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SOME VULCANIZATION TESTS OF GUAYULE RUBBER

BY

D. SPENCE, Research Associate
C. E. BOONE, Assistant Physicist
Bureau of Standards

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deterioration that the present communication has to deal. Vulcanization tests have been made on a number of samples, prepared for the most part from guayule shrub grown in central California, under carefully controlled conditions and shipped to Torreon in Mexico for extraction; some tests will also be found on the native Mexican product, derived from the shrub collected at random from the desert regions of northern Mexico and suitably treated. It should be noted, however, that in either case a considerable period of time, not less than two months, elapsed between the time of harvesting and the time of extraction of the shrub during which certain changes in the rubber in the plant were going on. As the results here show and as experience has taught, maximum improvement in physical quality is only attained when the shrub is treated before the physical degeneration of the rubber therein has taken place.

The means by which gauyule is obtained from the shrub at the present time are as follows: The whole shrub, root as well as branches, is first crushed by a series of crusher rolls in presence of water. After crushing, the mass is fed continuously with additional water to a tube mill or mills. These mills contain flint pebbles and revolve slowly on a horizontal axis. Their action on the shrub depends on the rolling motion of the pebbles in the presence of water and results in a further disintegration of the fiber out of the shrub and the agglomeration, or "worming," of the rubber substance into small, round, spongy particles, which vary in size with the condition of the shrub and the time of milling. The fine particles of rubber, or "worms," being lighter than water, float on the surface of the discharge liquor from these tube mills, while the bulk of the fiber and other impurities sinks and can readily be separated. The rubber "worms," which rise to the surface of the settling tanks into which the liquor from the tube mills is run, are skimmed off and collected. The "worms" are further purified and are then worked into sheets on sheeting rolls, well washed and dried.

In the tests which follow advantage has been taken of some of the most recent developments in rubber compounding. For example, zinc oxide and stearic acid have been used throughout, preliminary determinations having shown that while the acetone soluble substances of guayule rubber appear to produce the dissolution of the zinc oxide to some extent, on the whole better results are obtained when a small amount of a fatty acid, such as stearic, is added to the mix. Diphenylguanidine was used as an accelerator in all tests, except those given in Table 10. It is felt, however, that better results will be attained in the vulcanization of guayule rubber by a study of different organic accelerators under varying conditions.

Compounds of guayule rubber without admixture with other rubber on standard pure gum, zinc oxide, and gas-black formulas

have been made and tested in the usual way; also some tests have been made by substituting one-half of the plantation rubber in a given formula by guayule rubber. In this connection and in order to eliminate the effect of the resins present in the guayule rubber, and thus admit of a better comparison between the rubber of Hevea and that from guayule, a quantity of rubber free from resins was prepared from one of the samples of special guayule by extraction of the latter by means of acetone.

Values for tensile strength and ultimate elongation were determined according to standard procedure, as described in Bureau of Standards Circular No. 232. In regard to the figures for permanent set here given, these were obtained by stretching the test pieces (500 per cent for pure gum compounds and 400 per cent for compounded stocks) for 10 minutes, then releasing and measuring the "set" at intervals until permanent deformation was obtained.

Unless otherwise specified, the formulas used throughout for the testing of these materials were as follows:

	Pure-gum formula, parts by weight	Zinc-oxide formula, parts by weight	Gas-black formula, parts by weight
Rubber	100	100	100
Zinc oxide	3	80	28
Gas black			36
Sulphur	6	6	5
Stearic acid	1.5	1.5	1.5
D. P. G	.5	.5	.5

The sheets were cured for varying lengths of time at 40 lbs./in.^{2} steam pressure (141.7° C.) in an open platen vulcanizing press.

In Tables 1 and 2 will be found physical tests of three samples of guayule rubber prepared from the ordinary run of Mexican shrub taken from storage and treated as follows:

(A) Shrub (2 months old), milled immediately after crushing.

(B) Same shrub, milled two weeks after crushing.

(C) Same shrub, milled two weeks after crushing and treated for preservation of shrub.

TABLE 1.—*Mexican wild guayule on pure-gum formula*

Cure (minutes)	A			B			C		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs./in.^{2}	*Per cent*	*Per cent*	*Lbs./in.^{2}*	*Per cent*	*Per cent*	*Lbs./in.^{2}*	*Per cent*	*Per cent*
30	1,925	845		1,790	825		2,255	840	
45	1,975	765	5	2,070	780	8	2,560	765	7
60	1,965	745		2,035	745		2,545	740	
75	1,945	750		1,835	710		2,450	720	

Table 1 shows the deterioration which has occurred and is indicative of the effect of treatment to prevent the deterioration of the shrub.

TABLE 2.—*Mexican wild guayule on zinc oxide formula*

Cure (minutes)	A			B			C		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs./in.²	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*
45	1,990	805	---	1,890	740	---	2,590	775	---
60	2,040	755	11	1,845	695	24	2,675	730	17
75	1,985	725	---	1,905	670	---	2,555	715	---

The effect of the treatment of the shrub is likewise apparent (Table 2), using a zinc oxide formula.

In Table 3 will be found figures for the product from the cultivated shrub grown in California and shipped to Mexico for extraction. "A" represents the product extracted as received and "B" the same product extracted after crushing and treatment for the betterment of the rubber.

TABLE 3.—*Cultivated Californian guayule rubber on zinc oxide formula*

Cure (minutes)	A		B	
	Tensile strength	Elongation	Tensile strength	Elongation
	Lbs./in.²	*Per cent*	*Lbs./in.²*	*Per cent*
30	---	---	2,670	740
45	1,940	740	2,650	715
60	2,120	715	2,430	645
75	2,100	685	2,410	635
90	1,690	620	---	---

The results in the above table further show the effect of treating the shrub to prevent deterioration. A marked improvement of the tensile properties is apparent.

In the following tables (4, 5, and 6) are given physical tests on a lot of Californian cultivated shrub shipped to Mexico and extracted two months later. This lot of about 80 tons of shrub was run through in the usual way, except that part of the lot was treated to better preserve the quality of the rubber. Tests were made on air-dried and vacuum-dried samples, using pure gum, zinc oxide, and on gas-black formulas.

TABLE 4.—*Californian cultivated guayule on pure gum formula*

Cure (minutes)	C. G. V.[1]			C. G. P. A.[2]			C. G. P. V.[3]		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs./in.²	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*
15	---	---	---	2,310	845	---	2,535	800	---
30	1,630	905	---	2,395	740	11	2,400	695	12
45	1,990	855	---	2,290	695	---	2,315	685	---
60	2,035	815	8	---	---	---	---	---	---

[1] C. G. V., Californian cultivated guayule rubber, vacuum dried.
[2] C. G. P. A., Californian cultivated guayule rubber, treated and air dried.
[3] C. G. P. V., Californian cultivated guayule rubber, treated and vacuum dried.

TABLE 5.—*Californian cultivated guayule on zinc oxide formula*

Cure (minutes)	C. G. V.[1]			C. G. P. A.[2]			C. G. P. V.[3]		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs. /in.²	*Per cent*	*Per cent*	*Lbs. /in.²*	*Per cent*	*Per cent*	*Lbs. /in.²*	*Per cent*	*Per cent*
30	1,530	865		2,300	755	17	2,295	745	18
45	1,890	835		2,080	685		2,190	675	
60	1,975	770	17	2,080	655		2,030	635	
75	1,925	765		1,830	605		2,245	680	

[1] C. G. V., Californian cultivated guayule rubber, vacuum dried.
[2] C. G. P. A., Californian cultivated guayule rubber, treated and air dried.
[3] C. G. P. V., Californian cultivated guayule rubber, treated and vacuum dried.

TABLE 6.—*Californian cultivated guayule on gas-black formula*

Cure (minutes)	C. G. V.[1]			C. G. P. A.[2]			C. G. P. V.[3]		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs. /in.²	*Per cent*	*Per cent*	*Lbs. /in.²*	*Per cent*	*Per cent*	*Lbs. /in.²*	*Per cent*	*Per cent*
30							2,510	715	
45	1,995	790		2,430	690		2,525	680	27
60	2,135	765		2,620	655	27	2,475	655	
75	2,145	765	27	2,615	625		2,440	645	
90	2,020	720		2,355	625				

[1] C. G. V., Californian cultivated guayule rubber, vacuum dried.
[2] C. G. P. A., Californian cultivated guayule rubber, treated and air dried.
[3] C. G. P. V., Californian cultivated guayule rubber, treated and vacuum dried.

The results shown in the above tables confirm the previous results. In the three cases, using pure gum, zinc oxide, and gas-black formulas, better properties are obtained with the rubber from the treated shrub.

In the following tables (7, 8, and 9) will be found a comparison of the tensile and elongation properties of guayule rubber with those of a sample of first latex crêpe on the three test formulas used throughout. For the purpose of the test a large quantity of the Californian grown rubber, treated to better preserve the inherent quality of the rubber and tested under Tables 4, 5, and 6, was first extracted with acetone while still in the "worm" stage. The acetone soluble substances of the rubber were reduced to less than 1 per cent, and the rubber when air-dried was in the form of a hard, dry crêpe, with no signs of stickiness.

TABLE 7.—*Comparison of Californian cultivated guayule with first latex crêpe on pure gum formula*

Cure (minutes)	First latex crêpe			Extracted guayule rubber		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs./in.²	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*
30	2,680	785		2,810	725	
45	2,885	750	3	3,220	715	10
60	2,860	765		2,900	660	
75	2,435	785				

TABLE 8.—*Comparison of Californian cultivated guayule with first latex crêpe on zinc oxide formula*

Cure (minutes)	First latex crêpe			Extracted guayule rubber		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs./in.²	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*
30	3,125	695	----	2,650	730	----
45	3,430	680	11	3,000	685	12
60	3,275	665	----	2,845	665	----
75	2,770	590	----	2,780	675	----

TABLE 9.—*Comparison of Californian cultivated guayule with first latex crêpe on gas-black formula*

Cure (minutes)	First latex crêpe			Extracted guayule rubber		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs./in.²	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*
45	3,255	650	----	2,945	600	----
60	3,800	635	17	3,450	570	27
75	3,855	610	----	3,375	540	----

The guayule rubber when extracted with acetone shows tensile properties comparable with plantation crêpe. Using the pure gum formula the properties are enhanced, whereas the zinc oxide and gas-black compounds are slightly poorer in their physical properties.

In Table 10 will be found a comparison of plantation crepe with acetone extracted and processed Californian guayule, also with the same materials without extraction. This comparison was made on the following pure gum formula.

	Parts by weight
Rubber	100.0
Zinc oxide	3.0
Sulphur	2.0
Tetramethylthiuramdisulphide	.34
Ethylidine aniline	.50

TABLE 10.—*Comparison of Californian guayule with plantation crêpe on pure gum formula*

Cure (minutes)	Plantation crêpe		Extracted and processed guayule		Processed guayule	
	Tensile strength	Elongation	Tensile strength	Elongation	Tensile strength	Elongation
	Lbs./in.²	*Per cent*	*Lbs./in.²*	*Per cent*	*Lbs./in.²*	*Per cent*
5	4,270	730	3,800	685	2,665	830
10	4,280	670	4,105	650	2,640	800
15	3,860	655	3,755	630	2,110	770
20	3,350	625	3,450	615	1,845	745

The extracted guayule in this case possesses tensile properties practically equal to those of the plantation crêpe.

In Table 11 will be found a comparison of plantation crêpe with Californian grown guayule on a replacement basis. In this case the standard zinc oxide formula was used, and 50 per cent of the plantation crêpe contained in the original mixing (No. 1) was replaced by an equal weight of acetone extracted and treated guayule in No. 2 and a like amount of the original Californian rubber from which the extracted product was prepared in No. 3.

TABLE 11.—*50 per cent replacement of plantation crêpe by Californian-grown guayule (extracted and unextracted) on standard zinc oxide formula*

Cure (minutes)	Plantation crêpe (No. 1)			Californian-grown guayule					
				Extracted (No. 2)			Unextracted (No. 3)		
	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set	Tensile strength	Elongation	Permanent set
	Lbs./in.²	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*	*Lbs./in.²*	*Per cent*	*Per cent*
30	1,725	675	----------	2,000	670	----------	2,515	705	----------
45	3,150	665	11	3,110	640	12	2,890	645	14
60	3,175	625	17	2,995	610	17	2,525	610	20

The figures here cited make no pretense to be a complete evaluation of the physical properties of guayule rubber. They represent merely the results of a preliminary and somewhat superficial examination of the product from the guayule shrub grown under agricultural conditions in California and prepared by the best methods as yet possible. In the first place, all the samples of rubber tested were obtained from shrub 2 months or more old before extraction; it is probable that better results would have been obtained from the same shrub by prompt treatment of it to inhibit deterioration. The tests, furthermore, have all been made on test formulas and under vulcanizing conditions chosen more or less arbitrarily, without regard to the specific requirements for the best vulcanization of this rubber. There is still much left to be determined in this connection.

Notwithstanding the limitations of necessity imposed on the results so far, the tensile and elongation properties of the products prepared from the guayule shrub by suitable treatment indicate that, when properly prepared, the rubber from guayule will compare favorably with that from Hevea and can be used to a large extent as a direct equivalent therefor without appreciable diminution of the tensile elongation product. The great improvement in the product from guayule to be derived from the successful development of means to prevent the deterioration of the rubber in the shrub is clearly indicated, and it is by no means to be assumed that the last word has been said in this connection.

The values obtained for permanent set are high by comparison with similar samples of plantation rubber, but here, again, much work remains to be done before definite conclusions can be reached.

Guayule rubber mixes readily with compounding ingredients, because it appears to have a greater capacity for dispersing them than ordinary rubber. This fact calls for a complete investigation of the best means for the utilization of the product to the best advantage.

Since the foregoing tests were made aging tests have been completed upon some of the vulcanized samples after eight months of storage in a box exposed to the outside temperatures, but protected from the light. The results before and after aging are given in Table 9.

TABLE 9

	Reference	Cure	Original test		After 8 months	
			Tensile strength	Elongation	Tensile strength	Elongation
		Minutes	*Lbs./in.²*	*Per cent*	*Lbs./in.²*	*Per cent*
Table 1	A	45	1,975	765	1,420	685
	B		2,070	780	1,110	570
	C		2,560	765	2,115	665
Table 4	C. G. V	45	1,990	855	1,590	770
		60	2,035	815	1,550	710
	C. G. P. A	15	2,310	845	2,095	770
		30	2,395	740	1,975	705
	C. G. P. V	30	2,535	800	2,400	690
		45	2,400	695	2,170	650
Table 5	C. G. V	45	1,890	835	1,735	700
		60	1,975	770	1,640	645
	C. G. P. A	30	2,300	755	2,055	675
		45	2,280	685		
	C. G. P. V	30	2,295	745	2,195	650
		45	2,190	675	1,995	615
Table 6	C. G. V	60	2,135	765	1,440	580
		75	2,145	765	1,280	510
	C. G. P. A	45	2,430	690	1,910	575
		60	2,620	655	1,810	500
	C. G. P. V	45	2,525	680	2,095	580
Table 7	First latex	45	2,885	750	2,465	655
		60	2,860	705	2,650	695
	Guayule	30	2,810	725	2,320	660
		45	3,100	715	2,495	615
Table 8	First latex	30	3,125	695	2,915	700
		45	3,430	680	2,860	650
	Guayule	30	2,650	730	2,550	640
		45	3,000	685	2,565	605

These results show the usual dropping off in tensile and elongation properties with aging. They also show the marked improvement in this regard brought about by suitable treatment of the shrub. The drop in tensile and elongation in the case of the treated product compares favorably with the results obtained with first latex crêpe under similar conditions. (Tables 7 and 8.)

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