Abrasion loss, mm ³	363.00	346.00	376.00	382.00	356.00
Tensile strength, N/mm ²	14.30	15.60	14.20	15.50	15.10
300% modulus, N/mm ²	1.32	1.56	1.56	1.47	1.54
Elongation at break, %	<i>777</i> .00	710.00	726.00	752.00	750.00
Tear strength, N/mm	26.00	25.50	27.20	26.50	28.70
Retention after ageing at					
70°C for 14 days					
300% modulus, %	140.00	140.00	148.00	141.00	140.00
Tensile strength, %	70.00	69.00	61.00	68.00	69.00
Elongation at break, %	92.00	91.00	82.00	91.00	92.00

^{*} See Table 1 for details

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excess sun dried sheet showed the maximum cure time. The conventionally dried sheet showed the maximum cure rate index while the other treatments recorded lower but similar indices. Technological properties were almost comparable in all cases. Retention in tensile properties after ageing was adversely affected only in the case of the sheet over-exposed to sun (Treatment 4).

The cure characteristics and technological properties of the HAF black filled compounds are presented in Table 7. Improvement in cure time and cure rate index in the case of the ACS-1 mix for the conventionally dried sheet was not observed in the black filled compounds. Technological properties of samples compounded using sun dried sheet rubber were identical to those of the control showing that a slight

over-exposure of sheets to sunlight does not adversely affect the vulcanizate properties of the rubber. However, the ageing properties of vulcanizates prepared from such rubber were observed to be inferior.

Though plasticity, Mooney viscosity and gel content of the fully or partially sun dried rubber were high, significant differences were not observed in their breakdown behaviour. Comparatively lower PRI was observed for sun dried samples and a slight over exposure to sun deteriorated the quality and appearance of the sheet rubber However, even this much exposure of sheet to direct sun did not adversely affect the initial vulcanizate properties of the rubber but the ageing properties of the vulcanizates from the excess sun dried sheets were inferior. Drying in sunlight during the initial

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Table 7. Cure characteristics and vulcanizate properties of HAF-filled compounds

Parameter	Treatment number*					
	1	2	4	7	8	
Optimum cure time (t _{so})	•					
at 150°C, min.	9.0	9.0	8.5	9.0	9.5	
Mooney viscosity,						
ML (1+4) 100°C.	50.7	50.6	44.1	52.6	46.9	
Cure rate index $[100/(t_{\infty}-ts_{2})]$	16.4	16.7	15.2	15.4	15.4	
Hardness, Shore A	59.0	59.0	58.0	58.0	58.0	
Heat build-up, Δt, °C	22.0	22.0	24.0	21.0	20.0	
Compression set, %	35.3	34.2	35.1	35.8	35.0	
Resilience, %	58.4	58.4	57.7	58.4	59.0	
Abrasion loss, mm ³	102.0	103.0	104.0	100.0	100.0	
Tensile strength, N/mm ²	28.8	28.5	27.0	28.3	28.9	
300% modulus, N/mm ²	12.8	12.9	11.9	11.5	12.8	
Elongation at break, %	546.0	550.0	602.0	559.0	555.0	
Tear strength, N/mm	110.0	107.0	108.0	108.0	110.0	
Retention after ageing at						
70°C for 14 days						
300% modulus, %	135.0	136.0	140.0	137.0	136.0	
Tensile strength, %	85.0	85.0	80.0	84.0	85.0	
Elongation at break, %	78.0	79.0	74.0	78.0	78.0	

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period with subsequent drying in smoke can render good quality to sheet rubber.

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^{*} See Table 1 for details