

EASY PROCESSING NATURAL RUBBER*

By

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SUMMARY

The competitive pressures that are applied by the synthetic rubber (SR) industry compel the natural rubber (NR) producing industry to plan so that NR is competitive in consumer product fabrication. Hot room treatment and premastication, which are necessary steps in the processing of NR and not SR, can be eliminated by adding to field latex a mineral oil which is a physical plasticiser at approximately 11% on the rubber and a peptiser which is a chemical plasticiser at approximately 0.2% or 0.3% on the rubber. This could be conveniently done in conventional ribbed smoked sheet (RSS) producing factories. The field latex is coagulated and converted to a grade of ribbed smoked sheet (RSS) called easy processing RSS.

INTRODUCTION

Nearly all the NR producing countries produce for export. The situation is different for synthetic rubber (SR), as only about one quarter of the SR produced enters international trade. Hence consumer requirements will have to be the determining factors in the processing of NR by producing countries. As tyre manufacturers consume more than 60% of NR, it is necessary to plan NR processing so that latex rubbers can go into tyres. A selection of natural rubber or synthetic is based not on market price alone, but on the total cost to the consumer on equivalent practical processing terms or marketable end use properties. Bekema (1969) has compared the processing steps of NR with that of SR, from unloading at the factory to dumping from the Banbury, and states that NR goes through four extra processes, viz. cleaning bale surfaces, hot room treatment (removal of crystallisation hardening), bale cutting, and plasticating (reduction of viscosity). Packing of NR in small 33½ kg polythene bales has overcome the deficiencies of bale cleaning and cutting.

CV rubber

Wood (1952) observed that large permanent increases in Mooney viscosity, of the order of 30 to 40 units, occurred when freshly prepared raw or masticated rubber was stored under conditions of low relative humidity. Chin Peng Sung (1969) has confirmed the findings of Sekhar (1964) that treatment of latex with hydroxylamine hydrochloride (at 0.15% by wt. on the rubber) prevents this storage hardening. This rubber, which is called viscosity stabilized (CV) of Mooney viscosity 60 ± 5 , could be used without plastication. In the manufacture of CV rubber, careful selection and blending of latices are necessary. Since there are in Sri Lanka clones such as PB 86, RRIC 36, WR 101, PB 28/50 and Tjir 1, which give according to Chin Peng Sung (1969), rubber with Mooney viscosity values of over 75 even after treatment, the manufacture of CV rubbers from latex, especially when the clones mentioned above preponderate, may not be feasible. CV rubber is fetching a premium of several Malaysian cts per kg over RSS 1. The use of CV rubber does not eliminate hot room treatment which is necessary in cold countries because of the crystallisation in the rubber. The world's production of CV rubber is approximately 1% of total NR production.

* Paper presented at the 6th Technical Seminar of the Indian Rubber Manufacturers' Research Association on 18th December 1972.



Addition of mineral oil

The addition of a mineral oil to latex before coagulation to make a tyre rubber, has come into prominence recently, even though initial work was done a long time back (Wentworth 1939). The Natural Rubber Producers' Research Association has stated that the addition of 11% of mineral oil on the d.r.c. to field latex gives an easy process rubber which undergoes little crosslinking or crystallisation on storage (Anon 1971). This should therefore eliminate both hot room and plasticator treatments. However, this type of rubber may have different flow properties from that obtained by premastication.

Fah & Chuan (1972) have described a process involving the coagulation of a latex oil mixture with wet crumb rubber. Malaysia's formula for a tyre rubber is a blend of 30 latex, 30 unsmoked sheets, 30 field grades and 10 plasticiser (a rubber process oil) and converted into block rubber. This rubber is now being evaluated by the RRIM in consumer factories but may have to be treated during manufacture with hydroxylamine also to give a viscosity of 60 ± 5 .

Addition of peptising agents

The plasticity of NR could be reduced by the addition of a peptiser which is a chemical plasticiser. Hastings (1939) investigated the incorporation of emulsions of "peptising agents" such as thiobetanaphthol into latex prior to coagulation and the subsequent softening of the dry rubber induced by the drying treatment to which the wet sheet is subjected. It was shown that the temperature used in the drying treatment affected the softness of the dry rubber. Hence he recommends that the strictest control of temperature be maintained in the smokehouse during smoking and hence the more modern type of smokehouses would be able to do this more efficiently. The storage of rubber containing peptisers indicated that a hardening occurred and in no single instance was a softening observed. This hardening is due to storage hardening. In Vietnam, in the 1950's, a rubber with a Mooney viscosity of around 35 was prepared and called FSP 35. The agent used to peptise FSP 35 rubber was either RPA 3 or Renacit 4 (Giger, 1952).

Giger (1953) has further stated that incorporation of the peptiser with the latex before coagulation and drying gives a rubber with improved processing properties and as the Mooney viscosity of such a rubber is much lower than that of smoked sheets, the plastication of this material requires less time and energy. According to the Natural Rubber Producers' Research Association, Technical Information Sheet No. 111, Socfin & Co. Ltd., Kuala Lumpur, Malaysia supplies Plastorub A and Plastorub B. These are softened natural rubbers prepared by the addition of peptising agents to latex prior to its conversion to sheet. Plastorub A has less peptiser than Plastorub B. Plastorub A has a Mooney viscosity of 50, is ready for compounding without previous breakdown, and contains approximately 0.5% of peptising agent.

Addition of mineral oil and peptiser

The plasticity of NR could be reduced by the addition of mineral oil which is a physical plasticiser and a peptiser which is a chemical plasticiser. The use on field latex of an aromatic oil at 10-12 phr and RPA 3 at 0.1 to 0.2 phr and coagulated without dilution may give a suitable "Easy process RSS for use in the Ceylon Tyre Industry" (Nadarajah *et al.*, 1971). In the work reported in this paper, both the physical plasticiser (aromatic oil at about 11 phr) and the peptiser (Renacit 4, RPA 3 or Renacit 7 at 0.1 to 0.3 phr) were added to field latex, which was processed into RSS and its processing and vulcanisate properties, investigated. RPA 3 (normal grade) contains 36.5% xylyl mercaptan. Preliminary results of the investigations into the preparation and properties of the resulting easy processing RSS are discussed in this paper.

EXPERIMENTAL AND RESULTS

The mineral oil used in our experiments was Ravolen X (high aromatic oil) obtained from the Ceylon Petroleum Corporation. A paste of Ravolen X was made by dissolving 2.5 parts by weight of coconut oil fatty acid in 100 parts by weight of Ravolen X, and mixing this with 17.5 parts by weight of 1% ammonia solution. The ammonia content of the slurry is approximately 0.15 per cent. This paste could be added after dilution with an equal volume of water to field latex. The amount added was such that the oil was about 11% weight of the rubber.

The peptisers used in our experiments were Renacit 4 (zinc pentachlorothiophenol) or Renacit 7 (zinc pentachlorothiophenol + activator) or RPA 3 (xylyl mercaptan). The peptiser (Renacit 4 or Renacit 7) at 0.2% or at 0.3% by weight of the rubber was dispersed in the Ravolen X before it was made into a paste. The use of a colloid mill or a high speed stirrer is necessary to get a good dispersion. The RPA 3 was emulsified with Dupanol OS (10 RPA 3, 1 Dupanol OS, 89 water) and added directly to the latex. The approximate d.r.c. of the field latex was obtained by the metrolac. A more accurate value can be obtained by the rapid drc method (Rubber Research Institute of Ceylon, 1970). The required amount of the oil emulsion was mixed while stirring into the field latex and formic acid added at the rate of about 2 g per kg dry rubber content as a 1% solution. This is approximately half the normal dosage used in pale crepe or RSS manufacture. The correct dosage of acid is at the point where bromocresol green indicator paper when dipped into the acidified latex gives a yellowish green colour (Rubber Research Institute of Ceylon, 1970). The coagulum is converted in the normal way into RSS and is called easy processing RSS.

The easy processing RSS obtained from latex of clone PB 86 was tested for mineral oil content by the acetone extraction method, for Mooney viscosity and for Wallace plasticity. The results obtained are given in Table 1.

TABLE 1
PROPERTIES OF EASY PROCESSING RSS

Sample	Mineral oil content by acetone extraction	Mooney viscosity	Wallace plasticity
Control	3.4	80	59
11% oil	12.3	64	44
11% oil and 0.2% Ren 4	11.9	67	42
11% oil and 0.3% Ren 4	10.7	66.5	41
11% oil and 0.4% Ren 4	11.0	65	40.7
11% oil and 0.3% Ren 7	10.1	65.5	38
11% oil and 0.15% RPA 3	11.4	63	37
11% oil and 0.3% RPA 3	11.6	60	34
11% oil and 0.45% RPA 3	11.4	54	27

In compounding easy processing rubber, two basic approaches are possible. Firstly, easy processing rubber can be treated as 100% rubber hydrocarbon, in which case, the presence of extra plasticiser would be expected to reduce tensile strength and modulus somewhat. The degree of reduction of these properties would depend on the compound and the extra free plasticiser involved. The easy process rubber of the Natural Rubber Producers' Research Association (1970) is compounded according to this approach. Secondly, the type and level of plasticiser can be maintained at the same level as the compound which is practical only if the plasticiser level is greater than 10 phr.

Indications are that easy processing rubber replacing premasticated NR plus free plasticiser (10 phr), because of reduced molecular breakdown during mixing, can be broadly equated to premasticated NR plus 5 phr free plasticiser. In our experiment we have equated 105 kg of easy process rubber to 100 kg masticated rubber and 5 kg of Ravolen X.

Banbury mixing of easy processing NR

The easy processing RSS was mixed on a factory scale in a No. 3 Banbury according to the formulation given in Table 2; the mixing schedule is listed in Table 3. Mixing times should be sufficiently long to ensure adequate dispersion of the ingredients. If it is prolonged much beyond this, the rubber may be broken down too much and tear strength and tensile strength will suffer.

TABLE 2
FORMULATIONS USED FOR TREAD MIX WITH EASY PROCESSING RUBBER

Ingredients	Normal mix*	Easy processing mix
Masticated RSS	100.0	—
Easy processing RSS	—	105.0
Ravolen X	5.0	—
HAF Black	45.0	45.0
Stearic acid	2.0	2.0
Sulphur	2.3	2.3
Zinc oxide	5.0	5.0
Vulcafor BSM	0.3	0.3
Anti-oxidant (Nonox D)	1.0	1.0

* In masticating RSS for the normal mix, Renacit 4 at 0.2 phr is used.

TABLE 3
MIXING SCHEDULE IN NO. 3 BANBURY

Time in min	Normal mixing schedule	Mixing schedule for easy processing RSS
0	Load masticated RSS	Load easy processing RSS
1	Add powders (stearic acid, zinc oxide, accelerator and anti-oxidant)	Add powders
1½	Add Black	Add Black
3	Dump	
3½		Dump

It was noticed that the black was incorporated better if the easy processing rubber had 0.3% Renacit 7 in it than when it was not used. The compound without the Renacit 7 had to be milled a few times to incorporate the black fully. The sulphur was added immediately after dumping and the camelback extruded. There was no difficulty encountered during the extrusion process and the product had extrusion properties apparently identical with those obtained from a similar compound prepared from premasticated rubber.

The results obtained on testing the camelback compounds are given in Table 4.

TABLE 4

PROPERTIES OF TREAD COMPOUND FROM EASY PROCESSING RSS (A IS WITH PEPTISER (RENACIT 7) AND B WITHOUT PEPTISER)

Time of cure at 143 °C in min	Properties									
	Tensile strength in kg/cm ²		300% modulus in kg/cm ²		Elongation at break %		Permanent set		Tear strength kg/cm ²	
	A	B	A	B	A	B	A	B	A	B
20	286	283	123	98	565	605	29	32	114	122
30	277	286	118	101	548	580	30	34	103	128
40	262	262	104	91	560	587	27	29	81	99
50	274	262	87	90	620	608	26	23	86	81
80	247	243	98	86	573	535	21	17	77	76

The Wallace plasticity and the Mooney viscosity (ML 1 + 4) of RSS prepared from clone PB 86 and from its latex mixed with Ravolen X and peptiser, and after storage for about 4 months are given in Table 5. It will be noted that there has been no increase in Mooney viscosity or Wallace plasticity.

TABLE 5

EFFECT ON WALLACE PLASTICITY AND MOONEY VISCOSITY ON TREATMENT OF NR WITH MINERAL OIL AND WITH PEPTISER

Date of manufacture	Date of testing	Ravolen X 11.5%		RSS with Ravolen X, (13.9%) and Renacit 7 (0.3%)		RSS with Ravolen X (10.2%) and Renacit 4 (0.3%)	
		Wallace plasticity	Mooney viscosity	Wallace plasticity	Mooney viscosity	Wallace plasticity	Mooney viscosity
8.6.72	22.6.72	41	65	35	65	37	68
	12.9.72	40	65	34	64	36	65
	31.10.72	40	65	32	62	36	65

Mill mixing of easy processing NR

Although mills are giving way to other more effective mixing equipment, the units in operation today are still scheduled to play key roles in the industry. The first prerequisite in mill mixing is the formation of a polymer band around the front roll and a polymer bank. One of the attractive characteristics of Natsyn 400 (synthetic cis-1, 4-polyisoprene) when compared with NR and other synthetic polymers is its rapid breakdown during milling and mixing (O'Mahoney, 1970). Qualitatively natural rubber is tougher and more nervy than polyisoprene especially at temperatures well below 100° C (Bristow *et al.*, 1969). The test for mill breakdown was done as given below and the amount of mill breakdown assessed by Mooney viscosity and Wallace plasticity determinations at various intervals of time. The milling was done on a 6" x 12" laboratory mill with friction ratio 1: 1.4 and roll speeds 22 rev/min front and 31 rev/min back. 500 g of rubber were used on the full width of the roller, at a roll temperature of 70 ± 5°C and at a gap of 1.25 mm (0.05 in.). The results obtained for different grades of easy processing NR are given in Table 6. The milling behaviour of easy processing rubber is good. Its lower initial viscosity enables it to form a smooth band faster than those with higher viscosities.

TABLE 6

MILLING BEHAVIOUR OF RUBBER FROM LATEX OF CLONE PB 86

Grade of rubber	Time to form coherent band	Mooney Viscosity (M) and Wallace Plasticity (W) after milling in min							
		1		2		3		4	
		M	W	M	W	M	W	M	W
RSS	2 min 20 sec	79.5	53	79	46	78	42	75	40
RSS + 11% oil	2 min 0 sec	63	35	62	33	61	31	54	29
RSS + 11% oil + 0.1% Ren. 4	1 min 40 sec	65	39	67	35	64	33	60	31
RSS + 11% oil + 0.2% Ren. 4	1 min 35 sec	65	37	61	33	58	31	50	26
RSS + 11% oil + 0.3% Ren. 4	1 min 50 sec	67	40	63	37	60	32	58	30
RSS + 11% oil + 0.15% RPA 3	1 min 20 sec	60	34	58	30	54.5	28	53	28
RSS + 11% oil + 0.3% RPA 3	50 sec	59	31	56	30	52	27	51	26
RSS + 11% oil + 0.45% RPA 3	40 sec	47	25	45	23	43	22	42	22

Easy processing RSS was made containing approximately 10% Ravolen X and 0.3% Renacit 4. The Mooney viscosity and Wallace plasticity were 57 and 31 respectively. This rubber was used on an open mill for making a retread compound at Don Somapala Corporation Ltd., Kelaniya, Sri Lanka and it was found that premastication could be eliminated, the load on the mill was considerably reduced and the hardness of the vulcanised retread equal to their normal product.

DISCUSSION AND CONCLUSIONS

Carbon black is not readily incorporated into unmasticated NR. The rubber has first to be softened by heat and mechanical working (masticated), and the addition of carbon black decreases the plastic flow of the rubber so that subsequent processing such as extrusion is affected. Normal extents of mastication of NR lead to about a ten fold decrease from the initial molecular weight of 10^6 (Bristow & Watson, 1963). The mastication step can be avoided if the plasticity of the NR is sufficiently low. Thin brown or thick brown crepes or SMR 20 which are made from field coagula are the preferred rubbers in tyre manufacture, as high grade rubbers require longer periods of mastication to reach a given viscosity. General purpose synthetic rubbers are supplied at Mooney viscosity levels of 50 ± 3 . For NR it is suggested that a Mooney viscosity of 60 ± 5 is suitable or a Wallace plasticity below 40 and preferably between 30 and 35 as it breaks down in mixing and compound viscosity will not be too low.

The clones recommended for large scale planting in Sri Lanka (Anon, 1968) with their hardness and the values of the Mooney viscosity after hydroxylamine treatment (Chin Peng Sung, 1969) are given in Table 7. Mooney viscosity depends on the average molecular weight of the polymer and the extent of plastication imparted by the non rubbers present in the rubber.

TABLE 7

PLANTING MATERIAL RECOMMENDED FOR LARGE SCALE PLANTING IN SRI LANKA

Clone	Hardness	Mooney viscosity after hydroxylamine treatment
RRIC 45	Medium	54
Wagga 6278	Hard	—
PB 86	Hard	76
RRIC 36	Hard	90
Glen 1	Soft	54

Since the clone PB 86 which is the most popular clone planted in Sri Lanka has a Mooney viscosity of 76 after hydroxylamine treatment, it may be difficult for Sri Lanka to produce CV rubber of hardness 55 to 65 especially because three of the five clones recommended for large scale planting give hard rubbers. The treatment with mineral oil (11%) and also with peptiser may enable Sri Lanka rubber to be comparable if not better to CV rubbers for use in the tyre industry. Peptisers function (Bristow & Watson, 1963) by being radical acceptors for mechanochemical scission in cold mastication or promoters of oxidative scission at higher temperatures.

Processing operations are those that come between the initial mastication of the rubber and vulcanisation, *i.e.* operations of mixing, calendering, frictioning, extrusion, moulding and combining with textile fabrics or cords. The basis of all these processes is the flow or viscous deformation of the rubber, or more precisely its rheological behaviour (Scott, 1962). Thus for instance there exists considerable rheological differences between natural rubber and synthetic cis-polyisoprene. Unmasticated RSS 1 and synthetic cis-polyisoprene have comparable Mooney viscosities when

measured as usual at 100-120 °C, but quite different values are found at lower temperatures, RSS 1 giving much higher values. Similarly differences are found in the temperature dependence of extrusion pressure under low shear conditions. Qualitatively natural rubber is tougher and more nervy than polyisoprene especially at temperatures well below 100 °C (Bristow *et al.*, 1969). Table 6 shows that the incorporation of peptiser, reduces the nerve of NR during mill mixing more rapidly than when it is not used.

The use of peptisers will not only assist in the mastication at high temperatures in the Banbury, but will also by breaking down the nerve assist in processing, especially at temperatures below 100 °C. RPA 3 causes a certain amount of molecular weight breakdown at smokehouse temperature (approx. 50 to 60 °C). However, the breakdown of the molecular weight of NR by RPA 3 at approx. 100 °C will be very considerable and RPA 3 cannot be used as a peptiser in new process rubber manufacture, but it has been and can be used in RSS manufacture.

The economics of manufacture of easy processing rubber is important. Since mineral oil is approximately half the price of NR, it should be possible to produce for export, easy processing NR with 0.2% RPA 3 at approximately the same price as RSS 1. A scheme to increase quality production of RSS in Sri Lanka (Nadarajah *et al.*, 1972) envisages the setting up by the Government of the necessary Group Processing Centres (GPCs) to manufacture RSS. The manufacture of easy processing RSS at these GPCs and sending it to a Central Factory for conversion to block rubber is worthy of investigation as a development project to investigate whether its production in large volume supply and at an economic price is possible in Sri Lanka. "Easy Processing Block Rubber" is a new rubber that has to be developed, technologically investigated and its advantages advertised to the consumer. After the consumer (mainly tyre manufacturers) has evaluated it, we hope that he would be prepared to share the economic advantages with the producer. Sri Lanka is a producer of high quality rubber, *e.g.* RSS 1 and since there would be in the future only a limited demand for such a grade of rubber, it is necessary that Sri Lanka produces a tyre rubber for which tyre manufacturers would be prepared to pay a fair price because it is tailor made to suit their requirements.

Oil extension of easy processing rubber

Nadarajah (1969) has discussed the use of oil extended natural rubber (OENR) in passenger car retreads in Sri Lanka. In his work, the practical suggestion was to use the Banbury to mix the oil. However, there are in Sri Lanka several retreaders who use open mills for compounding, and it is not possible to add large amounts of mineral oil on the open mill. Work done by us at Don Somapala Corporation Ltd., Kelaniya, Sri Lanka has shown that it is possible to add easily mineral oil to easy processing rubber in the compounding stage. Whilst normal mixing with premasticated rubber takes 45 minutes, mixing of easy processing NR even after adding 10 phr of Ravolen X for oil extension took only 25 minutes.

The use of easy processing RSS is an important step in the manufacture of oil extended natural rubber compounds on open mills. However, it will be necessary to add 4 phr more carbon black for every 10 phr of mineral oil used, to get the same hardness. The easy processing rubber to be used on open mills may be made without the peptiser. Because of its high resilience, NR if used in new treads or in retreads of passenger car tyres must be oil extended if it were to be comparable in skid resistance to OESBR. Due to the winding nature of Sri Lanka's roads and the prevalence of wet conditions during a good part of the year, every aspect of safety for the motorist must be considered and in this respect tyres with a minimum tendency to skid are important. It is hoped that the Bureau of Ceylon Standards,



Sri Lanka will consider the importance of oil extension when deciding on specifications of compounds based on NR for use in passenger car treads in Sri Lanka.

ACKNOWLEDGEMENT

We thank Dr. O. S. Peries, Director, RRIC. for encouragement and the Board of the R.R.I.C. for permission to present this paper at the Sixth Technical Seminar of the Indian Rubber Manufacturers' Research Association.

Our thanks are due to Messrs B. Kuznetsov and K. C. Rasaratnam of the Ceylon Tyre Corporation for helpful discussions and to Messrs. Buddy Amath and K. D. C. Marcus also of the Ceylon Tyre Corporation for testing of camelback compounds (Table 4). We are much obliged to Mr. Premasiri Perera of Messrs Associated Mortorways Ltd., Kalutara for assistance in carrying out experiments in their No. 3 Banbury, and to Mr. M. Ariyaratne of Don Somapala Corporation Ltd., Kelaniya for assistance in the experiments done on the open mill. We thank Mr. Douglas de Fonseka, Superintendent, Pahan Estate, Matugama and Mr. B. H. de Alwis, Superintendent, Miriswatte Estate, Welipenna for assistance in manufacturing the easy processing RSS. We also thank Mr. W. S. E. Fernando, Assistant Rubber Chemist, R.R.I.C., for assistance in some of the experiments, and Hayleys (Ceylon) Ltd., for samples for Renacit 4 and Renacit 7.

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