

PREPARATION OF HEVEACRUMB FROM FIRST FRACTION COAGULUM

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SUMMARY

Production of Heveacrumb in a pilot plant installed at Peenkande Estate is discussed with special reference to conversion of first fraction coagulum to Heveacrumb. The information presented in this paper is intended to assist in the evaluation of Heveacrumb rubber from first fraction coagulum. Production records, from the time the pilot plant has been commissioned for regular production, are set out with analytical data on raw rubber properties of each consignment lot. Data illustrating the behaviour of first fraction coagula in two representative compounds, both in the form of crumbled rubber and pale crepe, are also given.

INTRODUCTION

A pilot plant to upgrade low quality rubber by the Heveacrumb process was set up at Peenkande Estate, Uda Karawita, by the Rubber Research Institute. The raw material for the plant was mainly derived from first fraction coagulum which is a by-product in sole screpe and light-coloured pale crepe manufacture. It may be worthwhile to mention at this stage that the total production of latex crepe (of quality ranging from 1X to 2) and sole crepe in Ceylon, for the year 1969, was approximately 40,000 tons. It is estimated that about 2,250 tons of fraction coagulum would have been concurrently processed as a by-product of lower quality.

The pilot plant has served to evaluate the economics of conversion of fraction coagulum into block rubber sold to a technical specification (Karunaratne & Thompson, 1969). The cost of manufacture was worked out on the basis of recorded hours of work at each point and it was suggested in the analysis that reduction of costs will accrue with more mechanisation, resulting in less labour for handling. The cost of production was worked out at 3.49 cts per lb of dry rubber. This is inclusive of the cost of castor oil and the cost of polythene for wrapping, but neither the cost of crating nor the transport costs have been taken into consideration. Since late the price of castor oil has increased due to a scarcity of this commodity in the world market and this has added a further one cent to the cost of manufacture. This analysis has shown that, basically, block rubber production from low grades, be it fraction coagulum or any other field coagula, by the new process of manufacture will result in the reduction of processing costs and could add to the foreign exchange earnings from the marketing of natural rubber (NR). A redeeming feature as far as the Peenkande factory is concerned, where the pilot plant is operated, is the ease of congestion in the factory specially during the high cropping months. The experiment has clearly shown that estates can pool their resources to manufacture new process rubbers from their own raw material and at the same time help them out of congestion due to increase in crop due to tapping of high-yielding clones and due to more replanted areas coming into tapping.

The Heveacrumb pilot plant has been in operation since January 1968 and it was not until the beginning of 1969 that a trial five-ton consignment of Heveacrumb prepared from first fraction coagulum could be despatched to a marketing organisation in France, for market evaluation. Subsequently this first fraction crumb rubber has been sampled to various customers, who have successfully incorporated them into the same type of applications where they have developed a market for equivalent grades of Malaysian origin.

The main equipment of the pilot plant consists of a set of two creping rollers, adopted for crumbling, a single stage crumb rubber dryer, constructed by the General Electric Company in Malaysia based on a design made by the Rubber Research Institute of Malaya (RRIM/GEC type of dryer) and a 30-ton baling press. The single stage dryer has an output of approximately 100 lb of dry rubber per hour and on an average eight-hour shift the estate is in a position to work out a crumb rubber output of approximately 700 lb. Batch-wise the production of crumb rubber has been restricted to lot sizes of 700 lb or less; rubber with consistent properties has been produced and the estate was in a position to market 50 tons of Heveacrumb from first fraction coagulum by the end of 1969.

Processing procedure

Field latex, stabilized with sodium sulphite at the rate of 0.15 lb per 100 lb of dry rubber, is collected from three separate divisions, and a 15% first fraction is removed from the bulked and undiluted latex, from each division, prior to coagulation. For the manufacture of extremely light-coloured sole crepe it has been the estate's practice to add upto 0.5 lb of sodium bisulphite per 100 lb of dry rubber prior to taking a fraction. Reduction of the dosage of bisulphite to 0.05%, based on dry rubber, as recommended for Heveacrumb manufacture (Graham & Morris, 1966) did not have a significant effect on the rate of drying of crumb rubber but resulted, as expected, in a noticeable darkening of the sole crepe produced from the fractionated latex. Removal of this enzymic discolouration, once it sets in, is a difficult job even when the reduction of bisulphite dosage is made good or better at a later stage prior to complete coagulation. Carotenoids and other non-rubber constituents concentrated in the first fraction caused a significant browning of the end-product irrespective of the adjustment of other additives *viz.* sodium sulphite and bisulphite.

Blending process

The fraction coagulum from the three divisions is blended by four passes through the maserator (doubling after each pass) into a mat approximately $\frac{1}{4}$ in. thick and passed through a castor oil applicator which works on a lick roll principle. This introduces a smear of castor oil on the surface of the mat. The main disadvantage in the castor oil applicator is that accurate metering of castor oil is difficult and as such there is a tendency for uneven and sometimes excessive smearing of castor oil on the surface of the mat.

Crumbling process

Crumbling is achieved by passing the mat of rubber, after castor oil application, twice through the first roller and once through the second roller. These rollers, incidentally, are a set of creping rollers which have been regrooved to facilitate crumbling. Sometimes, two passes, once through the first roller and once through the second roller are sufficient to crumble the rubber satisfactorily. Crumbled rubber is soaked in water prior to loading into the dryer.

Drying process

The wet particles of rubber are then loaded into trays and each full tray of crumbled wet rubber is conveyed by hand to the crumb rubber dryer. The one-stage dryer is heated by a Woods heat exchanger fed with diesel fuel. The trolley into which the rubber is loaded holds 20 trays and the 12-in. bed of granules in each tray takes about six hours to dry. Operating on crumbled rubber the dryer capacity is approximately 600 lb, equivalent to a drying rate of 100 lb per hour. Drying is carried out by through circulation convection drying at atmospheric pressure, where heat is conveyed by air which at the same time removes the moisture. Any

obstacle to through circulation will result in over-drying in some areas and under-drying in other areas. Excessive softening of the rubber can lead to consolidation of the rubber in the bottom of the tray and this has frequently resulted in a stewing effect causing the rubber to be tacky and such rubber has to be remilled into blanket crepe. Heveacrumb particles are discrete when wet, but their natural tack is returned during drying. When drying is complete the entire bed becomes a semi-coherent mass which could be lifted and handled as a block. In addition to humidity, air velocity, bed depth and pressure drop, drying characteristics are affected by temperature and our experience with fraction rubber is that drying temperature should not exceed 105°C.

Baling

The 13" × 26" blocks of dry crumbled rubber, each weighing approximately 35 lb, are removed from the drying trays and lots of 75 lb are weighed out, taking one block each from two drying trays plus a make up balance from a third tray. Each lot of weighed rubber is transferred to a 30-ton hydraulic press and pressed together to give a compact bale. The press box has been adjusted from time to time to turn out two standard bale sizes viz. the standard synthetic bale size of 28" × 14" × 7" or the bale size of 22½" × 15" × 7" which fits into a standard pallet measuring 48" × 40" × 46". The bale is wrapped by hand in 150 gauge polythene and heat-sealed. A 800 gauge polythene film has been used subsequently.

The bales wrapped in 150 gauge polythene are later shipped in kraft paper bags to give further protection and stencilled on the outside with the appropriate identification marks. Since the introduction of the 800 gauge polythene film shipping in paper bags has been discontinued.

Gross contamination

Contamination by paper, metallic particles, inclusion of virgin rubber and undried areas in the centre of certain bales have been reported.

Most of these defects can be checked and prevented by proper care and attention during manufacture. A problem of great concern is the undried areas in the middle of certain bales. The estate has been advised to check each block of crumbled rubber for wet patches immediately after removal from the drying trays.

Data on batch-wise production

Six bales from each batch of not more than ten bales were sampled and separately tested as indicated below. The sample comprised two corner samples of approximately one pound total weight, and this was homogenised before testing at the Rubber Research Institute.

Sample homogenisation

The sample is passed ten times through the rolls of a laboratory mill, heated to 70 ± 50°C, and running at friction speeds at a nip setting of 1.25 mm rolling and upending between passes.

RESULTS AND DISCUSSION

Test data on twelve 5-ton consignment lots of Heveacrumb from fraction rubber are given in Table 1. Sampling, in the case of the first few batches have been extensive. Samples were tested from each compartment of the dryer prior to pressing. Once the production was stabilized sampling intensity was reduced to six samples from each batch, which never exceeded 700 lb.

The nitrogen values of fraction rubber fluctuated between 0.4 and 0.6 due partly to poor control of the fractionation and partly to batch to batch variation in the non-rubber constituents. It is important to note that all nitrogen values recorded fell below 0.65 and within the specification limits for nitrogen as laid down in the Standard Ceylon Rubber (SCR) specifications (Anon, 1969).

TABLE I
TEST DATA ON FIVE-TON CONSIGNMENT LOTS OF
HEVEACRUMB FROM FRACTION COAGULUM



Lot No.	No. of samples tested	SCR tests (mean values)						
		Dirt % wt	V.M. % wt	PRI	N ₂ % wt	Ash % wt	Cu (ppm)	Mn (ppm)
2	143	0.019	0.5018	81.8	0.435	0.216	<8.00	<10.00
3	111	0.022	0.4747	84.6	0.489	0.296	<8.00	<10.00
4	107	0.020	0.4706	84.2	<0.600	<0.500	<8.00	<10.00
5	108	0.020	0.4205	82.8	<0.600	<0.500	<8.00	<10.00
6	81	0.018	0.4388	83.9	<0.600	<0.500	<8.00	<10.00
8	21	0.018	0.4352	85.3	<0.600	<0.500	<8.00	<10.00
9	16	0.016	0.4280	88.2	0.470	0.230	1.70	1.00
10	15	0.019	0.4485	87.4	<0.600	<0.500	<8.00	<10.00
11	18	0.022	0.4200	80.9	0.450	0.210	0.13	0.96
12	18	0.021	0.4000	83.5	0.430	0.150	2.00	2.00
13	20	0.028	0.2900	83.4	0.470	0.230	2.00	2.00
14	18	0.022	0.2800	73.1	0.470	0.230	2.00	2.00

For most batches an accurate determination of copper and manganese was not attempted and it was sufficient to ascertain only copper and manganese limits specially because the PRI values were in the region of 80. The exact values of copper and manganese content in fraction rubber are calculated for lot No. 9 and the figures quoted for this lot could be considered as average for this type of rubber. In the case of dirt, a $3 \times$ standard deviation factor has been added to the final dirt value.

Table 2 gives the vulcanisate properties of a pure gum compound based on the ACS 1 formula and a comparison is made with fraction rubber of Malaysian origin. Lower scorch rate in the case of fraction rubber from Peenkande is suggestive of a dilution of the effective non-rubber constituents which would play a part in the modification of the cure rate of the ACS 1 mix. This is also reflected in lower tensile strength and modulus values.

TABLE 2

VULCANISATE PROPERTIES ON THE ACS 1 COMPOUND

(For comparison, results on typical fraction crumb of Malaysian origin are given)

Property	Peenkande yellow fraction crumb	Typical fraction crumb of Malaysian origin
Tensile strength, kg/cm ²	208.0	225.0
Elongation at break, %	770.0	700.0
Modulus at 100% elongation, kg/cm ²	8.4	8.4
Modulus at 300% elongation, kg/cm ²	19.7	14.2
Modulus at 600% elongation, kg/cm ²	75.6	93.2
Lupke resilience, % at 30°C	71.0	74.0
Hardness, IRHD	41.0	45.0
Mooney scorch at 120°C, min	9.5	5.0

Behaviour of fraction rubber in tread and carcass formulations

The tread and carcass formulations employed were based on a conventional sulphur/sulphenamide accelerator system and were chosen as being typical of present day practice. Compounding comprised base stock mixing in a laboratory mixing mill with subsequent addition of sulphur and accelerator in masterbatch form. Details of compounding are set out in the appendix.

Table 3 gives the vulcanisate properties of typical tyre tread and tyre carcass compounds based on fraction crumb rubber and fraction pale crepe. Tensile properties of the two rubbers are very close, fraction crumb having a slightly higher tensile strength and modulus. Laboratory abrasion tests (Dupont) show that fraction pale crepe has inferior abrasion resistance compared with fraction crumb. Whilst recognising the limitations of such tests it is suggested that, under service conditions, tyre treads based on fraction pale crepe may wear faster than tyre treads based on fraction crumb. Resilience values are very close in the two rubbers, but the quality of black dispersion controlled by mixing arrangements is certain to dominate the situation and swamp any small differences.

TABLE 3

VULCANISATE PROPERTIES OF TREAD AND CARCASS COMPOUNDS
(Cured at 140°C/40 min)

Vulcanisate property before ageing	Fraction crumb rubber		Fraction pale crepe	
	Tread compound	Carcass compound	Tread compound	Carcass compound
Tensile strength, kg/cm ²	252.0	258.0	248.0	250.0
Elongation at break, %	560.0	608.0	540.0	610.0
Modulus M 100, kg/cm ²	27.2	20.6	24.5	21.5
Modulus M 300, kg/cm ²	111.9	71.7	108.5	85.2
Modulus M 600, kg/cm ²	—	251.8	—	248.1
Hardness, IRHD	65.0	65.0	65.5	66.5
Lupke resilience, %	59.5	70.5	59.5	71.0
De Matti flex life, K/C	80.0	55.0	97.0	68.0
Dupont abrasion index	106.0	—	92.0	—

Vulcanisate property after ageing for 14 days at 70°C

Tensile strength, kg/cm ²	220.5	240.0	216.0	237.1
% retention of T.S.	87.7	93.0	87.1	94.8
Elongation at break, %	420.0	546.0	400.0	555.0
Modulus M 300, kg/cm ²	155.8	105.6	159.9	104.0

CONCLUSIONS

Even though a sufficiently large quantity of Heveacrumb from fraction coagulum has not been produced and consumed, the results so far obtained have proved that Heveacrumb from fraction coagulum can be satisfactorily used in applications where good static properties and ageing resistance are of paramount importance. The results even indicate that this rubber has good dynamic properties. This opens up the possibility of suitably mixing good quality field coagula, predominately of the cup lump grade, with fraction coagulum and this advantage may be exploited when it comes to a question of large estates having to pool their stocks of field coagula to build up sufficient material to run a block rubber plant. A policy to upgrade off-quality rubber in this manner will also help the larger estates to reduce the burden of finding extra drying accommodation which would necessarily involve additional capital commitments.

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APPENDIX

DETAILS OF COMPOUNDING
(all quantities in parts by weight)

(A) Tread compound		(B) Carcass compound	
(Base stock)		(Base stock)	
Test rubber	95	Test rubber	95
HAF black	45	FEF black	30
Dutrex R	5	Zinc oxide	5
Zinc oxide	4	Stearic acid	2
Stearic acid	1.5		
Nonox HFN	1.5	Santoflex DPA	2
<i>Standard vulcanizing masterbatch</i>		<i>Standard vulcanizing masterbatch</i>	
Test rubber	5	Test rubber	5
Sulphur	2.5	Sulphur	2.5
CBS	0.5	CBS	0.5

Mixing

Basic mixing conditions were the same for both tread and carcass compounds,

QUESTIONS AND ANSWERS

Question : Isn't it advisable for future factories to adopt the mechanical process due to the following reasons ?

- (a) Increase in price of castor oil has increased the cost of production by one cent per lb ;
- (b) It is a problem to control the addition of castor oil as a smear on the crepe mat ;
- (c) There will be saving of foreign exchange if castor oil is not used ;
- (d) Mechanical process has been a success. Addition of a creper will improve quality further. (Anon).

Answer : (a) The latest development in the Heveacrumb manufacture as reported by the Rubber Research Institute of Malaya is the use of a creper-hammermill instead of a set of crumbling rollers for size reduction. With this arrangement castor oil is used not as a crumbling agent but as an agent to disperse aggregates of particulate rubber, and it has been worked out that the castor oil requirement can be stepped down to about 0.1% on the basis of dry rubber. Thus the amount of castor oil is reduced to 1/7th the recommended quantity if it were used as a crumbling agent using a set of rollers for crumbling. This will counter very favourably with the increase in price of castor oil.

- (b) The methods available for the application of castor oil have their shortcomings, but the castor oil content if reduced to about 1/7th the usual amount as indicated in the answer to question (a), there is a greater margin of safety and accurate control becomes less important.
- (c) There will be a saving of foreign exchange when the amount of castor oil used is reduced to 1/7th the amount used previously.
- (d) Choice of the process is dependent on the type of material one handles. There are a number of processes, both mechanical and mechano-chemical, for block rubber manufacture and all these processes have proved to perform satisfactorily.

Question : Can a cheaper oil readily available be used in place of castor oil — say coconut oil in crumb rubber ? (Mr. H. G. S. Mendis).

Answer : The oil should be incompatible with the rubber.

A wide range of such oils have been tried out as a crumbling agent for natural rubber. Castor oil has proved to be the most efficient. Undesirable qualities of coconut oil will be necessary if it were to act as a crumbling agent for rubber, and therefore the use of coconut oil for this application has been discouraged.

Question : What is the heat build-up properties of the first fraction rubber Heveacrumb in comparison with Heveacrumb from ordinary rubber latex ? (Anon).

Answer : Heat build-up as assessed by the resilience test on a standard tyre tread and carcass compound is comparable for the two types of rubbers, namely fraction crumb rubber and whole latex crumb rubber.