VARIATION IN PLANTATION SHEET RUBBER.

BY

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Examination of Exhibition Samples.

An examination of the vulcanisation properties of samples of smoked sheet exhibited at the Malayan Agri-Horticultural Exhibition in August 1931 led to the collection of data summarised in Table I.

Procedure. Samples of not less than 5 lbs. were taken at random from each case of 224 lbs. and vulcanised under the standard conditions now adopted by the Institute and described in the appendix. In the Table are also included the results of smoked sheet prepared in August and collected from two estates which did not exhibit. Columns 1 and 2 give serial numbers referring to the various estates and column 3 gives the thickness of the smoked sheet. Column 4(a) states the time of cure in minutes giving the maximum tensile strength at break while column 4(b) gives the maximum tensile strength at break in kilogrammes per square millimetre. A true comparison, however, of the tensile properties of the sample is obtained by reference to column 5 which states the tensile strengths obtained at an elongation of 650 per cent when the samples are vulcanised under uniform conditions, viz: at a cure of 80 minutes. This value is termed the " modulus."

Results. From the analysis of the results we find by comparison of the coefficients of variation (C. of V.) given at the foot of each column:—

(1) The variation in thickness is decidedly high.

(2) The variation in rate of cure is 14 and the average rate of cure lies between 80 and 90 minutes.

(3) There is considerable uniformity in the values for the maximum tensile strength at break.

(4) The variation in modulus is high.

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Smoked Sheet.

Samples Exhibited at M.A.H.A. Exhibition 1931.

TA	IBI	ĻΕ	Ι.

1	2	3	4 (a)	4 (b)	5
R.R.I. Ref. No. H.	Estate No.	Thickness of smoked sheet mm.	Cure giving maximum tensile strength at break Mins.	Maximum tensile strength at break Kgs/mm?	Tensile strength at 650% elon- gation at 80 minutes cure Kgs/mm ²
848	1	1.89	120	1.55	_
849	2	2.34	. 90	1.46	0.62
850	3	2.81	80	1.49	1.29
851	4	3.84	80	1.51	0.88
852	5	3.23	80	1.56	1.08
853	6	3.96	80	1.36	1.04
854	7	3.56	90	1.66	0.69
855	8	3.38	90	1.51	0.52
856	10	2.64	90 -	1.49	0.77
857	12	3.44	90	1.58	0.60
858	13	4.25	80	1.47	0.99
859	14	4.33	70	1.52	0.94
860	15	3.93	90	1.32	0.62
861	16	3.56	80	1.49	0.99
862	17	2.36	80	1.50	1.00
863	18	3.88	120	1.39	0.48
864	19	3.18	90	1.47	0.77
865	20	2.44	80	1.45	0.64
866	21	4.60	70	1.40	0.76

1	2	3	-1 (a)	1 (b)	5
R.R I. Ref. No. II.	Estale No,	Thickness of smoked sheet mm.	Care giving maximum tensile strength at break Mins,	Maximum tensile strength at break Kg5/mm²	Tensile strength at 650% elonga- tion at 80 minutes cure Kgs/mm ²
867	23	3.22	90	1,50	; 0.77
868	24	2.82	90	1.34	0.55
869	25	3.96	80	1.44	1.10
870	26	3.35	70	1.42	0.69
871	27	3.79	70	1.21	0.79
872	29	4.12	80	1.46	0.88
873	30	2.69	90	1.41	0.72
874	31	3.99	50	1.46	0.62
875	32	3.00	60	1.37	2
876	33	2.38	80	1.53	0.91
877	35	2.42	80	1.12	0.56
878	36	3.20	80	1.45	0.71
879	38	3.71	80	1.53	0.87
880	39	2.82	80	1.50	0.84
881	41	3,79	80	1.45	0.67
882	42	3.73	80	1.47	0.82
883	43	2.86	90	1.31	0.58
884	44	2.93	80	1.28	0.59
885	45	3.41	80	1.22	0.43
886	46	2,95	80	1.44	0.75
887	47	3.19	7 0	1.30	0.65
888	48	3.51	70	1.37	0.83

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1	2	3	4 (a)	4 (b)	5
R.R.I. Ref. No. H.	Estate No.	Thickness of smoked sheet m.m.	Cure giving maximum tensile strength at break Mins.	Maximum tensile strength at break Kgs/mm ²	Tensile strength at 650% elonga- tion of 80 minutes cure Kgs/mm ²
889	49	2.62	80	1.34	0.62
890	50	3.50	80	1.46	0.79
891	65	3.06	l 1 80	1.36	0.74
892	66	3.43	105	1.27	0.46
1031	67	 	90	1.32	0.82
	68	2.81	90	1.48	0.78
	Mean S.D. C. of V.	= 3.28 = 0.604 = 18	Mean = 83 S.D. = 11.14 C. of V= 14	Mean = 1.43 S. D. = 0.104 C.of V.=7	Mean = 0.76 SD. = 0.185 C.of V. = 24
Ş	5.D. = 1	$\sqrt{\frac{\sum d^2}{n}}$		$V_{\cdot} = \frac{100}{100}$	\times S.D. Mean

Discussion. The most illuminating of these results is No. 4 which shows that the samples vary in their response to vulcanisation and exhibit a significant lack of uniformity.

Eaton and Bishop⁽¹⁾ made a similar study of samples of smoked sheet collected from the Malayan Agri-Horticultural Exhibition, 1927. Although the technique adopted at that time for mixing and vulcanisation differed from our present practice, a comparison of the results on a common basis shows the same order of variation.

As an indication of the degree of variation which may be obtained in uniform rubber due to methods of vulcanisation adopted, we quote in Table II the results obtained for different portions of a sheet of sole crepe selected at random from a case of 100 lbs., prepared by one estate, whose crepe invariably fetches top market price. In sampling, the sheet of crepe was rolled tightly into a cylinder and eight samples of 200 grammes each taken by cutting sections perpendicular to the axis. The end portions were neglected. From each of the eight portions, a mix was prepared and the eight mixes vulcanised separately at 100 minutes. Each mix gave four portions for vulcanisation from each of which two rings were taken for testing. The Table quotes the loads, in kilo-

TABLE II.

Sole Crepe.

Loads in Kgs/mm² at 650% elongation at 100 minutes cure.

Mix No.		Mean.			
1	0.96	0.99	0.97	0.96	0.07
	0.96	0.99	0.97	0.99	0.97
2	0.92	0.92	1.01	0.91	0.03
	0.92	0.91	0.95	0.94	0.93
3	0.98	0.95	1.01	0.97	0.02
	0.94	0.93	1.05	1.01	0.96
4	0.86	0.84	0.90	0.88	0.95
	0.87	0.84	0.87	0.85	0.85
5	0.94	0.93	0.97	0.96	0.04
	0.93	0.92	0.90	0.95	0.94
6	1.02	0.98	1.02	1.04	0.07
	0.99	0.95	0.97	0.96	0.97
· · · · · · · · · · · · · · · · · · ·		Mean	= 0.95		
		S.D.	= 0.05		
		C. of V	. = 5		8

grammes per square millimetre, obtained at an elongation of 650 per cent. The analysis of the figures given at the foot of the Table shows that the variation in the values obtained is very low, the coefficient (C of V.) being 5 as compared with a coefficient of 24 for the M.A.H.A. Exhibition samples.

Examination of Rubber from Selected Estates.

The nature of the results recorded above led us to investigate the variation in rubber prepared on the same estate at different times. From eight of the estates on the M.A.H.A. Exhibition list further cases of smoked sheet of 224 lbs. each were collected from crop manufactured early in November. The methods of sampling the cases were similar to those adopted in August and the two sets of results are presented for comparison in Table III. It will be seen that only in one instance-that of estate No. 4-has the rate of cure remained unaltered, while for estates Nos. 26 and 27, rubber which had in August a rate of cure higher than the average is now in November lower than the average, the difference being 20 minutes. In this connection it should be noted that in August the sheets were very much thicker and less uniform in thickness than those prepared in November. In the other samples examined, the rate of cure has altered to the extent of 10 minutes. The values for maximum tensile strength at break have not substantially altered. The extent of the changes may be appreciated by reference to the modulus figures in column No. 4.

TABLE III

1	2	2	3	3 (a)		3 (b)		4.		
Estate No.	Thiel o smo sho	cness f oked eet m.	Cure max te stren bre M	Cure giving maximum tensile strength at break Mins.		Maximum tensile strength at break Kgs/mm ³		imum Tensile isile strength at 6503 elongation at 84 reak minutes cure /mm ² Kgs/mm ²		nsile h at 650% tion at 80 tes cure Jmm ²
	Aug.	Nov.	Aug.	Nov.	Aug.	Nov.	Aug.	Nov.		
8	3.38	3.03	90	80	1.51	1.37	0.52	0.61		
4	3.84	3.59	80	80	. 1.51	1.25	0.88	0.72		
67			90	100	1.32	1.32	0.82	0.49		
26	3.35	3.03	70	90	1.42	1.28	0.69	0.58		
27	3.79	3.05	70	90	1.21	1.43	0.79	0.42		
68	2.81	2.81	90	80	1.48	1.33	0.78	0.83		
14	4.33	3.25	70	80	1.52	1.38	0.94	0.87		
12	3.44	2.98	90	80	1.58	1.45	0.60	0.67		

COMPARISON OF AUGUST AND NOVEMBER SHEET.

To ascertain to what extent the changes observed might be due to methods of sampling, cases were collected at random from estate No. 68, and an examination made of portions drawn from different parts of each case. Three cases of 224 lbs. each were taken corresponding to different crop dates. The results summarised in Table IV indicate that there is a very great lack of uniformity in the rubber in cases Nos. 1 and 2 collected in August, while that in case No. 3 collected in November may be said to be almost uniform in quality since the variation in load is within the limits of experimental error (vide appendix).

TABLE IV.

VARIATION IN A CASE OF 224 LBS.

Sheet vulcanised under standard conditions at 90 minutes. Load per square mm. determined at 650% elongation. Case No. 1 from sheet prepared during 1st week in August. Case No. 2 from sheet prepared during 4th week in August. Case No. 3 from sheet prepared during 1st week in November.

Case 1. Load Kgs/mm²			Case 2. Load Kgs/mm²			Case 3. Load Kgs/mm²			
1.48			1.31			1.09			
1.	53		1.:	21		1	1.07		
1.	1.44 1.16					1.09			
0.	0.71			0.96			.03		
0.	70		1.07		1.07				
					:	1	.09		
Mean	_	1.17	Mean = 1.14		Mean		1.07		
S.D.	=	0.40	S.D. $= 0.12$			S.D.	=	0.02	
C. of V.	=	34	C. of V.	C. of V. = 11			=	2	

Since we were aware that factory practice on estate No. 68 was in process of alteration during the period August/November, we decided to extend our investigation to the examination of cases of smoked sheet prepared in November from three estates

among the M.A.H.A. exhibitors on which we know that good supervision governs manufacture and attention is paid to the processes of bulking the latex, coagulation, machining and drying with a view to securing a uniform product. As before, samples were drawn from different portions of a case and the loads determined at 650 per cent. elongation when cured for 80 minutes. The results are presented in Table V. The coefficients show that the variation in the case collected from estate No. 12 is almost of the same order as that obtained for the uniform sample of sole crepe referred to in Table II. On estates No. 14 and 5, however, even when allowance is made for experimental error, the variation is much higher, and in the case of estate No. 5 is almost double that of estate No. 12. Results have been selected to illustrate in Figures 1, 2 and 3, the differences obtained for samples drawn from two different portions of the same case. The curves show the representative stress/strain relationship for each sample.

TABLE V.

VARIATION IN A CASE OF 224 LBS.

Est:	ate No.	. 12.	Est	ate No	. 14.	Estate No. 5.			
0.66	0.78	0.64	0.72	0.66	0.82	0.52	0.54	0.61	
0.65	0.70	0.71	0.71	0.71	0.91	0.52	0.59	0.68	
0.70	0.70	0.67	0.71	0.70	0.88	0.53	0.50	0.66	
0.63	0.72	0.75	0.71	0.69	0.77	0.53	0.56	0.73	
0.63	0.68	0.76	0.70	0.66	0.85	0.59	0.58	0.74	
0.72	0.66	0.80	0.65	0.69	0.89	0.59	0.58	0.71	
			. [i			<u> </u>	
Mean	,==	0.70	Mean	=	0.75	Mean	_	0.60	
S.D.	=	0.05	S.D.	=	0.08	S.D.	=	0.08	
C. of	V. ==	7	C. of	v. =	11	C. of	v. =	13	
			l 						

Loads at 650% Elongation at 80 minutes cure-Kgs/mm².

Comparisons Based on Technical Mixes.

In accordance with the practice of most modern investigators, and as advocated by Martin in his remarks on the evaluation of raw rubber⁷, it was decided to ascertain the extent of the variations when the rubber was vulcanized in a technical mixing containing an accelerator. For this purpose, as an initial step it was decided to use the "Captax" mix which is typical of American compounding practice. Its composition on a weight basis is as follows:—

100	parts	Rubber	
6	*3	Zinc oxide	
3.5	"	Sulphur	
0,5	,,	"Captax" (mercaptobenzothiazole).	

The August and November smoked sheet from four estates were compared in this mix, by vulcanising for 40 minutes at a temperature of 126°C and determining the load in kilogrammes per square mm. at an elongation of 650 per cent. Table VI gives the results obtained for the same samples of sheet according to our standard procedure and using a rubber and sulphur mix, while for comparison are given the results obtained with the corresponding "Captax" mixes.

Load at e elongation 80 minut Kgs/	ULPHUR MIX 550 per cent n cured for e9 at 149°C mm²	"CAPTAX" MIX Loads at 650 per cent elongation cured for 40 minutes at 126 C Kgs/mm ²		
Angust	November	August	November	
0.69	0.58	0 61	0.83	
0.79	0.42	0.95	0.87	
0.60	0.67	0.43	1.00	
0.94	0.87	0.88	1.33	
	Load at elongation 80 minut Kgs/ August 0.69 0.79 0.60 0.94	Load at 650 per cent elongation cured for 80 minules at 149°C Kgs/mm²AugustNovember0.690.580.790.420.600.670.940.87	Load at 650 per cent elongation cured for 80 minutes at 149°C Kgs/mm²Loads at elongati 40 minu KgAugustNovemberAugust0.690.580.610.790.420.950.600.670.430.940.870.88	

TABLE VI

The differences obtained under the two methods of procedure are more readily appreciated by reference to Figures 4 to 7 (a) and (b) showing the stress/strain relationship for each vulcanised sample. Confirming the results of discussions which we have had with representatives of various large manufacturing concerns, it is evident that it is a difficult matter to correlate the results obtained with a simple rubber-sulphur mix with those obtained with a technical mix containing "Captax." In the case of estate No. 26-vide Figures 4 (a) and (b)-the relationship between August and November rubber as shown in a rubber-sulphur mix is completely reversed in a "Captax" mix. The difference in moduli of rubber from No. 27 for a rubber-sulphur mix is greater than is the case with No. 26-viz: 0.37 as compared with 0.11-and, although the position is not reversed in a "Captax" mix, the curves for August and November rubber are brought very much closer together--vide Figures 5 (a) and (b). In the remaining two estate samples the difference in modulus with a rubber-sulphur mix is much smaller than in the preceding two cases, but the curves for a "Captax" mix are much wider apart. The variations obtained indicate the desirability of expressing the results of vulcanisation tests in terms of a standard technical mix as well as in terms of the usual rubber-sulphur mix. The results are reminiscent of those obtained in the vulcanisations carried out on rubber containing variable proportions of different sugars².

As will be seen, the foregoing results are similar to those obtained by Dinsmore³ an extract from whose article is given below:

"With the object of providing data on variability, rate of "cure was adopted as a criterion and it was demonstrated "that the variation between bales of the same lot of rubber "was as great as the variation between different lots. It was, "shewn that there was also considerable variation throughout "the rubber in a single bale.

"In further tests carried out to determine the efficacy of "blending, tests were made from each of a number of batches "of rubber taken from the breakdown mills. In all, 48 sam-"ples representing 48 batches were taken over a 7 day period "and were studied in a pure gum captax friction, activated "with zinc oxide, with results as follows:—

			Tensile			700%	Modulus
			Kg./cm².			k	Kg./cm².
" 24	tests	between	160-180	б	tests	between	140-160
~ 15	tests	between	140-160	7	tests	between	120-140
" 9	tests	between	120-140	18	tests	between	100-120

"This shews a rather wide variation even after blending.

"Another check made to see the effect of using a high "grade tread rubber which had been tested in the friction "formula above, shewed these results on 20 samples.

FRICTION TEST-20 SAMPLES.

Tests.			700% Modulus
			Kg./cm².
2, between			 160180
1, between	•••	•••	 140160
10, between	•••		 120—140
7, between	•••	•••	 100—120

TREAD TEST-20 SAMPLES.

Tests.			500% Mod	
	<u>:</u>		Kg./cm².	
3, between		•••		170-190
9, between	•••			150-170
8, between		•••		130-150

"Point for point there was a general correspondence be-"tween the high and low tests for the two stocks although "there were some reversals. Here again the fluctuation is "considerable. The above data are typical of a large mass "which, unfortunately, time did not permit of condensing to "suitable form for this hastily prepared review.

"It is now pertinent to ask whether these results are re-"flected in any practical performance tests. Two rubbers "were selected by the friction formula, giving moduli of 120 "and 57 respectively. These were compounded in a solid tire "stock where the differences were apparently entirely elimi-"nated. However, on a blowout test, the *high* grade rubber "gave about 45 per cent. more mileage than the *low* grade. "Two other rubbers were selected having modulus figures in "the friction stock of 147 and 37. Here the solid tire stock "shewed about 8 per cent. difference in modulus, but there "was 50 per cent. difference in the mileage to blow out.

"In a high grade balloon tread stock, rubbers which were "selected because they gave moduli of 104 and 53 in the fric-"tion test, gave 88 and 84 in the tread stock with abrasion loss "figures of 10.9 and 11.5 respectively. The road wear resist-"ance was in the ratio of 112 to 100.

"Another test was made on a pneumatic truck tire carcass "for blowout. The rubbers tested 105 and 70 modulus. On "a slow speed there was no difference beyond the experi-"mental error. The high speed test shewed a difference of "60 per cent. in favour of the high-test rubber."

Discussion.

While it is recognised that the adoption of rate of cure and tensile strength of the vulcanised rubber may not be entirely indicative of the true characteristics of any particular type of rubber, it is felt that rate of cure and tensile strength of the product provide a very reasonable criterion for determining the uniformity of any particular product, and there has been sufficient work done to shew that the other characteristics of rubber, such as those associated with ageing and plasticity, are subject to the same degree of variability. Investigations on plasticity have been carried out in the Dutch East Indies and by the Rubber Research Scheme, Cevlon. (5 and 6).

From these results, it is obvious that in spite of the knowledge we possess and the work which has been done on the preparation of rubber of uniform quality, there is still a very marked variability in the product of even first class European estates. The divergence of the results recorded for plasticity may possibly be accounted for by the difference in age of the samples tested by the two investigators, but it is also open to assumption that the variations originated in the preparation of the samples.

It is thus evident that one must eliminate variables due to methods of preparation before attempting to compare any two kinds of rubber. The extent of the influence exerted by variations in estate factory practice on the rate of cure and tensile strength of the raw product were sufficiently demonstrated as early as 1914 by Eaton, Grantham, and Day⁴. Further investigations carried out along these lines show conclusively that it is possible only by precise control of all the operations involved to produce a raw rubber possessing uniform characteristics. The calibration of variability is a first essential in the examination of rubber from different clones of budgrafted stock and in the comparison of smoked sheet with air-dried sheet.

It is of great significance that, at the present time, when the price of plantation raw rubber is at the lowest level reached since the beginning of the industry, the world consumption of reclaim is yet as high as 36 per cent. of the total.

In other words, although the 'raw product can compete in price with reclaimed rubber the latter is still very widely used, and there are good grounds for stating that the explanation of this striking fact is that manufacturers find in reclaim desirable properties which are not possessed by plantation raw rubber.

It is outside the scope of this paper to discuss the possibilities of preparing a plantation rubber possessing all the desirable characteristics of reclaim, but it is justifiable, in view of the marked variation in first grade sheet disclosed by the above results, to emphasise the need for standardising our methods of preparation whereby the uniformity of our product may be more closely related to manufacturing requirements.

From the results published by previous investigators and the results recorded elsewhere in this Journal², it has been shown that variability in rubber is caused by the presence of serum substances derived from the parent latex. Therefore, if the product of plantation factories is to be uniform in quality, the proportion of serum constituents present in the rubber must be reduced to a minimum or to a constant proportion. Modern methods of estate factory practice provide means for accomplishing this.

Summary.

An examination of the samples of sheet rubber collected at a recent Exhibition discloses a high degree of variation in the vulcanisation properties of the rubber.

Similar variations in the normal produce of first class estates are recorded. Other properties of rubber such as plasticity, may be expected to show similar variability.

Figures are given to indicate the extent of the irregularities and how they would be reflected in technical mixes.

The bearing of this on the industrial uses of raw rubber is discussed.

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APPENDIX.

The following sample of the form of the certificate which is issued by the Institute on samples sent for vulcanisation tests gives a summary of the standards and technique which have been adopted and which apply to the results quoted in the present article.

Certificate of Quality of Rubber.

This certificate is based on the results obtained by the follow ing technique adopted in vulcanisation and testing in the Experimental Vulcanising Laboratory of the Institute.

Mixing. The standard mixing consists of 20 grammes of sulphur and 200 grammes of rubber. Three such mixings are made from the sample and from each mixing 4 equal portions of 40 grammes each are obtained for vulcanisation.

Curing. The mixings are kept for a period of 24 hours, after which they are cured in moulds in a steam autoclave at 149°C using a standard N.P.L. thermometer to control the temperature.

Method of Testing. 24 hours after vulcanisation two rings are cut from each portion of 40 grammes by means of a Schopper ring-cutting machine (rotating knives) giving a ring of 3.80 millimetres width. The average thickness of the ring is obtained by a micrometer screw gauge applied to the uncut test-piece

The rings are stretched to breaking point on a Scott Rubber Testing Machine which records automatically on a chart the stress/ strain relationship for the sample.

Corresponding to each period of cure, 6 rings are tested, and the average of the six results recorded.

Modulus. This is the load in kilogrammes per square millimetre obtained at an elongation of 650 per cent at a cure of 80 minutes. This gives a figure which is used to compare different samples. It is more accurate than the figure for tensile strength at break which is subject to a large experimental error.

Maximum Tensile Strength at Break. The load at break is expressed in kilogrammes per square millimetre. (Kgs/mm²).

Rate of Cure. This is the period of cure which gives the maximum tensile strength at break.

Experimental Error. The results recorded for modulus are used as criteria for classifying the rubber tested. The actual figures are subject to an experimental error not greater than $\pm 4\%$.

The figures for "maximum tensile strength" are subject to a greater experimental error and should only be regarded as an indication of the normality of the sample.

The results obtained for the samples in this report may be compared with the following figures which are the average values for standard types of rubber examined and recorded in the Institute's laboratory.

Type.		Modulus or Load at 650 per cent elongation at 80 minutes cure, Kgs/mm ²	Cure giving maximum tensile strength at break Minutes.	Maximum tensile strength at break Kgs/mm ²
Smoked Sheet	[0.76	80 to 90	1.43
Unsmoked Sheet		0.61	90	1.38
No. 1. Crepe		0.60	100	1.37
Fine Hard Para	•	0.83	90	1.48











