DEVELOPMENT OF ENERGY SAVING FORMS OF NATURAL RUBBER

A THESIS SUBMITTED BY

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CERTIFICATE

This is to certify that this thesis is a report of the original work carried out by Mr.E.V. Thomas, under my supervision and guidance at the Rubber Research Institute of India, Kottayam 686 009. No part of the work reported in this thesis has been presented for any other degree from any other University.

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22nd April 1985.

DECLARATION

I hereby declare that the work presented in this thesis is based on the original work done by me under the supervision of Dr. D.Joseph Francis, Professor and Head of the Department of Polymer Science and Rubber Technology, University of Cochin, Cochin 682 022, at the Rubber Research Institute of India. No part of this thesis has been presented for any other degree from any other institution.

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The work was done at the Rubber Research Institute of India (R.R.I.I), Kottayam 686 009. The permission given by the Director of Research, Dr. M.R. Sethuraj and the former Director, Sri.V.K. Bhaskaran Nair, for executing this work at the Rubber Research Institute of India and presenting the data for my doctorate degree is also acknowledged gratefully. Evaluation of some part of the work like, preparation of tyres and its endurance tests was done out side the Rubber Research Institute of India. Assistance was received from Sri.Zacharia George and Sri. George John of M.R.F., Madras and Sri. P.K. Mohamed of Apollo Tyres, Chalakudy, Kerala. The assistance given by these technologists and the permission given by the respective managements are also gratefully acknowledged.

contd...

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22nd April 1985.   E.V. THOMAS

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CHAPTER - I

INTRODUCTION

CHAPTER - I

INTRODUCTION

Natural rubber is produced in certain species of plants by bio-synthesis, in the form of an aqueous milky white dispersion called latex. Rubber containing latex is present in more than 2000 species[1]. The rubber content of the latices of only a few of these plants, is high enough for commercial exploitation. Among these, Hevea brasiliensis is the most important source of natural rubber today. Mention may also be made about Parthenium argentatum, Castilla elastica, Manihot glaziovii, Ficus elastica, Cryptostegia grandiflora and Funtumia elastica as other rubber yielding plants. Studies conducted in this thesis are on rubber derived from the latex of Hevea brasiliensis, as this produces 99 per cent of the world's natural rubber. This rubber plant is a sturdy tree which attains a height of 30 m when grown full. Latex is present in almost all parts of the plant, but only the bark of the trunk is exploited.

Latex is extracted from the trees by inflicting controlled wounds on the bark through a slopping cut. This process is known as tapping and is done daily or once in two days. Latex flows through the tapping panel and is chanelled into collection cups. This is collected from the field 3 - 4 hours after tapping. The bulk of the crop (75 - 85%) from rubber plantation is in the form of latex. The remaining quantity is collected in coagulated form, from tapping panel (lace scrap), collection cups (cup scrap) and the foot of the tree (earth scrap) These coagulated forms are together called field coagula rubbers.

Rubber latex can be processed into ribbed smoked sheets (RSS), pale latex crepe (PLC), solid block rubber, speciality rubbers, or preserved concentrated latex. Field coagula rubbers can be processed either as estate brown crepe or as solid block rubbers. Rubber goods manufacturing industry uses these types of rubbers in the production of different kinds of rubber products. Consumption of these processed rubbers in India for the past four years is given in Table - I below(2).

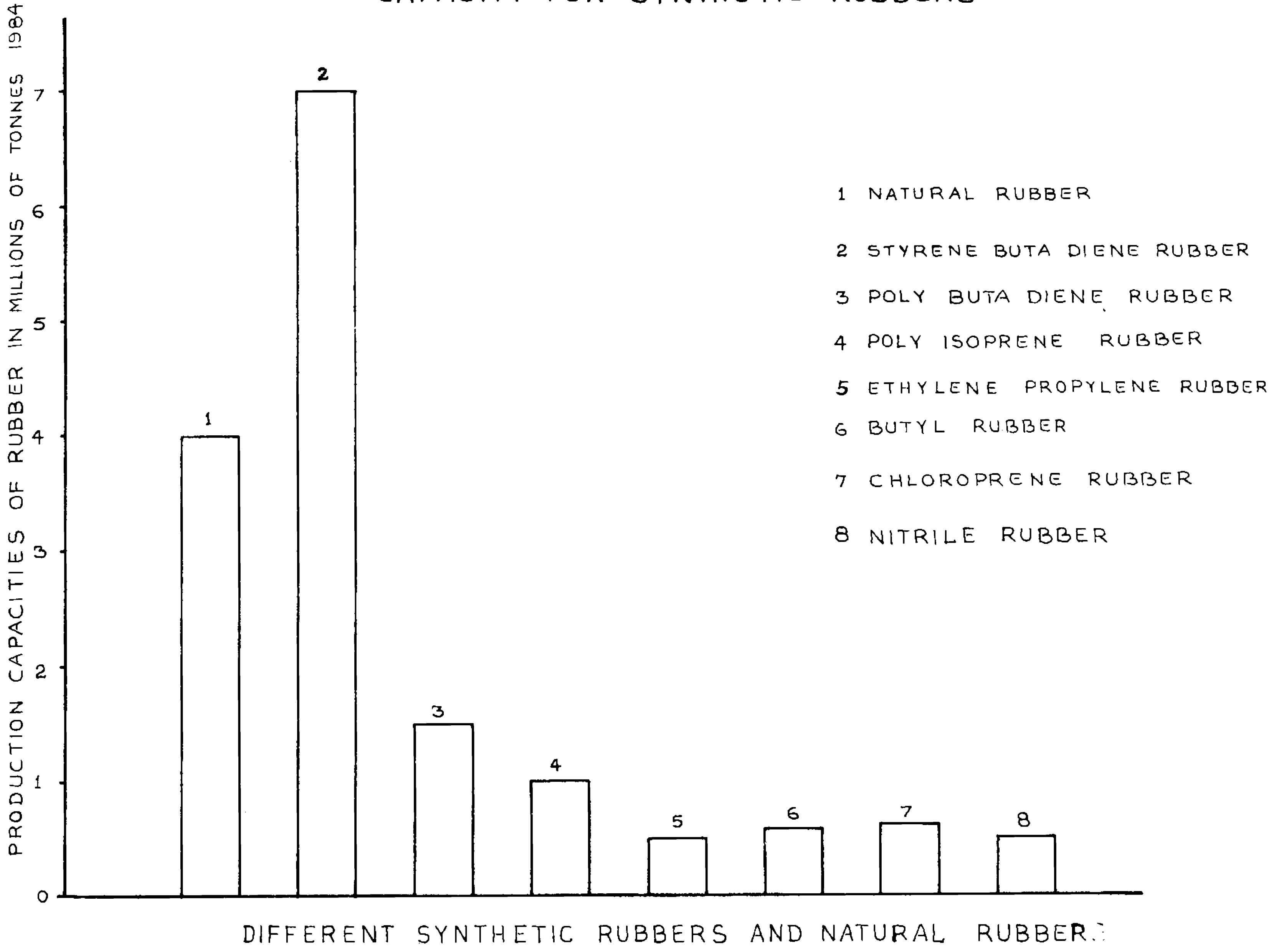
TABLE - I

	Year			
	1979-80	1980-81	1981-82	1982-83
	(in tonnes)			
Ribbed smoked sheets	1,11,910	118,425	128,530	131,935
Pale latex crepe	1,980	1,775	1,415	1,950
Estate brown crepes	28,395	31,940	32,425	27,950
Technically specified rubber	11,270	7,670	11,120	16,840
Concentrated latex	11,690	13,820	14,930	16,850

The first four categories of rubbers listed in Table - I are dry forms of processed rubbers. They are used in the production of articles like automobile tyres and tubes, cycle tyres, retreading materials, foot-wear etc. For production of these articles rubber has to be broken down in heavy duty mills or internal mixers and then compounding ingredients are incorporated into it. This step consumes appreciable amount of energy. Concentrated latex is used in the production of foam rubber, gloves, balloons, toys and some forms of adhesives. Compounding of latex for the manufacture of products is an operation involving lower energy consumption, when compared to that for the dry forms of rubber. But products from latex account for only ten percent or less in volume of the total rubber products manufactured. Thus for the conversion of a lion's share of the processed rubber to useful products, appreciable amount of energy is needed at the compounding stage.

Rubber product manufacturers use both natural and synthetic rubbers in product manufacture. These two are the main raw materials for rubber industry. World consumption of all rubbers today is 12.2 million tonnes. Natural rubber accounts for only a third of this. Synthetic rubbers have certain processing advantages over natural rubber. So the general purpose synthetic rubbers like styrene butadiene and poly butadiene have replaced natural rubber in many areas of application. Fig.I.1 shows the pattern of production of different types of rubber in the world[3].

FIG. 1.1 WORLD PRODUCTION OF NATURAL AND INSTALLED CAPACITY FOR SYNTHETIC RUBBERS



Developments in Natural Rubber Processing:

Natural rubber was earlier designated as crude rubber by the rubber manufacturers. This designation was given, since it had a high degree of variability. The objective of all the modern processing methods was to give natural rubber an appearance and processing properties comparable to synthetic rubbers. The first attempt was to improve the grading of natural rubber and to give a technological basis for this. Ribbed smoked sheets or Estate brown crepes are the conventional forms of processed natural rubber, These are graded by visual comparison methods. An attempt was made in the fifties to grade rubber based on cure properties(4). The red circle (fast curing) yellow circle (medium curing) and blue circle (slow curing) rubbers were thus marketed in small quantities. This grading system did not gain acceptance among consumers and so a modified system based on the purity and properties of rubbers was evolved. This is known as the technical specification scheme(5), It became clear gradually that a mere change in the system of grading alone is not sufficient in providing a better presentation for natural rubber. The new system of processing of natural rubber in solid block form is a direct consequence of this.

A number of processes were tried for arriving at an acceptable process flow line for the production of solid block rubber. Hevea crumb process(6,7,8) developed by the

Rubber Research Institute of Malaysia suggested the use of castor oil along with latex at the stage of coagulation. The coagulum so obtained is macerated successively in between rollers kept at progressively diminishing nip settings. The coagulum is converted to crumbs, which are washed, dried and pressed in bale forms. Later mechanical systems were used for block rubber production(9,10,11). The rubber coagulum is macerated initially in crepers and then passed through extruders provided with a cutting knife at the tip of the extrusion die. In a later modification instead of this extrusion cutter, a hammer mill is used for size reduction of the coagula. After size reduction the operations of drying and baling are almost similar in all processes.

The new forms of rubbers can be converted to products using less energy than what is required when conventional forms of rubbers are used. Natural rubber processed in conventional sheet or crepe forms are transported in trucks or rails as separate bareback bundles. The time for unloading such loose bundles is 7 to 10 times that required for unloading the same quantity of synthetic rubbers arriving in pallets in similar carriages(12). Bareback bales pick up dirt during shipping and transport and so additional work is necessary for cleaning bale surfaces. The large bundles are to be cut into smaller sizes before being fed to banburry or other mixers.

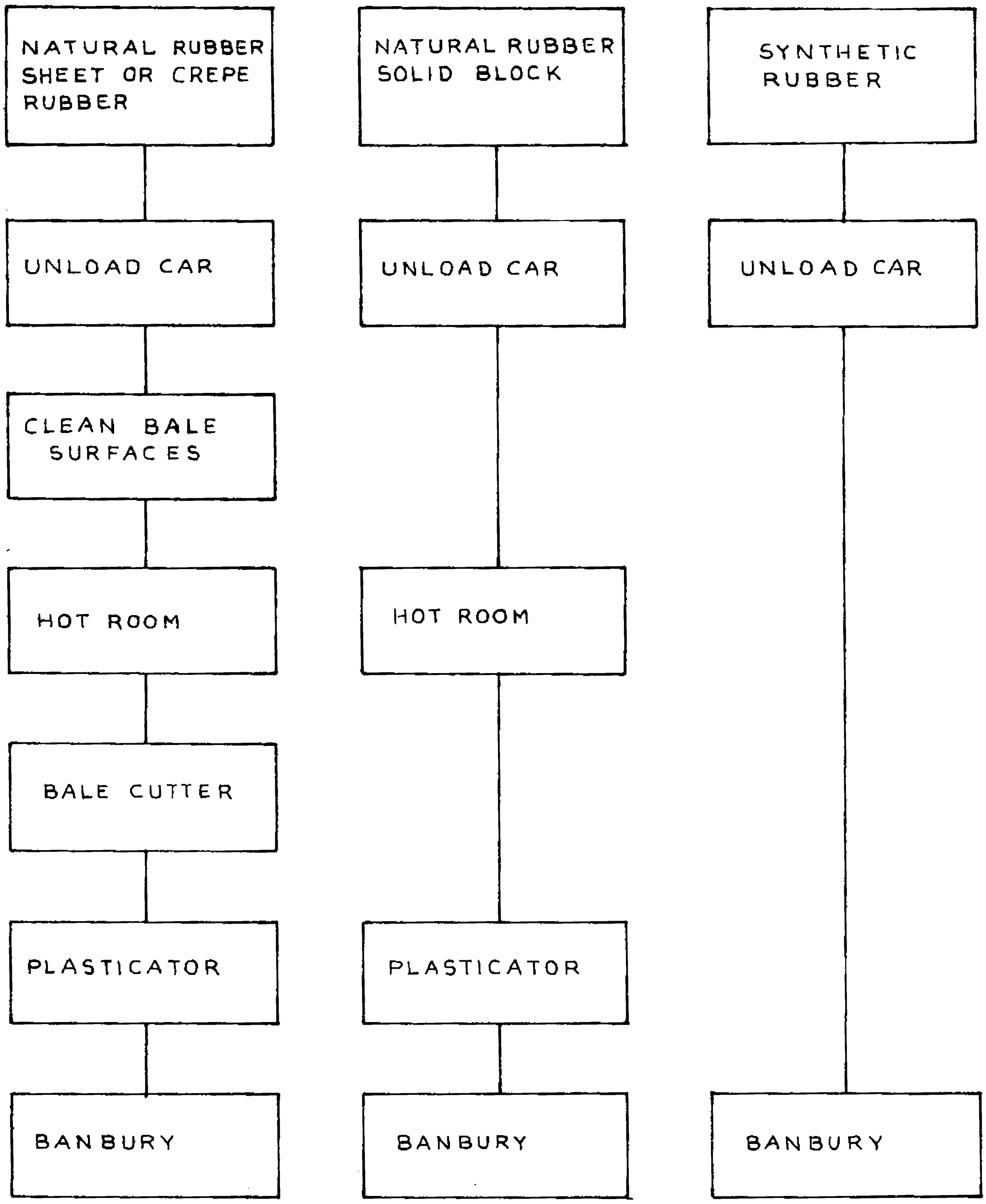
The advantages of modern forms of solid block rubber over conventional sheet or crepe rubbers in a consumers' factory can be seen from (Chart I.1) Synthetic rubbers are also included in the chart for comparison.

Thus modern forms of processed rubbers are helpful in saving labour in the consumers' factory. In countries like India hot room treatment is not necessary for natural rubber, irrespective of the form in which it is processed, but pre-mastication in plasticators is necessary.

The production of viscosity stabilised form of block rubber marks the next stage of development(13,14). This rubber is produced by incorporating hydroxylamine hydrochloride with latex at the rate of 0.15 percent on weight of rubber content. The latex is then coagulated and processed as solid block rubber. This rubber has low and constant viscosity. If proper care is exercised at the stage of processing, this rubber can be supplied to consumers as a grade of natural polyisoprene, comparable in appearance and processing properties to general purpose synthetic rubbers.

Another development in NR processing was the production of oil extended natural rubber(15,16,17) (OENR). This rubber can be used as substitute for oil extended SBR (OESBR). They are prepared by incorporating oil with rubber at the processing stage. They can also be used by the consumers without pre-mastication. Other important types of modified natural rubber

CHART I.1 COMPARISON OF STEPS INVOLVED IN CONSUMERS' FACTORY FOR USING DIFFERENT FORMS OF RUBBER



which have processing advantages are oil carbon black master batches(18,19,20), Latex stage rubber compounds(21,22), blends of natural rubber and polybutadiene[23], tyre rubber(24,25,26) and superior processing rubbers(27,28,29).

Review of work on Energy Consumption in Polymer Industries:

In rubber product manufacture direct plant energy is involved in following steps: (a) Mastication and mixing, (b) Shaping and (c) Curing. Appreciable amount of energy is required in the first step as the rubber has to be softened and plasticised in heavy machinery for incorporating various compounding ingredients. When compared to general purpose synthetic rubbers, natural rubber consumes more energy at the stage of mastication and mixing. Synthetic rubbers are available in several tailor-made forms, which provide processing advantages to consuming industries. Attempts were made from early days for improving the processing of natural rubber. The necessity for such an improvement was not seriously felt by the NR producing countries until synthetic rubber production started in large scale in different consuming countries.

The need for conserving energy and managing it is very much felt now mainly on account of the enhanced cost of mineral fuels. In countries like USA all major industries have evolved policies for making their production, energy-efficient. Thus at the year end 1979 Du Pont Company in USA was able to reduce their energy consumption per unit of production by 25% below the level established in 1972 by adopting energy saving methods. On an yearly basis this works out to 14.9 million barrels of oil(30). Need for evolving energy efficient processes by individual industries is amply clear from this. Jack Entwistel(31) has compared the energy involved in production of synthetic fibres with that of natural rubber. The subject "product energy" was brought to the attention of rubber industry by Frank Burton(32). He has defined product energy as the energy required to make a unit quantity of the product. Product energy of a typical rubber compound as worked out by Burton is given in Table-II.

TABLE - II

	BTU/lb	Percentage of total
Raw material energy	50,000	65
Direct plant energy	6,000	8
Indirect plant energy	16,000	21
Scrap/defect energy	5,000	6
	77,000	100

For calculation of the raw material energy, the energy content of polymers and compounding ingredients are needed. Values on most of these are available from various published data. J.D.Byam et al(33) reported the extent of energy saving possible in elastomers using internal mixers. They have also compared the energy advantages of thermoplastic rubbers in product manufacture. H.D.Harris and Ben

Harrison(34) have described a system for product line energy determination for rubber products. It is reported that the manufacturing area accounts for 76.4 percent of the total energy consumed in any product. The energy involved for manufacturing is worked out as 9092 BTU per pound (76.4 per cent of 11900 BTU).

The components of product energy(32) are listed in Table-III.

TABLE - III

PRODUCT ENERGY

1. Raw material energy	- The energy content of raw materials including the energy to transfer to the plant,where they are to be used.
2. Direct plant energy	- The energy put into mixing and processing of product in the plant.
3. Indirect plant energy	- Energy used in the plant, including offices and other facilities, not directly related to specific manufacturing operations.
4. Scrap and defect energy	- The energy lost when product is scrapped at any stage of production.
5. Total product energy	- The entire energy content of a product, that is the sum of items 1 to 4.

Although very little work has been done in India on energy saving in rubber goods manufacturing industry, there are reports on achievements in this field by heavy industries like steel. The present work is envisaged in this context to suggest energy economising methods in some of the operations in rubber goods manufacturing industry.

Scope of the present work:

Although the physical and technological properties of modified rubbers were compared with those of conventional rubbers in the earlier studies reviewed above, the extent of energy saving possible by their use has not been quantified. In the present work it is proposed to determine the energy saving possible by using the following forms of natural rubber: 1. Oil extended natural rubber, 2. peptised natural rubber, 3. latex stage compounds. The process of production and quality control of the above types of processed rubbers are proposed to be standardised. It is also planned to work out a methodology for utilising field coagula for production of constant viscosity rubbers. The variation in processing properties of rubber from popular Indian clones will be examined and those with energy advantages in the processing stage will be identified. In conclusion a recommendation for adoption of a commercially advantageous processing procedure among Indian rubber plantation industry will be given.

The thesis is divided into the following chapters:

Chapter I - Introduction

Chapter II - Materials and methods

Chapter III - Oil extended natural rubber from latex (OENR-L)

Chapter IV - Processing of Natural Rubber latex with incorporation of peptisers

Chapter V - Investigation of clonal variations in processing properties of natural rubber and optimum use of field coagula in blending with latex coagula.

Chapter VI - Latex Stage Compounding

Chapter VII - Summary and Conclusion.

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CHAPTER - II

MATERIALS AND METHODS

CHAPTER - II

MATERIALS AND METHODS

The different materials and the test procedures used in the experimental work described in Chapters III to IV are given here. Wherever a standard test method like ASTM or ISI is available, only reference to the method is given. The most important material used in the study is natural rubber. The rubber chemicals and other compounding ingredients used were procured from reputed suppliers. The chemicals procured were examined for their conformity to national standards, wherever such standards were available.

Materials

Natural Rubber: The rubber latex and field coagulam used in the study were collected from the two experiment stations of the Rubber Research Institute of India at Kottayam and Ranni. For the study reported in Chapter V, it was necessary to collect latex from twelve different clones. So, for this project latex was collected from a private estate, M/s Malankara Rubber Producing Company, Thodupuzha, Kerala State. A clone is a group of plants multiplied by vegetative propagation from a mother plant. The twelve clones whose latices were collected for the study are:

RRII - 105: This is a high yielding plant developed by the Rubber Research Institute of India.

RRIM - 600: This also is a high yielder developed by the Rubber Research Institute of Malaysia.

GT - 1 : This was developed in Indonesia. It is also recognised as a high yielding clone.

Gl - 1 : This is a moderately good yielder. Known for high magnesium content in its latices. This was developed in Malaysia.

PR - 107 : This is a moderately high yielding clone, developed in Indonesia. Its latex has a tendency to blacken, because of high polyphenol oxidase activity.

RRIM - 623: Developed by the Rubber Research Institute of Malaysia. Moderately good yielder.

RRIM - 628: This also was developed by the Rubber Research Institute of Malaysia.

PB - 86 : This clone was developed by the Prang Besar group of estates in Malaysia. This is found to be a high yielder in some regions. It is also known for the whiteness of its latex.

RRII - 118: This clone was developed by the Rubber Research Institute of India.

Tjir - 1 : This is an old Indonesian clone

PB5 - 139 : This also was developed by the Prang Besar group in Malaysia.

RRIM - 605: This clone was developed by the Rubber Research Institute of Malaysia. It is known to yield rubber of comparatively low molecular weight.

Field coagula consist of lace scrap, cup scrap and earth scrap. In the present study only the first two are used. This also was collected from the experiment stations of RRII.

Rubber Chemicals.

Accelerators:

CBS : N-cyclohexyl 2-benzothiazyl sulphenamide. This was produred from M/s Alkali & Chemical Corporation of India Limited.

MOR : N-oxy diethylene benz thiazol 2-Sulphenamide. This was procured from M/s Poly oleifins India Ltd. (Formerly Mindia Chemicals).

MBT : 2-Mercapto benzothiazole. This chemical was procured from M/s Alkali & Chemicals Corporation of India Ltd. The product conformed to specifications given in IS 6918 (1972).

Antioxidant & Antiozonants:

PBNA : N-phenyl-β-napthyl amine. This was supplied by M/s Alkali and Chemical Corporation of India Ltd.

SP : Styrenated phenol. This is a non-staining antioxidant supplied by M/s Alkali and Chemical Corporation India Ltd. It conformed to specifications given in I.S.7351 (1974).

Flectol-H Polymerised 1,2 Dihydro 2 2 4 - Trimethyl Quinoline

This is a powerful non-blooming antioxidant used in rubber industry. This was supplied by M/s Poly Olefins India Ltd.

Santoflex-13. N(1-3 Dimethyl butyl) N'-phenyl p-phenylene diamine

This is a good antiozonant and it provides antiflex cracking and antiozonant properties to rubber. This was also supplied by M/s Poly Olefins India Ltd.

Other Chemicals:

Zinc oxide used in the study was of commercial grade. It conformed to specifications I.S. 3399 (1973)

Stearic acid used in the study was of commercial grade and it conformed to specification given in I.S. 1675 (1960)

Sulphur used in the study conformed to specifications given in I.S.8851 (1978).

Peptisers & Plasticisers:

Renacit-7: This is penta chloro thio phenol with activating and dispersing additives. It was supplied by M/s Bayer India Limited.

RPA-3 (Xylyl mercaptan): It is used as a bleaching agent in rubber processing industry. It is manufactured by M/s E.I. Du Pont De Numours, USA.

Rubber Process Oil:

Aromatic rubber process oil was used in all the studies reported here. This was procured from M/s Hindustan Petroleum Corporation. It conformed to following specifications.

Specific gravity	-	0.99
Saybolt viscosity 100°F	-	6621

Viscosity Gravity Constant - 0.907
Aniline point - 25°C
Flash point - 245°C

Fillers: HAF - Black used in the study was manufactured by M/s Philips Canbonr India Ltd. It conformed to specificcation given in IS 7497 (1974).

China Clay: The inorganic filler china clay used was commercial grade. This was used in the studies reported in Chapter VI.

Other Chemicals - **Hydroxyl amine hydro chloride & ethylene glycol:** used in the studies are standard laboratory chemicals. The former was procured from M/s Sarabhai M. Chemicals, Baroda and the latter from M/s Glaxo Laboratories India Ltd. (Chemical Division) Bombay.

Test Methods & Procedures

Preparation of Solid Block Rubber:

For the investigations mentioned in Chapters III, IV & V rubber in solid block form was used. The production of solid block rubber involves four steps, viz. coagulation of latex, size reduction of the coagulam, drying, leaching, grading and packing. Rubber latex is coagulated with acetic or formic acids. The coagulam is machined through macerators, and crepers and crumbled using hammer mills. The crumbs are collected in a pool of water washed and transferred to perforated Aluminium trays. These are then moved through tunnel driers, where a current of hot air is circulated.

Drying temperature is around 100°C. Usually drying will be over in about 4 hours. The dry crumbs are then pressed in hydraulic press to obtain solid blocks of standard weight and dimensions. The grading of the rubber is done as per technical specifications given in I.S. 4588 after analysis.

Preparation of constant-viscosity rubber:

Natural rubber undergoes hardening during keeping by internal condensation. This is believed to be due to the presence of some aldehydic groups in the polyisoprene chain[1]. The tendency for hardening can be arrested by deactivating the aldehydic groups using hydroxylamine hydrochloride(2). The rubber prepared this way is called constant viscosity rubber. For its preparation fresh latex should be treated with hydroxylamine hydrochloride at the rate of 0.15 percent on its rubber content. Subsequently the latex can be processed as solid block rubber as described earlier. This type of rubber will have low and constant Mooney Viscosity.

Crepe Rubbers:

Both latex and field coagula can be processed in the form of crepe rubber. When the starting material is latex, it has to be first coagulated. Subsequent operations involve milling in heavy duty mills and simultaneous washing in water to remove all foreign matter. The number of passes through macerators and crepers will be larger when field coagulam is used as it may contain more foreign matters. The milled crepes will resemble blankets. They are dried in air at temperature below 40°C. When latex is used as the starting material, the crepes are called latex crepes. The crepes obtained from field coagula are called Estate Brown crepes (EBC).

Raw Rubber Properties:

The raw rubber properties investigated in the studies and the procedures used are given below:

Properties	Procedures
Dirt content*	I.S.3600 Part I (1966)
Ash content	-do-
Volatile matter	-do-
Nitrogen content	-do-
Po	I.S.3660 Part III (1971)
PRI	-do-
Accelerated storage hardening test (ASHT)	I.S.3600 Part IV (1979)
Mooney Viscosity	ASTM - D - 1646 (1981)

For dirt content estimation a slight modication was introduced in the IS procedure based on development work done at the Rubber Research Institute of India. In the IS procedure the peptiser recommended for dissolution of rubber in mineral turpentine is xylyl mercaptan. Due to the non-availability of this chemical alternative peptisers were tried and copper oleate was found to be a most efficient substitute. This modiciation was approved by ISI in 1984.

Processing Properties.

Mixing in Brabender Plasticorder: Brabender Plastograph manufactured by M/s Brabender OHG Duisburg, West Germany, is a device for measuring torque during mixing. The measured torque is a function of viscosity changes taking place in a mixing chamber fitted to the instrument and the torque is plotted continuously against time. For studies on mastication behaviour, the following test conditions were selected.

Mixer capacity	-	75 ml
Weight of rubber	-	70 gm
Rotor speed	-	60 rpm
Chart speed	-	3 cm/minute
Temp. of mixing chamber	-	75°C
Time of mastication	-	10 minutes

The rubber samples were passed through the Laboratory Mixing Mill twice at 5 mm nip setting before feeding into the plasticorder. The graph obtained for the mastication of different samples were evaluated after combining them into one graph.

Compounding: Compounding was carried out as per procedures in ASTM-D-3184-80 and 3182-82. Mixes were prepared in David Bridge Laboratory mixing mill for small scale evaluations. For large scale mixing a Sohal Commercial Mill of 14" x 30" size installed at the Institute was used.

Energy of Mixing / Compounding: The two roll rubber mixing mill used was of 14" x 30" size. The drive system of the mill was connected to an electric energy meter. Initially the mill rolls were warmed to the mixing temperature of 65 ± 5°C. The reading in the energy meter was taken at the start of the mixing operations. The mill was allowed to work continuously till the mixing operations were completed. Reading was taken when the rubber was completely converted to a smooth band and also when the compounding operation was completed with full incorporation of all the ingredients From this the energy required for band formation and that for complete compounding can be calculated. Since the weight of rubber used for compounding is known, the energy in KWH for one kilogram rubber compounding can be calculated.

Cure Properties: Scorch time and cure time were evaluated in different studies reported here. For scorch time studies Mooney Viscometer was used. The procedure adopted was ASTM-D 1646-81. For cure time determination Monsanto Rheometer model R-100 was used. The temperature of the test was 150°C. Other test conditions were as follows:

(i) Arc - 3°
(ii) Chart speed 60 minutes
(iii) Range 0 - 100

Moulding: The test slabs for assessing physical properties were prepared in standard moulds by compression moulding in an 18" x 18" steam heated hydraulic press. The curing time in each case was selected on the basis of rheometer studies. The vulcanisation procedure used was as per ASTM-D-3182 (1982)

Physical Properties: Modulus, Tensile Strength and Elongation at break. These tests were carried out according to ASTM-D-412-80, using dumb-bell specimens. All the tests were carried out at 27 ± 2°C. A Scott tensile tester model L-6 was used in the studies.

Tear Resistance: The test was carried out as per method given in ASTM-D-624-81. This property also was measured in the Scott tensile tester model L-6 using crescent test specimens.

Hardness: Shore-A type Durometer was used to find out the hardness of vulcanisates. The method employed was ASTM-D-2240-81.

Rebound Resilience: Scott rebound pendulam was used in resilience studies. The procedure used was ASTM-D-1054 (1979). Dunlop tripsometer was also used for assessing this property.

Abrasion resistance: Du Pont Croydon Abrasion Tester was used in the present studies. The procedure consists in abrading two weighed specimens against standard abrasive paper under fixed speed and load for a definite duration. The weight loss and volume loss for unit time was calculated and expressed as a measure of the abrasion resistance of the material. The procedure broadly corresponds to IS-3400, Part - III (1965).

Compression Set: ASTM-D-395-82 method B was used for compression set studies.

Flex Cracking - Flex cracking was determined using a Wallace Flexometer. The procedure corresponds to ASTM-D-430-73.

Heat Build up: Goodrich flexo-meter was used for heat build up studies. The procedure used was ASTM-D-623-78. Tests were carried out with cyclendrical sample of 1 inch in height and 0.7 inch in diameter.

Molecular Weight Determination.

Molecular weight determination reported in ChapterIV was carried out by the viscosity average method. The details of the procedure employed was as per BS 1673 Part VI 1969. Toluene was used as the solvent.

Ageing Studies: Ageing studies were carried out in accordance with B.S. 903, Part A 19' 1956, Method 'A', Cell type oven method. Dumbell specimen for tensile strength studies were prepared and aged in the oven at pre-determined temperature for specified periods. The percentage retention of physical properties was assessed.

Ozone Resistance Tests: The ozone resistance of vulcanised samples were assessed as per ASTM-D 1171 - 74 using a Mast Ozone Chamber model 700-1. Here ozonised air is generated with the help of an Ultraviolet lamp. Triangular test piece of specified dimensions were prepared and formed them into a loop for keeping the samples under strain. After mounting, the samples were allowed to rest for 72 hours, at room temperature in an ozone - free atmosphere. The samples were then introduced into the ozone chamber at an ozone concentration of 50 pphm (parts per hundred million). The temperature of the test was 40 ± 1°C. The samples were exposed for 72 hours and visual assessment of crack intensity was made at 24 hours interval.

For estimating the ozone concentration a known volume of air is passed with a 4 per cent solution of potassium iodide buffered to pH 7. The liberated iodine is titrated against 0.002 N sodium thiosulphate solution and ozone concentration calculated.

CHAPTER - III

OIL EXTENDED NATURAL RUBBER FROM LATEX (OENR-L)

CHAPTER - III

OIL EXTENDED NATURAL RUBBER FROM LATEX (OENR-L)

Oil extended styrene-butadiene-rubber (OESBR) is used extensively in the production of passenger car treads. It showed better road grip on icy roads. So in winter car tyres this material was used in preference to natural rubber. Till 1972, oil was much cheaper than rubber and so use of oil extended rubber in preference to ordinary rubber was helpful to consumers in producing passenger treads at cheaper compound cost. Making use of this low price of oils the synthetic rubber manufacturers produced large quantities of oil extended SBR and popularised among consuming industries with the result that by 1960 natural rubber was almost completely replaced by many manufacturers from compounds used in passenger car tread production.

Oil extension of natural rubber was started in this background to increase the competitive position of natural rubber with oil extended general purpose synthetic rubbers, particularly SBR. In earlier studies, oil extension of natural rubber was done only in dry stage, in mixing mills[1] or banbury. Grosch *et al*[2] had evaluated the properties of OENR-treated compounds with those of OESBR treads. The Malaysian Rubber Producers' Research Association evolved suitable formulations and processes[3,4] for the production of passenger car treads using oil extended natural rubber.

Producing countries began research work on oil extended natural rubber from latex (OENR-L)[5,6,7]. Oil was added as an emulsion to latex followed by coagulation, size reduction and drying. One of the difficulties faced by earlier workers in producing OENR-L was the absence of satisfactory method for emulsigying oils. Excessive cold flow of the processed OENR-L was another problem. A procedure was described by Cheong Saifah and co-workers[8] for preparing stable oil emulsions. Chin peng Sung[5] has described a method for reducing cold flow properties of OENR-L by using hydrazine hydrate. Chin peng Sung and Oconnel[9] also reported that undiluted field latex gave better processing properties for OENR-L. Physical properties of OENR-L from three mineral oils, viz. aromatic, naphthenic and paraffinic, were studied. Use of OENR-L in retreads of passenger car tyres was reported in the work of Karunarathna[10]. Thomas[12] and co-workers reported that the use of OENR-L reduced the compounding cost. The study also revealed that for compounding OENR-L, peptisers were not necessary and the time for compounding it in banbury was reduced by 25 percent when compared to that required for conventionally processed natural rubber. In the present study, the following aspects are evaluated.

1. The properties of OENR-L prepared with and without incorporation of hardners.
2. The variation in properties of OENR-L with variation in oil content.
3. The physical properties of OENR-L and energy consumption at compounding stage in comparison to those of conventionally processed rubbers.

4. Performance of passenger car tyres produced using OENR-L in comparison to those from OESBR tyres.

5. The storage hardening properties of OENR-L with varying oil content.

Experimental

Preparation of OENR-L:

Oil emulsion was prepared by adding stearic acid to aromatic oil at the rate of 4 parts per hundred parts of oil. The oil was warmed just enough to ensure complete dissolution of stearic acid in oil. Oil containing stearic acid was then added to an equal volume of water containing around 0.5 percent ammonia (W/V). The method suggested by Cheong Saifah[7,8] was not used here as the above procedure standardised at the Institute in earlier studies[11] gave good emulsion and uniform blends with NR latex.

Field latex was seived to remove dirt and foreign matter and stored in a blending tank. Ammonia was added to latex to the extent of 0.1 per cent of its volume. The mix was gently stirred to ensure uniform mixing. 2 percent formic acid was then added at the rate of 300 ml per kg. rubber content in latex oil blend. Coagulation was completed by keeping the mix overnight and the coagulum was milled, washed crumbled and dried.

The dried OENR-L was processed in solid block form at the Pilot Crumb Rubber Factory of RRII. OENR-L was prepared with and without incorporation of hardeners. The hardner used was benzidine at the level of 0.15 percent on drc of latex. (Oil extended rubber prepared from dry rubber by mastication and mixing in mills or internal mixers is designated OENR).

Analysis and Testing of OENR-L:

The OENR-L prepared was first examined for acetone extract, accelerated storage hardening (ASHT) and all raw rubber properties described in IS 4588. Acetone extract was determined as per ASTM-D 297-76. Accelerated storage hardening , and raw rubber properties as in IS 4588 were also determined.

OENR-L was compounded in the following formulations, suitable for passenger car tyre tread production. In these formulations, OENR-L was considered as a composite of 100 parts rubber and 18 parts oil. The curatives were added for 100 parts polymer.

Ingredients		I	II	III
OENR-L (Benzidine)	..	118	..	..
OENR-L	..	..	118	..
Natural Rubber	..	..	..	100
Aromatic oil	..	..	..	18
Stearic acid	..	2	2	2
Zinc oxide	..	5	5	5
PBN	..	1	1	1
HAF Black	..	50	50	50
MOR	..	0.8	0.8	0.8
Sulphur	..	2.5	2.5	2.5

The mixing operations for laboratory evaluations of physical properties were done on a two roll laboratory mixing mill of 6" x 12" size. Mixing procedure adopted was that suggested in ASTM-D 3184-80 for black filled rubber compounds.

The rubber compounds prepared were evaluated for various processing and physical properties as per details below:

	Processing properties	Procedure
1.	Time for compounding in Mixing Mill	ASTM-D-3184-80
2.	Energy consumed for compounding in two roll mill	Standardised RRII (Capter - II)
3.	Mooney viscosity	ASTM-D-1646-81
4.	Scorch time	ASTM-D-1646-81
5.	Cure time using Rheometer	ASTM-D-2084-75
6.	Mixing in plasticorder	As per the instruction manual of Supplier.

Passenger car tyres were produced from OENR-L at M/s Apollo Tyres. The performance of these tyres were compared with those produced using oil extended SBR-(SBR 1712).

RESULTS AND DISCUSSIONS

The raw rubber properties of OENR-L is given in Table - I. It may be seen from this table that OENR-L conform to all the properties prescribed in IS 4588 for ISNR-5 grade rubber. In the case of OENR-L without benzidine,

TABLE - I

RAW RUBBER PROPERTIES

Properties	OENR-L with Benzidine	OENR-L	NR Control
Dirt content, %	0.026	0.030	0.040
Volatile matter, %	0.247	0.379	0.452
Ash content, %	0.213	0.192	0.147
Nitrogen content, %	0.370	0.367	0.356
Initial plasticity, (Po)	33	26	37
Plasticity Retention Index (PRI)	61	77	81
Mooney Viscosity ML(1+4) @ 100°C	75	51	83
Accelerated storage hardening test (ASHT) Number of units increased (Δ P)	NIL	NIL	20
Acetone extract, %	20.2	21.7	2.5

the Po value (Wallace Plasticity)was 26 as against a minimum value of 30 suggested for all grades. Acetone extract values indicated an oil content of around 18 percent. Although Po value of 30 suggested for ordinary rubber could be attained for OENR-L by using benzidine, it is not necessary that an oil extended raw rubber should conform to this Po value, prescribed for ordinary raw rubber. In fact a separate lower specification limit for Po is necessary for OENR-L. Results of ASHT indicated that oil extension of NR was helpful in reducing the tendency of NR to undergo storage hardening. Processing properties of OENR-L are given in Table - II.

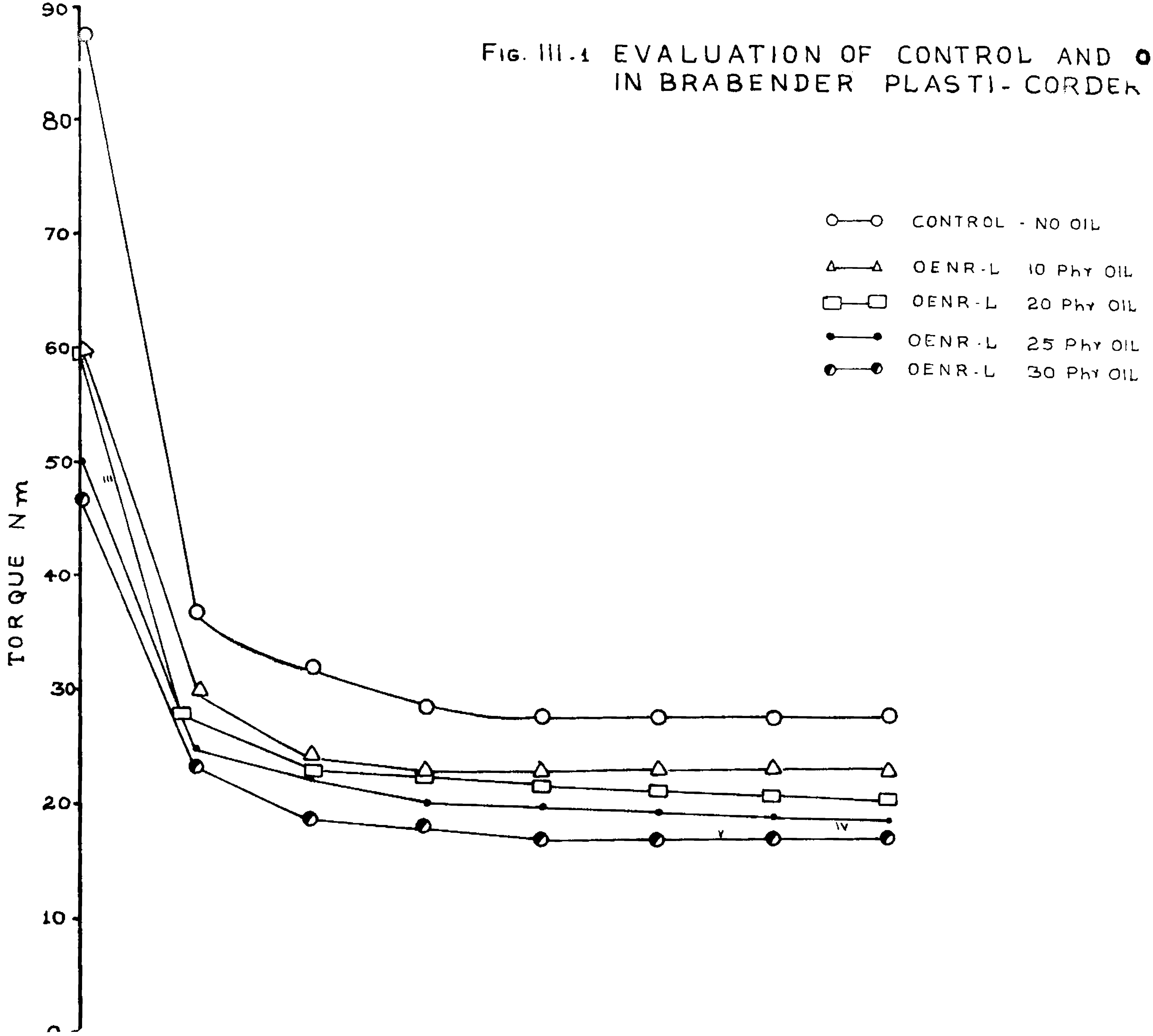
TABLE - II

PROCESSING PROPERTIES

Properties	OENR-L with Benzidine	OENR-L	NR Control
Band forming time, minutes	5	4	7
Energy consumed for band formation of 5 kg. sample KWH	1.66	1.25	2.08
Energy consumed for complete mixing of 5 kg. rubber, KWH	5.8	5.0	7.5
Time for complete mixing, minutes.	34	30	40
Mooney scroch time at 120°C minutes	28	27	31.56
Mooney Viscosity, ML(1+4) @ 100°C of the compound	51	50	51
Optimum cure time using Rheometer at 150°C, minutes	14.5	14.5	14.5

It may be seen from the data that OENR-L takes less time and power in comparison to unextended rubbers for compounding. The extent of energy saving possible upto band forming stage was 40 percent and for complete compounding 33 percent. There was no appreciable variations in scorch or cure properties of OENR-L in comparison to control except in that there was a marginal reduction in scorch time. Fig.III.1 gives the results of mixing in a plasticorder

FIG. III.1 EVALUATION OF CONTROL AND OENR-L IN BRABENDER PLASTI-CORDER



OENR-L having varying levels of oil. It is seen that the time for mixing OENR-L progressively diminishes as the oil content increases.

Technological properties of the vulcanizates are given in Tables III, IV and V.

TABLE - III

TECHNOLOGICAL PROPERTIES

Properties	OENR-L with Benzidine	OENR-L	Control
Tensile strength before Ageing (BA), MPa	21.40	22.17	19.40
Modulus at 300% Elongation, (BA), MPa	6.07	6.17	5.29
Elongation at break, (BA), %	688	706	698
Tensile strength (AA) at 70°C for 96 hrs, MPa	20.9	22.17	17.39
Modulus at 300% (AA) at 70°C for 96 hrs, MPa	7.82	7.78	7.74
Elongation at break (AA) at 70°C for 96 hrs, %	610	641	600

From Table - III it can be seen that the properties like tensile strength, modulus and elongation at break both before and after ageing were not affected by oil extending NR in latex stage. There was marginal improvement in these properties, probably owing to a more uniform mixing of the ingredients.

TABLE - IV

TECHNOLOGICAL PROPERTIES

Properties	OENR-L with Benzidine	OENR-L	Control
Compression set, %	32	38	31
Tear Strength, KN/m	23.7	24.6	22.5
Hardness, Shore A	50	50	49.0
Abrasion loss, CC/hr	1.64	1.60	1.65
Resilience, %	48.15	48.15	48.7
Heat Build up, ΔT, °C	33	33	33

TABLE - V

FLEXING STUDIES

No. of Kilocycles	OENR-L with Benzidine	OENR-L	Control
73.0	..	1	1
107.5	3	3	3
120.1	4	4	4
147.3	5	5	5
164.5	Failed	Failed	Failed

Tables IV and V give the physical and dynamic properties. It may be seen that the compression set of OENR-L without benzidine was slightly higher than the one containing benzidine. The tear strength of the control sample was lower than that for OENR-L. All other properties were comparable. The ozone resistance properties are not affected by oil extension in the latex stage as may be seen from Figs.III.2 and III.3 Ozone resistance of OENR-L was slightly better than that for OENR. Table - VI gives the variation in properties of OENR-L with varying oil content. It may be seen that as the oil content increased there was a decrease in properties like tensile strength, hardness, resilience, abrasion resistance and compression set resistance. From the data, it can be gathered that in oil extension of natural rubber, it is not advisable to increase the oil level above 25.0 phr.

Factory evaluation of OENR-L was carried out at M/s Apollo Tyres Limited, Chalakudy. Mixing was done in a size 11-D Banbury. Tyres were made using OESBR and OENR-L Results of the road wearing studies are given in Tables VII VIII and IX. Results of this study indicated that OENR-L and OESBR tyres behaved in the same manner during road test. Wearing properties of the passenger car tyres prepared with the two rubbers were also comparable.

TABLE . VI

VARIATION IN PROPERTIES OF OENR-L WITH OIL CONTENT

Properties	OENR-L 13 Phr Oil	OENR-L 15 Phr	OENR-L 21 Phr	OENR-L 23 Phr	OENR-L 26 Phr
Cure time at 150°C minutes	9	9.25	9.5	9.5	10
300% Modulus, before Ageing (BA), MPa	7.85	6.47	4.51	4.42	4.42
300% Modulus, after Ageing (AA) at 70°C for 96 hrs, MPa	9.81	8.64	5.79	6.28	5.4
Tensile strength, BA, MPa	24.04	23.75	21.49	20.71	20.61
Tensile strength, AA at 70°C for 96 hrs MPa	22.87	23.45	21.49	21.19	20.12
Elongation at break, BA, %	660	715	793	800	803
- Do - AA, %	574	652	732	715	726
Compression set, %	28.2	27.1	31.4	41.4	44.2
Hardness, Shore A	54	53	48	45	40
Resilience, %	53.2	53.2	51.9	49.9	47.6
Heat Build up, ΔT°C	31	31	29	30	31
Flex cracking resistance, kilo cycles to initiation	53.75	53.75	53.75	51.3	52.30
Kilocycles to failure	150.2	111.47	150.2	111.47	150.2

TABLE - VII

TYRE ENDURANCE TEST

A. Step speed test OENR-L. 5.20 - 14 6 PR (Rayon)
Drum Test : Constant load : 785 lbs.

Test details		Failure	Rating
Miles covered	Speed mph		
0 - 100	50		
100 - 137	75	Failed at 366 miles due to tread separation	Excellent
137 - 178	80		
178 - 220	85		
220 - 262	85		

Continue in 5 mph increments every 0,5 hrs. to failure.

TABLE - VIII

B. Step load test OENR-L. 5.20 - 14
6 PR (Rayon) Drum test. Constant speed 50 mph.

Test details		Failure	Rating
Miles covered	load lbs		
0 - 200	785		
200 - 500	855		
500 - 1700	915	Failed at 4300 miles due to tread separation.	Excellent
1700 - 2500	915		
2500 - 2900	915		
2900 - 3300	1100		

Continue test increasing the loading 157 lbs every 400 miles to failure.

TABLE - IX

ROAD TEST PERFORMANCE (Ist CHECK)

Vehicle No.1 covered (12957 KM)			Vehicle No.2 (covered 5033 KM)	
Tyre	Original NSD in inches	% wear	Original NSD in inches	% wear
OENR-L	$\frac{11}{32}$	51	$\frac{11}{32}$	22
Control	$\frac{11}{32}$	50	$\frac{11}{32}$	22

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CHAPTER - IV

PROCESSING OF NATURAL RUBBER LATEX IN ENERGY SAVING FORMS WITH INCORPORATION OF PEPTISERS

CHAPTER - IV

PROCESSING OF NATURAL RUBBER LATEX IN ENERGY SAVING FORMS WITH INCORPORATION OF PEPTISERS

Natural rubber undergoes hardening during storage[1,2]. Therefore, when it reaches the consumers' factory from producing estates, it attains a Mooney Viscosity in the range of 80 to 90. This difficulty can be overcome by incorporating hydroxylamine hydrochloride with NR latex at the time of processing[3]. The rubber marketed in this form is called constant viscosity (CV) rubber. Although hydroxylamine salts are not essential in rubber compounding, its addition is not in any way harmful. Peptising agents are also known to reduce Mooney Viscosity without affecting the rate of vulcanization and properties of cured rubber[5]. During mastication of rubber at high temperature, peptiser promotes rapid oxidation and hence reduces the Mooney Viscosity. The peptisers commonly used in rubber industry are different forms of activated penta chloro thiophenol, (Renacit-7). Mercapto benzothiazole (MBT) and Xylyl mercaptan (RPA-3) are also reported to possess peptising properties. Peptisers are added to dry rubber during compounding in banbury.

It was thought appropriate in this context to evaluate the effect of incorporating different peptisers to fresh field latex at the time of processing, on various raw rubber properties. The results of such an investigation are reported here.

In the first part of the study, the efficiency of peptising action of the three peptisers were compared and the dosage of each for the required level of peptising action was assessed. In the second part the processing and physical properties of the rubber prepared with incorporation of peptisers were assessed and compared with those of control samples. Control samples taken in this study were constant viscosity rubber and ordinary block rubber, both prepared from the same lot of latex used for preparing the peptiser incorporated test samples.

EXPERIMENTAL

The peptisers were converted to suitable colloidal form for ensuring thorough mixing with rubber latex. MBT and Renacit-7 were converted to dispersions by ball milling them separately with water and dispersing agent. The proportions used are given under:

MBT	100	..
Water	98	98
Dispersol-F	2	2
Renacit-7	..	100

RPA-3 is a liquid and so it was converted to an emulsion in water using ammonium oleate soap as the emulsifying agent.

The concentrations of peptisers added to rubber are given in Table-1. Constant viscosity and ordinary rubber were also prepared side by side from the same batch of latex. The processed rubber was then examined for their Mooney Viscosity (ML(1+4) @ 100°C). Raw rubber properties were evaluated as per the procedures given in Chapter - II.

Variation in molecular weight of rubber with variation in peptiser concentration was assessed by the viscosity average method. The peptised rubber was then compounded for evaluation of the processing and physical properties. Formulations for compounding are given in Table-II. The processing properties evaluated are mastication time, scorch time, compound viscosity and energy saving. Cure characteristics of the sample were evaluated using Monsanto Rheometer R-100. Technological properties such as tensile strength, elongation at break, modulus, hardness rebound resilience, compression set, abrasion resistance, flex resistance and heat build up properties were also evaluated. The details of procedures adopted are given in Chapter-II.

A separate study was undertaken to see the effect of incorporating antiozonants on flex properties of peptiser incorporated rubber. Formula used for this study is given in Table-III.

TABLE - I

PREPARATION OF RUBBER SAMPLES

Rubber content in latex	MBT % on rubber	Renacit-7 % on rubber	RPA-3 % on rubber
100	0.5(A_1)	0.5(A_2)	0.5(A_3)
100	0.25(B_1)	0.25(B_2)	0.25(B_3)
100	0.15(C_1)	0.15(C_2)	0.10(C_3)
100	0.10(D_1)	0.10(D_2)	0.05(D_3)
100	0.05(E_1)	0.05(E_2)	0.05(E_3)
100	0.025(F_1)	0.025(F_2)	....
100	....	0.010(G_2)	....

TABLE - II

FORMULATION OF COMPOUND

Ingredients	Parts by weight			
	MIX 1	MIX 2	MIX 3	MIX 4
Latex stage peptised rubber (MBT - 0.025 phr)	100	..	..	..
Latex stage peptised rubber (Renacit-7 0.01 phr)	...	100	..	..
CV Rubber	...	..	100	..
Control	...	..	..	100
Zinc Oxide	3.5	3.5	3.5	3.5
Stearic Acid	2.0	2.0	2.0	2.0
Polymerised 1,2-Dihydro 2,2,4-Trimethyl Quinoline	1.0	1.0	1.0	1.0
HAF Black	50	50	50	50
Aromatic Process Oil	5.0	5.0	5.0	5.0
Cyclohexyl Benz Thiazyl Sulphenamide	0.6	0.6	0.6	0.6
Sulphur	2.5	2.5	2.5	2.5

TABLE - III

FORMULATION OF COMPOUND

Ingredients	Parts by weight			
	MIX 5	MIX 6	MIX 7	MIX 8
Latex Stage peptised Rubber (MBT-0.025 phr)	100	...	...	...
Latex stage peptised rubber (Rencit-7 0.01 phr)	...	100	...	...
CV rubber	...	...	100	...
Control	...	...	...	100
Zinc oxide	3.5	3.5	3.5	3.5
Stearic Acid	2.0	2.0	2.0	2.0
Phenyl-β-Naphthyl Amine	1.0	1.0	1.0	1.0
N-(1,3-Dimethyl Butyl N-phenyl P-Phenylene Diamine.	1.0	1.0	1.0	1.0
HAF Black	50.0	50.0	50.0	50.0
Aromatic Process Oil	5.0	5.0	5.0	5.0
N-Cyclohexyl Benzthiazyl Sulphenamide	0.6	0.6	0.6	0.6
Sulphur	2.5	2.5	2.5	2.5

RESULTS AND DISCUSSION

Table-IV gives the variation in Mooney Viscosity and Wallace Plasticity of rubber samples prepared with varying concentrations of peptisers, MBT and Renacit-7. It may be seen from the table that there is decrease in Mooney Viscosity and Wallace Plasticity for raw rubber prepared with incorporation of peptisers in latex stage. The Mooney Viscosity was found to decrease with increase in concentration of peptisers. RPA-3 did not show any peptising action - under the conditions of this experiment. The dosage of these chemicals for dry mixing is usually between 0.1 to 0.3 phr. It is clear from the data in Table-IV that when added in latex stage, concentrations above 0.1 phr was harmful to rubber and samples which contained peptisers above this level were in a highly degraded form. Addition of peptising agents depolymerises natural rubber during drying process. The thermo-oxidative break-down of rubber during mastication is catalysed by peptisers. They are effective as radical acceptors at lower temperature in the absence of oxygen and as promoters of oxidative break-down through formation of primary radicals or as oxidation catalyst through dissociation of spontaneously formed chain hydroperoxides. Peptising agents also shift the start of thermo-oxidative reaction to lower temperatures. It can be seen from the data that when added in latex stage MBT at 0.025 phr level or Renacit-7 at 0.01 phr level could

TABLE - IV

EFFECT OF VARYING CONCENTRATION OF PEPTISERS ON RAW RUBBER (see TABLE I) PROPERTIES

Rubber Sample		Peptiser	Mooney viscosity ML(1+4) @ 100°C	Initial wallace plasticity(Po)	Plasticity Retention Index (PRI)	Accelerated Storage Hardening (P)
1.	A1	MBT	Rubber became tacky due to excessive degradation.			
2.	B1	"				
3.	C1	"				
4.	D1	"	49	22	54.5	+6
5.	E1	"	61	30	65.5	+10
6.	F1	"	62	30	65.5	+8
7.	A2	Renacit-7	Rubber became too much tacky.			
8.	B2	"				
9.	C2	"				
10.	D2	"	39	18	61.1	+4
11.	E2	"	47	21	57.2	+6
12.	F2	"	53	22	50	+6
13.	G2	"	60	30	74	+4
14.	Constant Viscosity Rubber	0.15 phr Hydroxyl Amine Hydrochloride	71	39	76.9	+3
15.	Control	Normal acid coagulated rubber	81	45	82.6	+12

degrade rubber to a Mooney Viscosity in the range 60 - 70, which is the viscosity range for premasticated rubber. So these concentrations were selected as the optimum level of peptisers, MBT and Renacit-7 respectively, for incorporation in latex stage.

Processing and technological properties were examined only on rubber samples prepared with these optimum concentration of peptisers. Control and CV rubbers were also simultaneously examined for these properties. From Tables IV and V, it may be seen that Po and PRI values are slightly lower for the peptiser incorporated rubber samples when compared to those for control and CV rubbers. But the values obtained for these properties were within the limits prescribed for ISNR-5 (IS-4588). All the other raw rubber properties for the control and treated samples were comparable. The viscosity average molecular weight of rubber samples prepared with incorporation of peptisers is given in Table-VI. It may be seen from the results that the molecular weight decreases with increase in concentration of peptisers. Energy saving possible using peptised rubbers is given in Table-VII. It may be seen that upto 46.7 percent energy can be saved in pre-mastication stage by using peptised rubbers in place of ordinary sheet or block rubbers.

Formulation used for compounding are given in

TABLE - V

RAW RUBBER PROPERTIES

Sample No.	Treatment	Nitrogen (%)	Ash content (%)	Volatile matter (%)	Dirt content (%)
1.	Rubber treated with 0.025 phr MBT at latex stage.	0.3721	0.3679	0.4812	0.0357
2.	Rubber treated with 0.01 phr Renacit-7 at latex stage.	0.3676	0.3655	0.5618	0.0381
3.	CV Rubber	0.3918	0.4185	0.5272	0.0417
4.	Control	0.3782	0.3504	0.4966	0.0393

TABLE - VI

VISCOSITY AVERAGE MOLECULAR WEIGHT

Concentration of peptiser MBT on Rubber	Molecular weight
Control	9.63×10^5
0.01 Phr	8.43×10^5
0.02 Phr	8.32×10^5
0.04 Phr	7.44×10^5
0.06 Phr	7.20×10^5
0.08 Phr	6.44×10^5
0.10 Phr	5.46×10^5

TABLE - VII

ENERGY SAVING BY USING PEPTISED NATURAL RUBBER

BATCH WEIGHT - 4 KILOGRAMS.

Sample details	Energy in KWH for 1kg. Rubber mastication.	Time for mastication of 4 kg.	Percentage saving in energy (%)
Control	0.375	5'45"	-
Rubber with 0.025 Phr Renacit-7	0.20	4'15"	46.7
Rubber with 0.05 Phr MBT	0.225	4'30"	40.0
CV Rubber	0.275	5'45"	26.7

Tables II and III. Mixes 1 & 2 are rubber samples prepared with incorporation of peptisers, 3 is constant viscosity rubber and 4 control. Processing properties are given in Table-VIII.

It may be seen that the mastication time is reduced appreciably for the rubber samples prepared with incorporation of peptiser. The reduction in mastication time for the samples prepared with incorporation of peptiser is to the extent of 45 percent when compared with that for the control. All other properties like compound viscosity, scorch time at 120°C and optimum cure time at 150°C were comparable for the control and treatments. From Table VII it may be seen that an energy saving of upto 45 percent during rubber premastication process is possible by using rubber prepared with incorporation of peptiser.

The technological properties of mixes 1,2,3 and 4 are given in Table-IX. Tensile strength, modulus, tear strength, elongation at break, hardness, rebound resilience, properties after ageing, abrasion resistance, compression set and heat build up of all samples are comparable. But flex resistance of peptised rubbers were inferior to CV and control rubbers. It may be seen from Table-IX that with the incorporation of antiflex cracking agents during compounding, the flex resistance of peptised rubbers can be increased considerably and it became better than control and CV rubbers.

TABLE - VIII

PROCESSING PROPERTIES

Properties	MIX 1	MIX 2	MIX 3	MIX 4
Mastication time (minutes)	4.5	5.5	7	8.5
Total compounding time (minutes)	32.5	31	33.5	36.5
Scorch time at 120° (minute)	20.5	21.5	24	21.75
Optimum cure time at 150°C (minutes)	9.5	9.0	9.5	9.5

TABLE - IX

FLEX PROPERTIES

Flex property	MIX 1	MIX 2	MIX 3	MIX 4
Flex Initiation K cycles	75.88	60.01	60.01	65.80
Failure K cycles	226.52	282.04	228.52	161.81

TABLE - X

TECHNOLOGICAL PROPERTIES

Property	MIX 1	MIX 2	MIX 3	MIX 4
1. Tensile strength (MPa)	23.52	23.52	23.52	23.52
2. Modulus (300%) (MPa)	8.82	8.82	8.62	8.82
3. Elongation at break (%)	610	610	610	610
4. Hardness (Shore A)	58	57	56	59
5. Resilience (%)	50.03	47.58	48.18	50.6
6. Abrasion loss (CC/hr)	0.806	0.9633	0.9328	0.8264
7. Compression set (%)	45.4	42.5	49.8	38.5
8. Heat build up (ΔT°C)	36	39	36	37
9. Flexing initiation (K cycles)	40.8	42.1	65.2	63
Failure (K cycles)	98.8	98.8	135.6	117.1
10. After Ageing at 70°C for 96 hours.				
(a) Retention of Tensile strength (MPa)	9.878	9.18	10.22	9.72
(b) Retention of Elongation at break (%)	86.9	85.2	90.0	86.06
(c) Retention of modulus (%) (MPa)	13.06	13.61	13.367	13.61

CONCLUSION

The incorporation of either of the two peptisers viz., MBT at 0.025 phr level or activated pentachlore thio phenol at 0.01 phr level to natural rubber latex, reduces, Mooney Viscosity of the resultant rubbers to the range of 60-70 which is the range of viscosity observed in premasticated rubbers. The mastication time required for these rubber is reduced to an extent of 45 percent when compared to control sample. The raw rubber processing and technological properties of these rubbers are comparable to CV and control samples. Molecular weight of rubber decreases with increase in concentration of peptisers.

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CHAPTER - V

INVESTIGATION OF CLONAL VARIATIONS IN PROCESSING PROPERTIES OF NATURAL RUBBER AND OPTIMUM USE OF FIELD COAGULA IN BLENDING WITH LATEX COAGULA

CHAPTER - V

INVESTIGATION OF CLONAL VARIATIONS IN PROCESSING PROPERTIES OF NATURAL RUBBER & OPTIMUM USE OF FIELD COAGULA IN BLENDING WITH LATEX COAGULA

Natural Rubber obtained from different clones of Hevea Brasiliensis have different Mooney Viscosity, gel content, non-rubber content and cure properties. There is also seasonal variations in these properties. The rubber produced from different clones have been categorised according to their Mooney Viscosity, in order to identify those, yielding rubbers with low, medium and high Mooney viscosities and the results are given in this chapter.

The rubber produced from field coagula in the form of estate brown crepe or solid block form (see Chapter II) is usually blended with sheet rubber prepared from latex at the time of compounding in manufacturer's factory. It may be advantageous to blend field coagula with latex coagula itself, to obtain a rubber of Mooney viscosity in the range of masticated natural rubber. The various aspects of blending wet and dry field coagula with latex coagula have been investigated and these results are also included in this chapter.

EXPERIMENTAL

Latices from thirteen clones were collected (see Chapter II) on four occasions at two months interval in order to assess the seasonal variation in properties also. The latices were diluted to 30% drc immediately after collection. After this, the latex collected from each clone was divided into two equal parts. One part in each case was used for making CV Rubber (see Chapter II) and the other processed in the usual manner. Both were prepared in crepe form.

Blending of Latex Coagula with Field Coagula:

The dry field coagula were soaked in water for about 24 hrs. and passed through a macerator giving sufficient washing to remove the non-rubber constituents. Repeated passing of this through the macerator gave a blanket which was used for blending. Wet field coagula prepared with hydroxyl amine hydrochloride were also blanketed separately by passing through the macerator. The latex coagulam was washed and blanketed in the macerator. Blends of latex coagula/dry field coagula and latex coagula/wet field coagula in the ratio given in Table-IX were prepared by blanket to blanket blending of appropriate quantities (on drc basis) of the respective blankets. Four to five passes through the macerator were required to get uniform blends of the two types of blankets. The homogenised blend thus obtained was then passed through a creper hammer mill, and crumbled. The crumbs were then dipped in a 0.3 percent solution of hydroxylamine hydrochloride solution for one hour to effect viscosity stabilisation. The treated crumbs were dried at 100°C and baled. This portion of the experiment was laid down on a statistical basis using the split plot design with three replication.

<u>Evaluation</u>:

The rubber samples prepared from latices of different clones and the latex and field coagula blends prepared in various proportions were separately tested for raw and vulcanisate properties. All the samples prepared were examined for raw rubber properties as detailed in IS.4588. The properties were examined as per procedures given in Chapter II.

The rubber samples were also examined for their Mooney viscosity and accelerated storage hardening. The rubber samples obtained by processing latices from the selected clones were evaluated for their cure characteristics as per ACS1 formulation. All the samples were compounded in a typical tyre tread receipe given below:

Rubber - 100
ZnO - 5
Stearic acid - 2
PBN - 1
Santoflex IP - 1
HAF Black - 50
CBS - 0.6
Sulphur - 2.5

The materials prepared from latices were compounded in another formula also for extrusion study by replacing

HAF black with SRF Black. The technological properties were evaluated according to procedure given in Chapter II.

Truck tyre treads were prepared using the rubber samples prepared from latex and field coagula blends at M/s Madras Rubber Factory Ltd, Vadavathoor and used for fabricating new tyres. The performance of these tyres were compared with those prepared using conventional forms of rubbers.

RESULTS & DISCUSSIONS.

Table-I gives the stabilised and non-stabilised Mooney Viscosity and initial plasticity of rubber prepared from latices of selected clones. It may be seen from this table that Mooney Visocisty of viscosity stabilised rubbers fall in the range of 50-93. Cure characteristics of the rubbers are shown in Table-II and III. It may be seen from the data that hydroxyl amine hydrochloride treatment slightly retards cure. This may be attributed to its slight acidic nature. In Table-IV classification of rubber is made on the basis of their viscosity range. It may be seen from the data that out of the 13 clones evaluated five gave rubber of medium visocisty in the range of 61 ± 5 This group included following clones, viz. RRIM-600, GT-1, RRII-105,GG-1 and GL1. It may be mentioned here that a vast majority of the newly planted and replanted area in India are covered by one or the other of these clones. So in the days ahead, it is possible to produce rubber in an

TABLE - 1

WALLACE PLASTICISTY & MOONEY VISCOSITY OF RUBBER FROM DIFFERENT CLONES

Sl. No.	Clone	Wallace Plasticity		Mooney Viscosity $ML(1+4)^{100^{\circ}C}$	
		Hydroxyl amine treated.	Untreated	Hydroxyl-amine treated.	Un-treated
1.	RRII 105	36	49	58	75
2.	RRII 118	53	59	79	86
3.	RRIM 605	32	53	50	80
4.	RRIM 600	36	55	56	82
5.	RRIM 623	45	56	70	79
6.	RRIM 628	54	63	81	88
7.	GT-1	38	56	60	76
8.	GG-1	41	47	66	78
9.	GL-1	36	51	60	75
10.	PR-107	46	55	69	78
11.	PB-86	53	64	77	90
12.	PB5/139	67	70	93	98
13.	TJir-1	56	63	83	89

TABLE - II

CURE CHARACTERISTICS OF THE RUBBER FROM DIFFERENT CLONES AT 150°C

Sl. No.	Clone	Scorch time (t2) (Mean) in mints.	Cure rate index (Mean)
1.	RRII 105	5	7.6
2.	RRII 118	5	7.1
3.	RRIM 605	4	7.7
4.	RRIM 600	5	7.7
5.	RRIM 623	7	5.9
6.	RRIM 628	6	5.9
7.	GT-1	4	8.3
8.	GG-1	5	7.2
9.	GL-1	6	6.7
10.	PR-107	7	5.5
11.	PB-86	4	8.3
12.	PB 5/139	7	6.7
13.	TJir-1	6	6.5

TABLE - III

EFFECT OF HYDROXYLAMINE ON CURE CHARACTERISTICS AND RETENTION INDEX OF RUBBER FROM DIFFERENT CLONES

Sl. No.	Clone	Untreated			Hydroxylamine treated		
		t2(min.) 150°C	Cure rate index 150°C	PRI	t2 (min) 150°C	Cure rate index 150°C	PRI
1.	RRIM 605	4	7.7	82	6	7.2	88
2.	RRIM 600	5	7.7	75	7	6.9	76
3.	TJir-1	6	6.5	81	7.5	6.1	80

TABLE - IV

CLASSIFICATION OF RUBBER FROM DIFFERENT CLONES BASED ON MOONEY VISCOSITY

Low Viscosity (51±5)	Medium Viscosity (61±5)	High Viscosity (66 and above)
RRIM 605	RRII 105	RRII 118
	RRIM 600	RRIM 623
	GT-1	RRIM 628
	GG-1	PB - 86
	GL-1	PR - 107
		PB 5/139
		TJir-1.

ideal viscosity range of 61 ± 5 if a proper and planned effort is made in finalising a processing policy for natural rubber in this country.

Raw rubber properties of visosity stabilised rubbers from those clones, coming in the medium viscosity range were compared with those of masticated RSS-1 in Table-V. All the properties are found to be better for viscosity stabilised type of rubbers. Table-VI gives the processing properties of the above rubbers when compounded in the tread receipe. It may be seen from the data that the mixing and compounding time for masticated RSS-1 and these rubbers in the viscosity range of 61 ± 5 are the same. In masticated RSS-1 already energy is expended for pre-mastication, whereas in viscosity stabilised rubbers prepared by selective blending techniques, energy saving is attained as their time for compound preparation, was the same as that required by masticated rubbers. The physical and dynamic properties of viscosity stabilised rubber prepared from the selected latices are compared with those of rubber from masticated RSS-1 in Table-VII. It may be seen from the data that the results are comparable. Table-VIII gives the extrusion characteristics of the rubber from viscosity stabilised latices. The extrusion properties are uniformly better.

Figure V.1 gives the seasonal variation in Mooney viscosity of viscosity stabilised rubbers from various clones. The effect of blending of clonal latices on Mooney

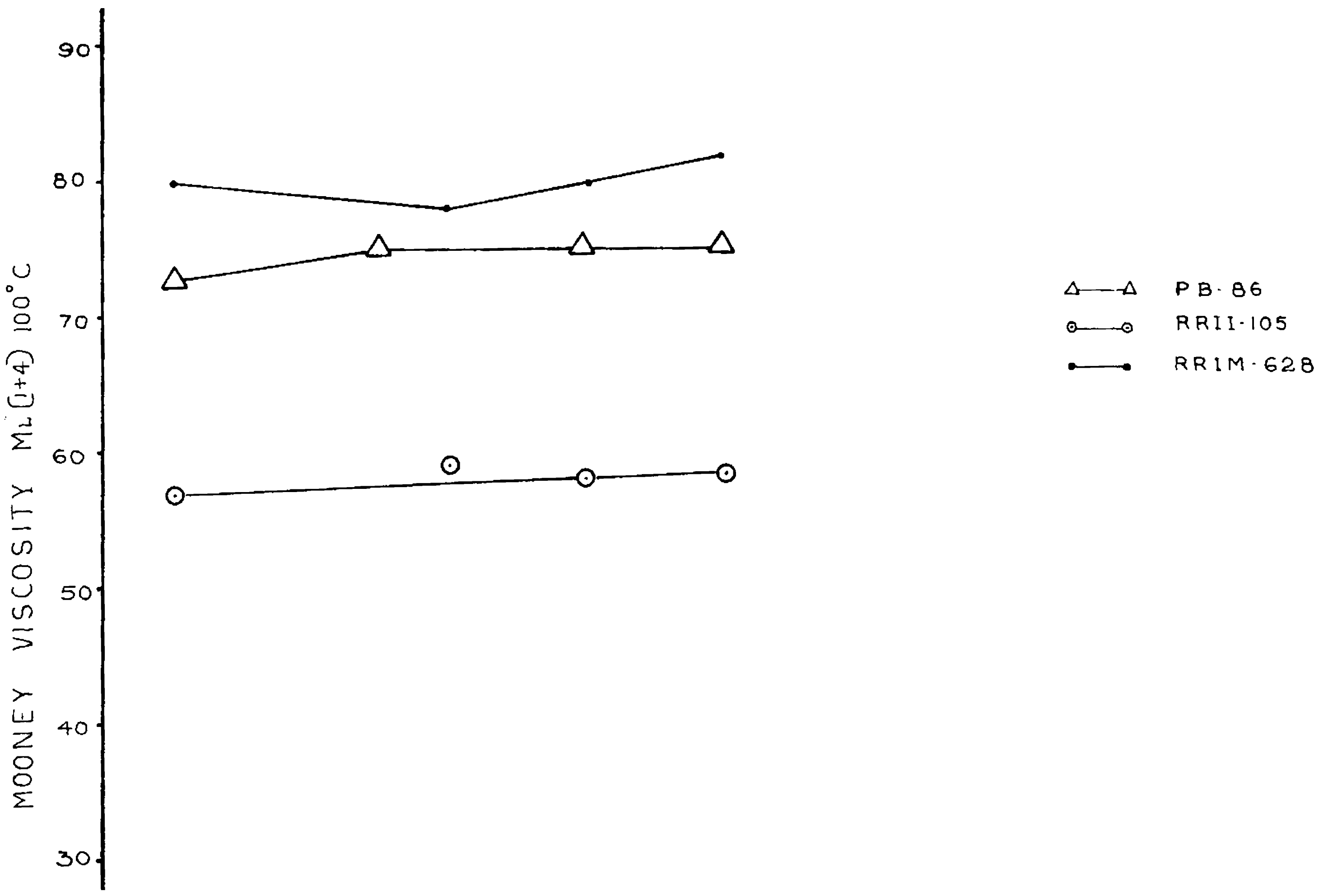


FIG. V. 1 SEASONAL VARIATION IN MOONEY VISCOSITY OF RUBBER FROM DIFFERENT CLONES.

TABLE - V

RAW RUBBER PROPERTIES

Property	Viscosity stabilised rubber (from the selected clones)	Masticated (RSS-1)
Mooney Viscosity ML(1+4)100°C	62	64
Wallace Plasticity (Po)	39	40
PRI	76	70
A.S.H.T	+4	+10

TABLE - VI

PROCESSING AND CURE PROPERTIES

Property	Viscosity stabilisation.	Masticated (RSS-1)
Time for mixing (Min.)	34	32
Mooney Scorch at 120°C (min.)	28	24
Optimum Cure time at 150°C (min.)	12	12

TABLE - VII

TECHNOLOGICAL EVALUATION

Property	Viscosity stabilised rubber	Masticated (RSS-1)
Modulus 300% MPa		
B.A	12.54	12.45
A.A	15.68	16.46
% retention	128	127
Tensile strength MPa		
B.A	24.69	24.79
A.A	24.01	23.91
% retention	97	97
Elongation at break %		
B.A	525	523
A.A	455	450
% retention	87	86
Specific gravity	1,13	1.13
Abrasion loss cc/hr	0.52	0.53
Hardness shore A	63	62
% resilience	55	54
Compression set %	38	38
Tear strength KN/M	121.5	118.58
Demattia flexing K/C	155	155

B.A - Before ageing

A.A - After ageing at 70±1°c for 96 hrs.

TABLE - VIII

EXTRUSION CHARACTERISTICS

Sample	Compound viscosity ML(1+4) 100°C	Length in Cm/Min.	Wt. in gms/ min.	Sp. Gravity	% Die Swell	Rating System B (ASTM)	T°C
Viscosity stabilised rubber.	33	195	139	1.04	38	3,3,4,4	17°
Masticated (RSS.1)	43	190	142	1.04	44	3,3,4,4	27°

TABLE - IX

RATIO OF LATEX & FIELD COAGULAM BLENDS

No.	Latex rubber	Field coagulum rubber (M1/M2)
S1	60	40
S2	50	50
S3	40	60
S4	30	70
S5	20	80

Viscosity of rubber is given in Fig.V.2. These observations are in agreement with the findings of Ching Peng Sung.[1]

Tables-X to XV gives raw rubber properties of blends of latex and field coagula rubbers on the basis of statistical study conducted on blends prepared from latex and the two types of field coagula materials in different proportions as per details in Table-IX. The results show that the variations in dirt content was significant at one percent level for both the blends of latex coagula with wet or dry field coagula. As the field coagulam content increased, the dirt content also increased for both the types of blends. But in the case of latex/wet field coagula blends, the dirt content was considerably lower when compared with that of latex/dry field coagula blend in all the blend ratios.

The variation in volatile matter content is found significant at 5 per cent level for both types of blends. The latex/wet scrap blends were having slightly higher moisture levels compared with the latex/dry scrap blends. Among the blends in each type, PRI was found to be significant at 5 percent level. But for both the types of blends 40:60 ratio of latex/field coagula gave consistant PRI of above 60. For both the types of blends nitrogen content and ash content were significant at 5 percent level, for the different ratios of blends. Among the blends prepared

FIG. V. 2 BLENDING OF LATICES FROM DIFFERENT CLONES FOR VISCOSITY ADJUSTMENTS

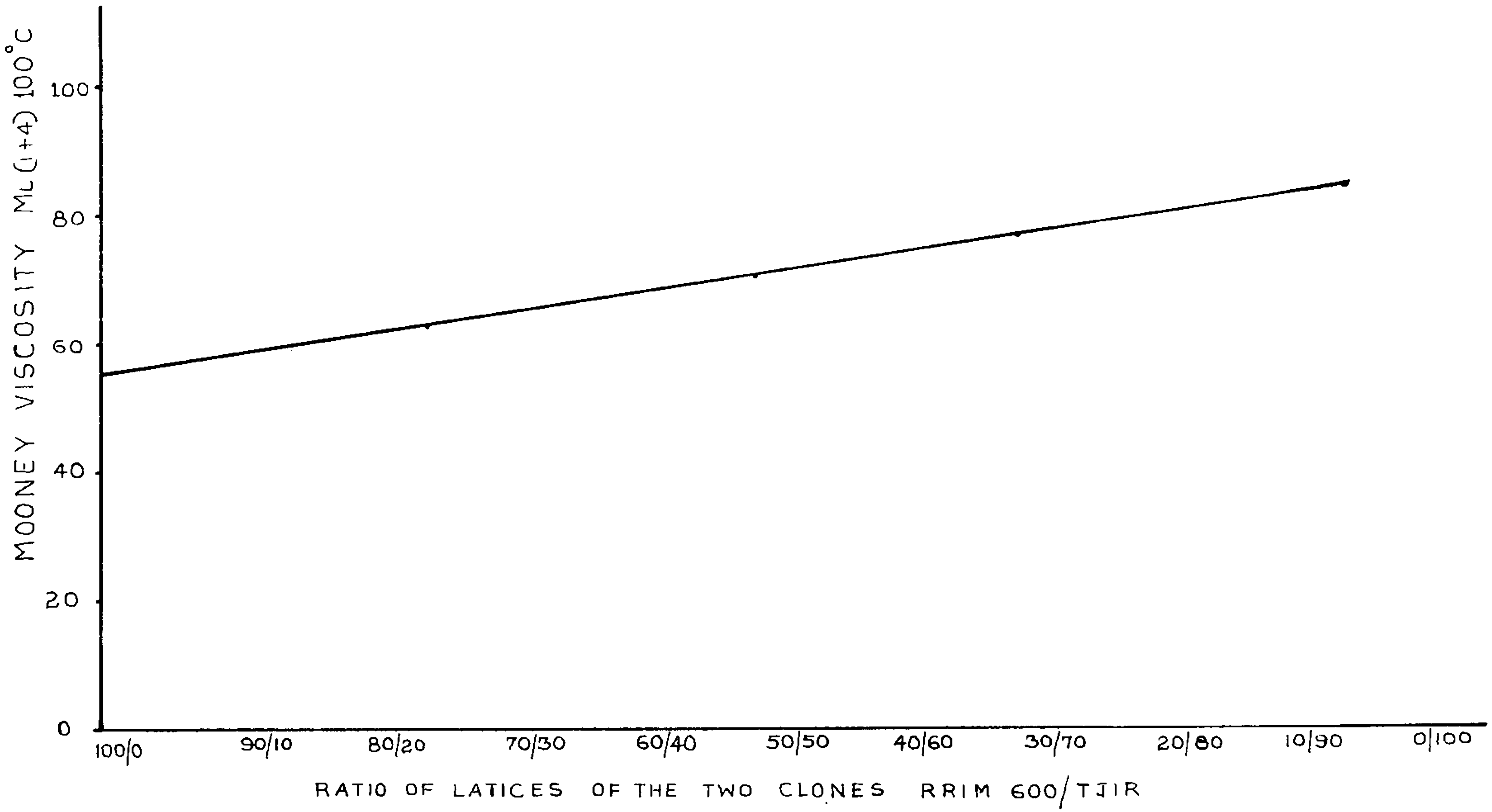


TABLE - X

MEAN DIRT VALUES

	M1	M2	Mean S;E.0.0042 C:D.0126+ 0.0173++
S1	0.0388	0.0297	0.0342
S2	0.0484	0.0188	0.0336
S3	0.0518	0.0271	0.0395
S4	0.0815	0.0336	0.0576
S5	0.0839	0.0429	0.0634
	0.0609	0.0304	0.0457

Mean: S:E. 0.0025
C:D. 0.0152+
0.035

TABLE - XI

MEAN VOLATILE MATTER VALUES

	M1	M2	Mean S:E.0.0058 C:D.0.0174+ 0.024‡
S1	0.2540	0.3470	0.3005
S2	0.2599	0.3398	0.2999
S3	0.2759	0.3733	0.3246
S4	0.2755	0.3591	0.3173
S5	0.2876	0.3884	0.3380
	0.2706	0.3615	0.3160
Mean S:E.	0.0095		
-	0.0584+		
	0.134‡		

+Sig. at 5% level ++ Sig. at 1% level

S.E Standard Error C.D Critical Difference.

TABLE - XII

MEAN PRI VALUES

	M1	M2	Mean S:E. 1.09 C.D. 3.27+ 4.50++
S1	67.3	74.3	70.8
S2	67.7	68	67.8
S3	64	65.3	64.7
S4	61	61.7	61.3
S5	59.3	57.7	58.4
Mean S.E: - C:D.	63.9	65.4	64.6

TABLE - XIII

MEAN NITROGEN VALUES

	M1	M2	Mean S:E.0.0072 C:D.0.0216+ 0.0297++
S1	0.4120	0.4284	0.4195
S2	0.3895	0.4158	0.4027
S3	0.3741	0.3693	0.3717
S4	0.3890	0.3985	0.3937
S5	0.3810	0.4154	0.3982
Mean	0.3887	0.4055	0.3971
S:E.-- C:D.--			

TABLE - XIV

MEAN ASH VALUES

	M1	M2	Mean S:E.0.0095 C:D.0.0285 0.0392
S1	0.2465	0.2517	0.2491
S2	0.2494	0.2603	0.2548
S3	0.2804	0.2890	0.2847
S4	0.2965	0.2855	0.2927
S5	0.3272	0.2507	0.2891
Mean	0.2807	0.2075	0.2741
S:E: --			
C:D: --			

TABLE - XV

MEAN MOONEY VISCOSITY VALUES

	M1	M2	Mean S:E:- C:D:-
S1	65	64.7	64.8
S2	64.3	64.7	64.5
S3	63.7	64.7	64.2
S4	64.7	67	65,8
S5	64.7	62	63.3
Mean S:E:	64.5	64.6	64.5

in different ratios, Mooney Viscosity was not found significant for both the types of blends. The mean value for this property was found to be in the range of 63 to 65.

The processing behaviour of the two selected blends of latex and field coagula (both wet and dry in the ratio 40:60)are compared in Table-XVI with those of masticated RSS-1. It may be seen from the results that the processing properties of the blends are comparable to those of masticated RSS-1. Energy consumption of these blends during mastication and compounding was assessed. These data are presented in Table-XVII along with those obtained for CV rubbers and ordinary block rubbers. It may be seen from the Table that viscosity stabilised latex/coagulam blends consume less energy in processing. In comparison to conventional blends it has shown energy saving to the extent of 50 percent upto the stage of mastication and band formation. It may be seen from the data that viscosity stabilised wet coagulam/latex blends showed greater energy saving than corresponding dry coagulam/latex blends.

Technological properties of the vulcanisates obtained from the two selected blends (60 coagulam and 40 latex on drc basis in both cases) were compared with those of vulcanisates prepared from masticated RSS-1 in Table-XVIII. The blends from latex and wet coagula gave properties comparable to those of masticated RSS-1. However, the blends from

TABLE - XVI

PROCESSING & CURE PROPERTIES OF THE 40/60 BLENDS AND MASTICATED RSS.1 TREAD COMPOUND

Property	Latex/ Fresh Scrap	Latex/ Dry Scrap	Masticated RSS.1
Time for band formation Min.	5	5	4
Time for complete mixing Min.	34	34	33
Mooney scorch at 120°C	21.5	21.5	22.5
Optimum cure time @ 150°C	12	12	11

+ Sig.at 5% level
++ Sig.at 1% level

TABLE - XVII

ENERGY SAVING POSSIBLE BY USING DIFFERENT FORMS OF RUBBER

Processing	Block Rubber CV	Block Rubber CV	Block rubber from latex/ scrap CV		Block rubber from scrap Non-CV	
			Wet scrap blend	Dry scrap blend	Wet scrap blend	Dry scrap blend
1. Band forming time - minutes.	8	12	6	6	12	9
2. Energy/kg in KWH for band formation	0.3	0.5	0.2	0.2	0.4	0.3
3. Percentage of energy saving for CV rubber at band forming stage	40%	..	50%	33%		
4. Energy /kg in KWH for complete compound in a typical tyre tread recipe.	0.65	0.83	0.59	0.65	0.77	0.77
5. Percentage of energy saving for CV rubber at the stage of complete compounding	22%	..	23%	16%	..	..

latex/dry field coagula gave vulcanisates of lower abrasion resistance, flex resistance and tear strength when compared with those of vulcanisates from masticated RSS-1.

From Tables-XIX and XX it is seen that the projected mileage for the treads from blends of latex and wet and dry field coagula was higher than those of treads from RSS/EBC blends. It could also be noticed from the results that the service performance of latex/wet field coagula blends was better than that of blends from latex/dry field coagula.

Reference:

1. Chin Peng Sung   J. Rub. Res. Inst., Malaya 22, 56 (1969).

TABLE - XVIII

PHYSICAL PROPERTIES OF THE LATEX-SCRAP 40/60 BLENDS AND MASTICATED RSS.1

Property	Latex/Fresh scrap	Latex/Dry scrap	Masticated RSS.1
Modulus 300% MPa			
B.A	12.25	11.76	12.44
A.A	15.68	15.19	15.87
% retention	128	129	127.5
Tensile strength MPa			
B.A	24.5	23.52	24.69
A.A	23.52	22.39	23.91
% retention	85%	8.4%	86%
Specific gravity	1.140	1.142	1.136
Abrasion loss cc/hr	0.5819	0.5998	0.533
Hardness shore A	64	63	62
Resilience %	54	52	54
Compression set %	41.64	42.53	38.57
Tear strength KN/M	117.6	107.8	118.58
Demattia flex k/c (Average to C stage)	115	107	136

B.A - Before ageing A.A - After ageing at 70±1°C for 96 hrs.

TABLE - XIX

EVALUATION OF SERVICE PERFORMANCE

Property		Control	Latex/wet scrap blend
Original skid depth (inch)		19/32	19/32
Distance covered (kms)		1281	1281
Present skid depth (inch)	(1)	16.15/32	16.28/32
	(2)	16.24/32	16.37/32
% Wear	(1)	15%	14.3%
	(2)	14.5%	13.84%
Projected milage (kms)	(1)	8539	8948
	(2)	8818	8254

TABLE - XX

EVALUATION OF SERVICE PERFORMANCE

Property	Control	Latex/dry scrap blend
Original skid depth (inch)	19/32	19/32
Distance covered (kms)	1132	1132
Present skid depth (inch)	16.4/32	16.62/32
% wear	13.68	12.52
Projected mileage (kms)	8272	9036

CHAPTER - VI

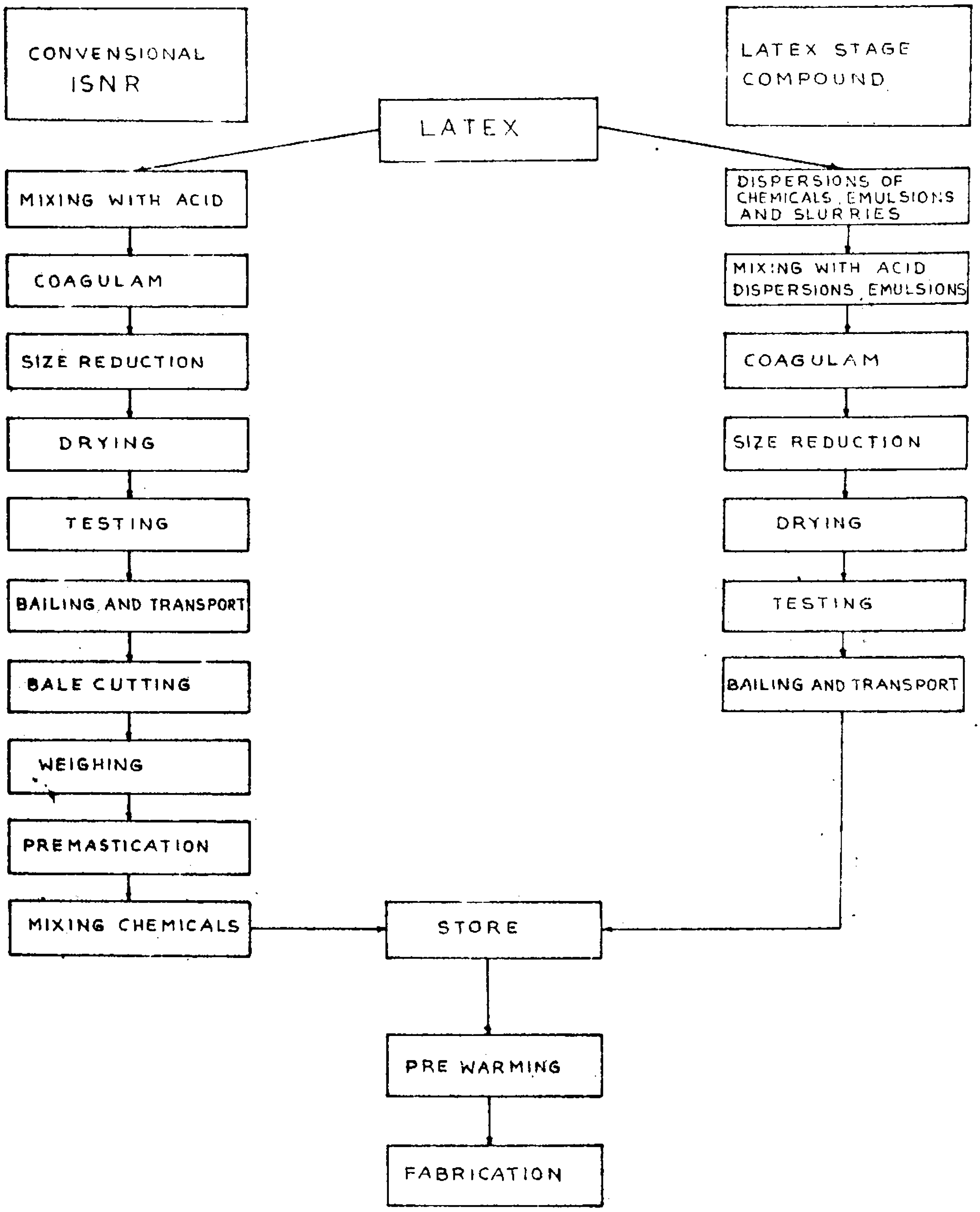
LATEX STAGE COMPOUNDING AS AN ENERGY SAVING METHOD IN NATURAL RUBBER PROCESSING

CHAPTER - VI

LATEX STAGE COMPOUNDING AS AN ENERGY SAVING METHOD IN NATURAL RUBBER PROCESSING

Preparation of partial compounds from latex is known in the rubber industry for over the past thirty years. Many early patents[1-4] describe the preparation of partial or complete compounds of natural rubber by latex stage mixing. The first commercial exploitation of this process was by Wilkinson[2] in 1926. The number of operations involved in using latex stage compounds in a processing factory is much less when compared to that needed in the case of conventional forms of rubber. (See Chart VI.1) Further more, considerable saving in energy is also possible. In earlier reports[3,4] the processing and energy advantages of latex stage compounds were not investigated. The main aspects examined in the present study are (1) Production of latex stage compounds from viscosity stabilised latices and latices containing peptisers, (2) Examining the processing advantages, if any and the extent of energy saving by using latex stage compounds, (3) Determining the efficiency of different retarders in controlling the scorch safety of latex stage compounds (4) The effect of withholding curatives as a means of controlling scorch tendency of latex stage compounds.

CHART VI.1 COMPARISON OF STEPS IN PRODUCTION AND CONSUMPTION OF LATEX STAGE COMPOUNDS WITH CONVENSIONAL FORMS OF RUBBER



In the first part of the study ordinary field latex was admixed with various compounding ingredients as per standard formula given in Table-1. The product was processed as crumb rubber at the Pilot Crumb Rubber Factory of the RRII, dried and evaluated for the various technological properties. Results are given in the same table. In the second part of the study viscosity stabilised latex, latex containing peptisers or latex containing mineral rubber process oil were used for mixing with compounding ingredients. The concentration of the ingredients used for viscosity reduction of rubber present in latex are as shown. (a) Hydroxylamine hydrochloride at a concentration of 0.15 percent on drc of latex, (b) peptiser at 0.1 percent on drc of latex, (c) Mineral plasticizer oil at 10.0 phr. The latex samples which were thus separately treated for viscosity reduction of rubber are then used in producing three different latex stage compounds in accordance with the formula given in Table - II. Results of evaluations are given in Table-III. In the final part latex stage compounds were prepared from viscosity stabilised latices with incorporation of retarders. Properties of latex stage compounds containing any of the retarders, benzonic acid, salicyclic acid or vulcalent A, are given in Table IV and those for - retarder Santoguard PVI-50 (N-cyclohexyl thio pthalimide) are given in Table V. The concentration of PVI-50 was varied from 0.25 to 1.0 phr. Latex stage compounds were prepared with different concentrations of accelerators to study its effect on scorch tendency. In another study all compounding ingredients other than accelerators were added to the latex to prepare latex stage compounds. Details

TABLE - I

FORMULATIONS AND PROPERTIES OF VARIOUS COMPOUNDS

Formulations	I Dry	II Dry	III Dry
Rubber - RSS - 1	100	-	100
Rubber (as latex)	-	100	-
Zinc Oxide	5	5	5
Stearic acid	2	2	2
PBN	1	1	1
China Clay	150	150	150
Naphthenic process oil	5	5	5
MBTS	0.8	0.8	0.8
DPG	0.2	0.2	0.2
Sulphur	2.5	2.5	2.5
Ethylene glycol	...	...	1.5
Properties:			
Mooney Viscosity	85	88	80
Scorch time (Minutes) 121°C	19.0	7.5	13.0
Cure time (") 150°C	14.0	6.0	13.0
Tensile strength (Mpa)	10.42	14.84	12.64
Modulus at 300% (MPa)	8.72	12.45	8.23
Elongation at break (percentage)	338	352	400
Compression set (%)	57.0	42.0	52.0
Hardness (Shore A)	57.0	62.0	60
Resilience (%)	49.0	53.0	49.0
Tear strength (KN/M)	36.26	46.06	38.22

TABLE - II

FORMULATIONS USED IN THE STUDY

Natural rubber[1]	-	100
Zinc Oxide	-	5
Stearic acid	-	2
PBN	-	1
China clay	-	150
Naphthenic Rubber Process oil	-	5
CBS[2]	-	(Variable)
Retarder[3]	-	(Variable)

1. Following treatments were given to NR latex used in the study.
   a) Control with no treatment.
   b) Viscosity stabilised latex containing 0.15 percent hydroxylamine hydrochloride on d.r.c
   c) Latex containing peptiser
   d) Latex with minor rubber process oil.
2. CBS was used in five different concentrations, viz. 0.25, 0.5, 0.75, 1.0 and 1.25 phr.
3. Four different retarders were used, viz. Benzoic acid, salicyclic acid, Vulcalent-A and Santoguard PVI-50. The selected PVI-50 was evaluated in four concentrations to fix the optimum.

of the formulations used for these studies are also included in Table - II. Energy required for compounding rubber was assessed using the method described in Chapter-II. About ten kilogram of latex stage compound was used for the study. The technological properties were investigated using methods described in Chapter - II.

RESULTS AND DISCUSSION

The data given in Table-I show that the mill mixed compound 1 prepared without ethylene glycol shows a slightly extended cure time, than compound 3 containing ethylene glycol. This is explainable because ethylene glycol is known to reduce accelerator adsorption on fillers. The latex stage compound 2 did not contain this additive. Still it did not show any problem of extended cure time in spite of the heavy china clay loading. However, it is desirable to use a slow curing and safe accelerator like CBS for the production of latex stage compound.

It is seen that the Mooney viscosity of latex stage compounds prepared from ordinary latices is very high and energy saving in processing cannot be achieved with such materials. An examination of the properties of latex stage compounds prepared from latices pretreated for visocisty reduction and control (Table-III) shows that the use of hydroxylamine hydrochloride or peptiser or mineral plasticiser oil, is helpful in producing latex stage compounds

TABLE - III

PROPERTIES OF LATEX STAGE COMPOUNDS PREPARED FROM LATICES PRE-TREATED FOR VISCOSITY MODIFICATION OF THE RUBBER

Properties	Treatments		
	Hydroxylamine treated upto 0.15 percent	Peptiser treated 0.1 phr Petachloro Thiophenol	Latex containing mineral oil
1. Mooney Viscosity (ML[1+4]100°C)	54	32	43
2. Scorch at 121°C (in mnts.)	25.5	..	27.2
3. Cure time at 150°C (in minutes)	15.0	25.5	16.0
4. Hardness Shore A	53.0	50.0	46.0
5. Abrasion loss cc/hr	3.1	3.7	3.9
6. Resilience (%)	55.0	57.0	56.0
7. Compression set (%)	47.0	..	47.0
8. Tear Strength KN/M	39.2	37.24	25.28
9. Tensile strength MPa	11.86	12.54	11.27
10. Elongation at break (%)	256.0	290	288.0
11. Percentage Retention of Tensile Strength (Ageing at 70°C for 96 hrs.)	104.0	83.5	68.4

of easily workable Mooney Viscosity of 60 ± 5. The physical properties of the latex stage compounds are marginally improved when it is prepared using viscosity stabilised latices. Peptiser incorporation or use of mineral oil, gave products having properties comparable to those of control compounds prepared by mill mixing.

Latex stage compounds do show a slight scorch tendency. In order to overcome this, addition of retarders was tried. It is seen that the three common retarders used in the study are not helpful in reducing the tendency to scorch, shown by latex stage compounds. A possible reason for this is their ineffectiveness in presence of moisture. However, Santoguard PVI-50, a retarder known to be active in presence of moisture is found to be quite effective. Optimum concentration of this chemical for getting desirable scorch resistance to latex stage compounds was found to be 0.5 phr. It may also be seen that the other physical properties are not affected by addition of this chemical, as is clear from Table-V. The recommended formulation taking all aspects into consideration is NR:100; ZnO:5; Stearic acid:2; PBN:1, China clay 150; Naphthenic Rubber process oil:5; CBS:1; Santoguard PVI-50:0.5.

An examination of the data in Table-VI shows that reducing the concentration of accelerator in production of latex stage compounds is not very useful in controlling

TABLE - IV

PROPERTIES OF LATEX STAGE COMPOUNDS PREPARED WITH INCORPORATION OF RETARDERS

Properties	Treatments				
	Latex stage compounds with retarder Benzoic acid	Latex stage compounds with retarder Salicilic acid.	Latex stage compounds with retarder Vulcalent A	Latex stage compounds in which sulphur added in mill	Dry mill mixed compound as (Control)
Mooney Viscosity ML (1+4) 100°C	55.0	54.0	59.0	46.0	...
Cure time in minutes at 150°C	7.0	8.0	10.0	9.0	20.5
Scorch time in minutes at 120°C	13.3	16.25	28.25	22.0	52.25
Cure rate index	33.3	28.6	22.2	25.0	4.17
Hardness (Shore A)	..	..	55.0	54.0	55.0
Resilience (%)	..	..	54	56.0	59.0
Abrasion resistance volume loss (cc/hr)	..	..	4.2	5.1	3.9
Tensile strength MPa	..	..	11.46	11.31	9.49
Percentage Retention of Tensile strength Ageing 70°C 96 hrs.	..	..	103.6	104.0	85.00

the scorch tendency. Rheograph of latex stage compounds in which accelerator addition was effected in mixing mill is given in fig. VI.1. It may be seen from the figure that the scorch tendency of latex stage compounds can be minimised by this method. All other ingredients including sulphur was added at the latex stage. Graph 2 gives the cure behaviour of the latex stage compound obtained in this manner. Its cure property is comparable to the mill mixed control sample. The modulus values appears to be better for this compound than the mill mixed sample.

The extent of energy saving possible in latex stage compounding was assessed by studies in an open 30" two roll mixing mill. Results of the study may be seen in Table-VII. It may be seen from the data that this method of processing has a definite advantage in that, its use in consumer's factory will lead to appreciable saving in energy. The study showed that an energy saving of upto 80 percent was possible at the stage of mixing and compound preparation if latex stage compounds were used in place of conventional sheet or crepe rubbers.

CONCLUSION

1. Latex stage compounds prepared from viscosity stabilised latices or latices containing selected peptisers can contribute to appreciable energy saving at the mixing and compounding stage in consumers' factory.

FIG. VI.1 VARIATION IN CURING CHARACTERISTICS OF LATEX STAGE COMPOUNDS WITH ORDER OF MIXING ACCELERATORS

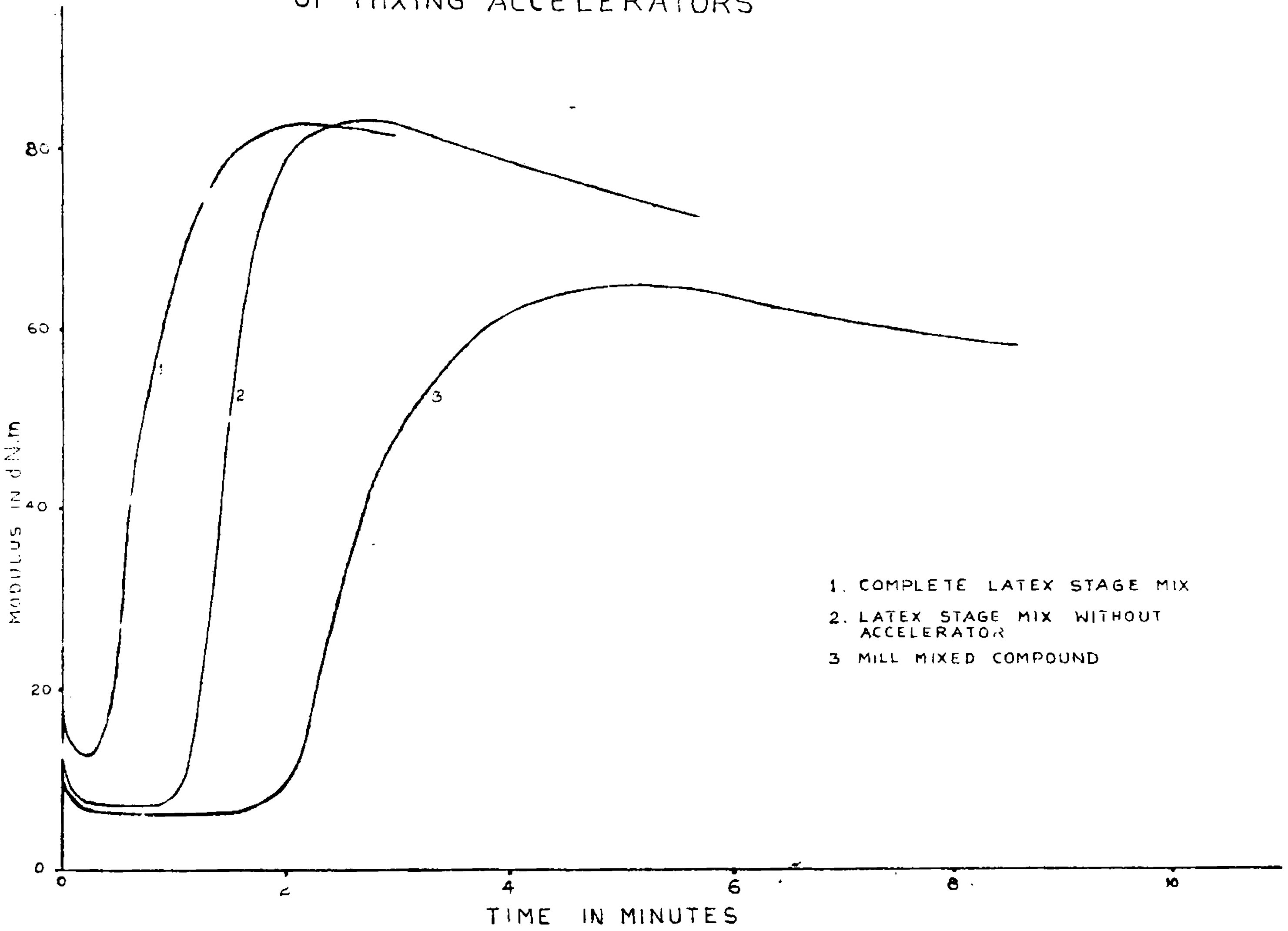


TABLE - V

PROPERTIES OF LATEX STAGE COMPOUNDS PREPARED WITH DIFFERENT CONCENTRATIONS OF RETARDER - SANTOGUARD - PVI-50

Properties	Concentration of Santoguard PVI-50 in Phr.				Control
	0.25	0.5	0.75	1.0	Mill mixed sample
Mooney Viscosity ML (1+4) 100°C	32	34	39	34	23.0
Scorch time in Mins. at 120°C	16.75	24.25	35.75	43.75	43.0
Scorch time at 150°C in Mins.	5.0	6.0	7.5	9.0	9.0
Cure time in Mins. at 150°C	8.5	9.5	11.0	13.0	15.0

TABLE - VI

PROPERTIES OF LATEX STAGE COMPOUNDS CONTAINING VARYING LEVELS OF ACCELERATOR

	Concentration of CBS (accelerator in Phr)					Control
	0.25	0.5	0.75	1.0	1.25	Dry mixed 1.0 Phr
Mooney Viscosity ML(1+4) 100°C	48	40	47	43	39	...
Mixing time in Mins.	4.45	4.0	4.45	4.0	4.25	44.0
Scorch time in Mins. 121°C	14.25	12.25	11.8	12.25	10.0	45.0
Cure time at 150°C in Mins.	16.0	13.5	12.0	10.0	6.0	14.0
Tensile strength MPa	9.72	8.47	11.4	11.03	9.36	12.11
Elongation at break %	244	250	220	234	226	243.0
Tear strength KN/M	38.71	37.44	46.65	46.65	44.98	42.92
Abrasion loss cc/hr	3.7	3.1	2.41	2.2	1.8	2.2
Resilience (Percentage)	53.5	50.6	51.5	52.20	53.5	64.6
Hardness (Shore A)	51.0	59	66	62	60	65.0

2. The use of Santoguard PVI (N-cyclohexyl thiopthalmide) is helpful in reducing scorch tendency of latex stage compounds.

3. Addition of accelerators in prewarming stage in open mills also helps in controlling scorch.

4. Addition of glycols to rubber compounds containing silica or silicates to prevent accelerator adsorption on fillers can be avoided if latex stage compounding procedure is used.

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CHAPTER - VII

SUMMARY AND CONCLUSIONS

CHAPTER - VII

SUMMARY AND CONCLUSIONS

Four different methods of processing natural rubber in energy saving forms were described in Chapters III to VI. The methods are summarised in Chart - 7.1.

It may be seen from the chart that the new processes involve some additional steps and consequent enhancement in cost of processing. However, this is more than offset by the considerable savings in energy. The Mooney Viscosity of the four modified forms of rubber and that or the control are given in Fig.7.1. The energy saving possible under the processes is shown in Fig.7.2. The advantages of the modified forms in relation to conventional form of rubber is clear from the two histograms. The adoption of processing procedures of the type described here by the natural rubber producing industry will improve the competitive position of natural rubber in relation to general purpose synthetic rubbers.

Suggested procedure for adoption of the processes among Indian Rubber Plantations.

India at present produces around 1,75,280 tonnes of natural rubber. The different forms in which these are processed may be seen from the following statistics.

CHART VII.1 STEPS INVOLVED IN THE PRODUCTION OF NEW FORMS OF PROCESSED NATURAL RUBBER

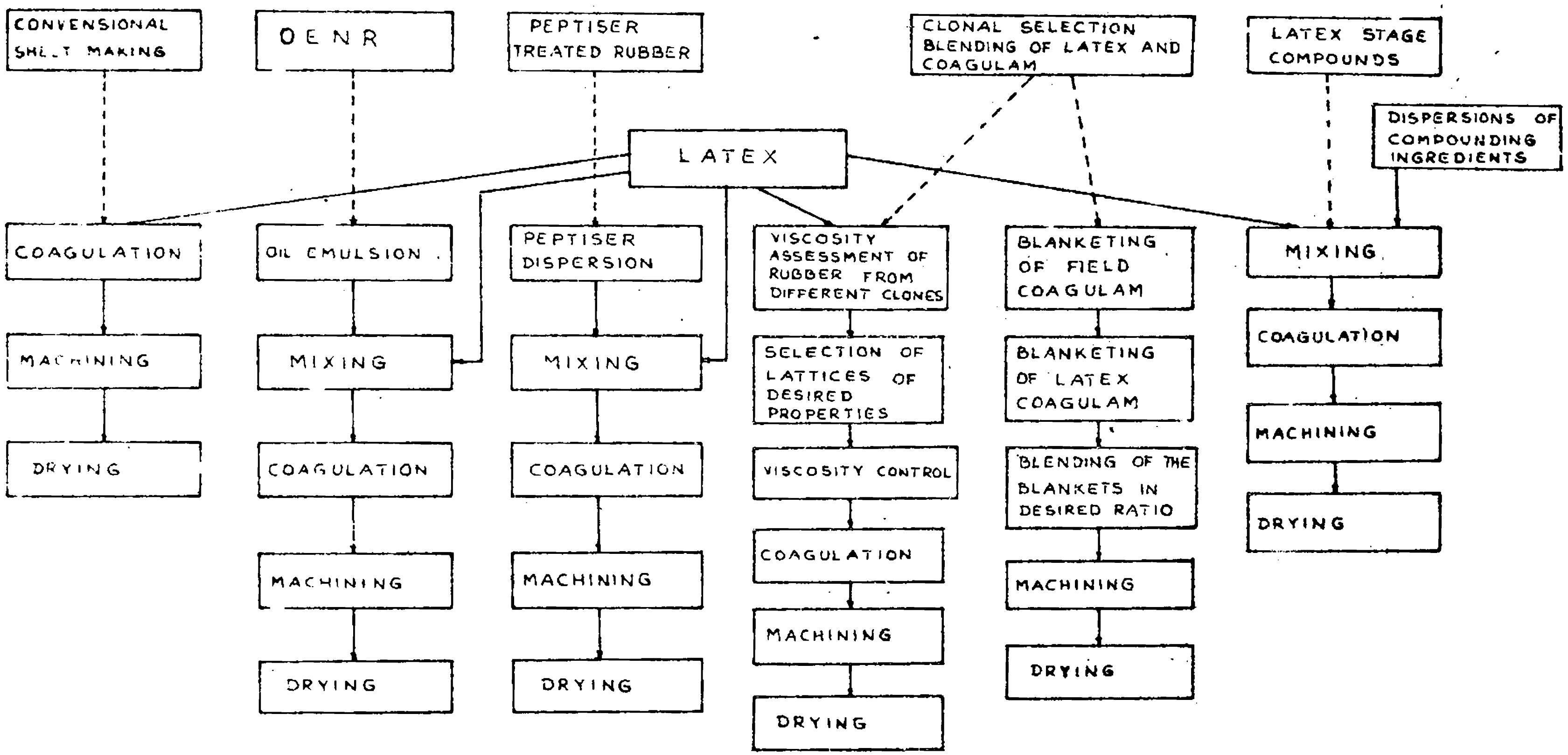


FIG.VII.1 MOONEY VISCOSITY OF PROCESSED RUBBER $M_L(1+4)\,100^\circ C$

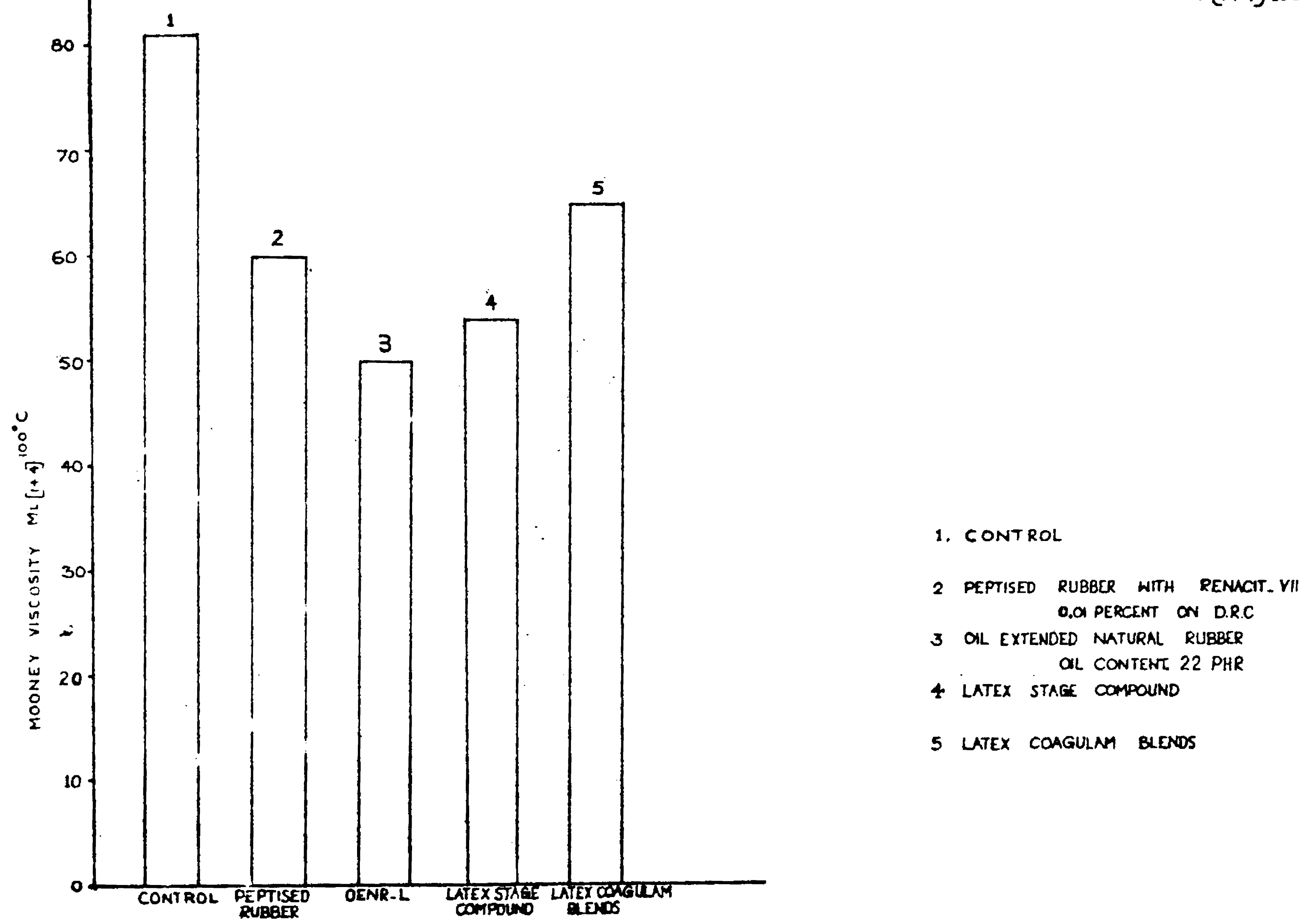
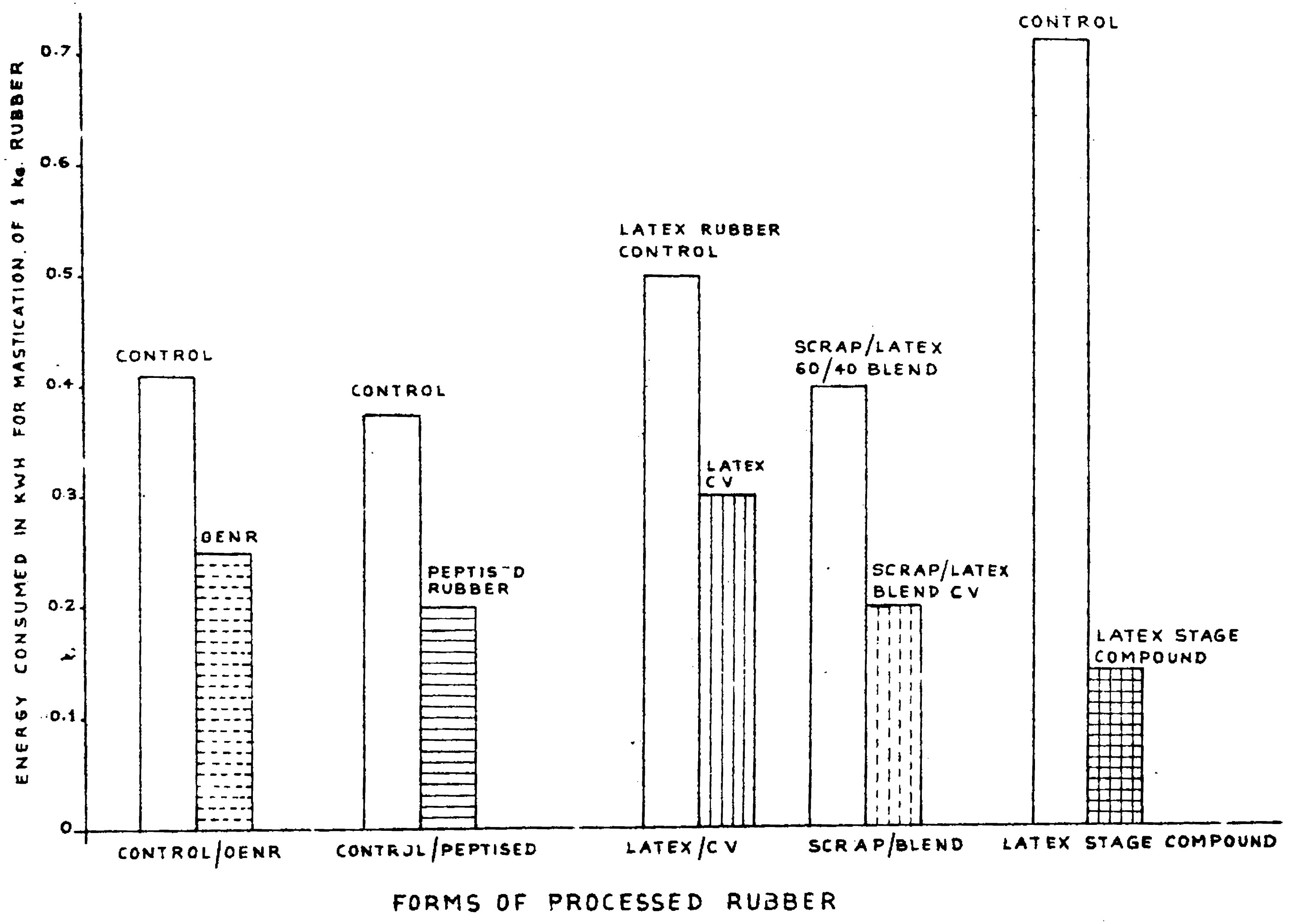


Fig.II ENERGY SAVING OF THE PROCESSED RUBBERS



	Forms of processed rubber	Quantity in tonnes during 1983'84
1.	RMA sheets	121565.00
2.	Latex concentrate	16930.00
3.	Solid block rubber	2919.00
4.	Pale latex crepe	2180.00
5.	Others	31686.00
	Total	175280.00

Item 5 includes mostly estate brown crepes, processed from field coagulam. The use pattern of rubber among different consuming industries will have to be examined before a procedure for processing raw material in tailor made forms is discussed. Details are given below:

	Products	Consumption of NR in tonnes during 1982-83
1.	Automobile tyres and tubes	98879.00
2.	Cycle tyres and tubes	23575.00
3.	Camel back	10048.00
4.	Foot wears	21059.00
5.	Belts and hoses	13076.00
6.	Battery boxes	615.00
7.	Cables and wires	769.00
8.	Latex foam	7645.00
9.	Dipped goods	6084.00
10.	Others	13795.00
	Total	195545.00

The properties and processing behaviour of individual forms of processed rubbers and the possible quantity that can be produced under each type is described below:

1. Oil Extended Natural Rubber

This is a type of natural rubber which can be used for passenger car tyre production. In most Indian tyre factories SBR-1712, the oil extended form of styrene butadiene rubber is used. The exact quantity used by our tyre industries is not known. SBR-1712 is available from the factory of M/s Synthetics and Chemicals at Bareli, Utter Pradesh. However, there are definite advantages to the consumers by using oil extended natural rubber in place of SBR-1712, as reported in the studies of Grosch and Co-workers (Chapter-III). The investigations in the present study also confirmed this. There is scope for production of around 7,500 tonnes of oil extended natural rubber in this country. A possible specification for the oil extended natural rubber is formulated from consideration of the studies in Chapter-III and given in Table - 7.1. It is preferable to market this type of rubber under a trade name in line with the procedure followed by the synthetic rubber manufacturing industry. A name that can be adopted is OENR-L.

2. Peptised Natural Rubber

This process produces natural rubber in the viscosity range of 55-65 (ML(1+4) @ 100°C). It may be noted that

TABLE - VII.1

Specifications for Oil Extended Natural Rubber
(Oil content 25 Phr)

Properties		Limits
1. Dirt content percent	..	0.05
2. Ash content	..	0.50
3. Volatile matter	..	0.8
4. Mooney Viscosity ML(1+4)100°C	..	55±5
5. PRI (Minimum)	..	60
6. Acetone extract percent. max.	..	28.0
7. Nitrogen content	..	0.60

---

peptisers are used in compounding dry forms of processed rubber like RMA sheets, solid block rubber or EBC grades. However, the advantage of the present procedure is that the lowering of viscosity can be brought about using only 5 to 10 percent of the peptiser required in dry stage operations. The rubber thus obtained can be used with advantage by the following sectors of the industry.

1. Automobile tyres and tubes
2. Camel back
3. Cycle tyres
4. Moulded goods
5. Mouldings, lining compounds etc.

It is believed that a good percentage of sheet rubber produced in this country can be processed with incorporation of peptisers. The solid block rubber processing factories being set up under co-operative societies with World Bank assistance can also use part of their capacity for producing this type of rubber. The resultant product can be marketed as rubber of reduced viscosity. The viscosity of this type of rubber falls in the same range as that of pre-masticated rubber. RRII was receiving pre-masticated rubbers for testing from various tyre companies, who were procuring masticated rubber from small industries in Kerala. Monthwise variation of the viscosity of such rubber along with that of the peptised rubbers prepared according to the procedure standardised in

Chapter-IV is given in Table-II. The data clearly show that peptised rubbers can be used with advantage by the rubber consuming industry in place of masticated rubbers. The energy saving possible by this is already discussed in Chapter IV. By slow and gradual propagation among the small and medium farming community, it is possible to bring a substantial level of the rubber production under this type. The rubber so processed may also be marketed under a trade name PNR-55-65. Around 40 to 50 thousand tonnes of rubber can be processed in this form in the course of five to six years.

3. Latex Stage Compounds.

The advantage of this type of processed rubber is that it helps the consumers by ensuring supply of a complete compound from a single source. All ingredients used in product manufacture are added to latex and the resultant mixture is processed as a ready to use compound. The findings of the study show that it is desirable to admix accelerators with rubber compound in the mixing mill. The addition of all other compounding ingredients to rubber was done in latex stage.

This type of processed rubber can be used by the following sectors of consuming industry.

1. Foot wear
2. Moulded products
3. Extruded products

TABLE - II

MOONEY VISCOSITY ML(1+4)100°C MASTICATED RUBBERS

Month of testing	Mooney Viscosity ML(1+4)@100°C of Masticated Rubber	Mooney Viscosity ML(1+4)@100°C of Peptised Rubber.
July 1983	56.0	60.0 ± 5
August, 1983	55.0	
September 1983	62.0	
October 1983	52.0	
November 1983	61.0	
December 1983	59.0	
January 1984	56.0	
February 1984	53.0	
March 1984	63.0	
April 1984	62.0	

The consuming industry using such a compound has to come to some sort of understanding with the producing industry for the easy acceptance of this type of processed products.

It is realistic to assume that around 30 percent of the present consumption of rubber in foot wear industry can be diverted to this type of product in the course of three to five years through proper compaign. Similar efforts among moulded and extruded product manufacturers also can lead to further populatisation of this product. Efforts of this nature are fully justified as the product provides substantial energy saving at the mixing and compounding stages. The trade name suggested for this product is NR-LMB.

4. Selection of clones yielding rubber of low viscosity.

There are two interesting findings under this study. It is shown that the latices from those clones which yield rubber of medium Mooney Viscosity can be processed as energy saving forms of rubbers by giving suitable treatments. The clones that yield medium viscosity rubber are the following:

RRIM - 600
RRII - 105
GT - 1

These are high yielding clones. A sizable area under the new planting and replanting programmes organized

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RRIM - 600
RRII - 105
GT - 1

These are high yielding clones. A sizable area under the new planting and replanting programmes organized by this country is covered by one or the other of these clones. So it is easily possible for the Indian Plantation Industry to produce rubber of medium Mooney Viscosity if viscosity stabilisation procedure is adopted in an organised way among the plantations. This type of rubber can be treated at par with the peptised rubbers produced from latices. The same end uses as that for peptised rubbers can be envisaged for this type of rubber also. It is also likely that the peptised rubbers will find better acceptability among consumers than this grade of rubber. So a possible production of this type of rubber is not estimated.

4.1 Blending of latex and field coagula.

Blending procedure for latex and field coagulam has been standardised. The process is important in that it gives a method of up-grading the low priced coagula rubber which account for around 20 percent of the crop from rubber plantations. It is possible to assume that around 20 to 25 thousand tonnes per annum production of this grade of rubber is possible in this country. The quantity is estimated in the lower side as it is not possible to eradicate the conventional forms of processing overnight. Total coagulam material available at present is around 40 thousand tonnes. Around fourty percent of the coagulam is expected to be diverted to the new type of processing.

These blends are suitable in making treads of heavy duty tyres. They can also be used as base rubber in products, like cycle tyres, camel back, moulded goods, hoses etc. The suggested name for this blend is NR L/C-60.

The different forms of processed rubbers, their suggested names and estimated production are given in the table below.

	Nature of the product	Suggested name	Estimated production in tonnes/ year by 1989-90
1.	Oil extended natural rubber	OENR-L	7,500
2.	Peptised natural rubber	PNR-55-65	50,000
3.	Latex stage compounds	NR-LMB	...
4.	Latex coagulam blends	NR L/C - 60	25,000

Quantity estimation is not included under item 4, since a reasonable projection is difficult with the information available.

LIST OF ABBREVATIONS USED IN THIS THESIS

NR	Natural rubber
OENR	Oil extended natural rubber
OENR - L	Oil extended natural rubber prepared from latex.
RMA	Rubber Manufacturers' Association
RSS	Ribbed Smoked Sheet
EBC	Estate brown crepe
CV	Constant viscosity rubber
PNR 60 ± 5	Natural rubber prepared in the Mooney Viscosity range of 60±5 with incorporation of peptiser in latex stage.
NR - LMB	Natural rubber latex stage compound
NR L/C 60	Natural rubber latex and field coagulam blend with field coagulam content of 60 parts and latex coagulam content of 40 parts.
PLC	Pale latex crepe rubber
SBR	Styrene buta diene Synthetic rubber
CBS	Cyclo hexyl benz thiazyl sulphenamide
MOR	Morpholino benz thiazyl sulphenamide
MBTS	Mercapto benz thiazyl di sulphide
DPG	Diphenyl Guanidine
PBN	Phenyl β naphthyl amine
SP	Styrenated phenol
Santoguard PVI	N-cyclo hexyl thio thalimide
RPA - 3	Xylyl Mercaptan

Renacit - 7	..	Pentachloro thio phenol with additives
HAF Black	..	High abrasion furnace black
PRI	..	Plasticity Retention Index
VGC	..	Viscosity Gravity Constant
$ML\ (1+4)^{100^\circ C}$	..	Mooney viscosity determined using large rotor after a dwell time of one minute and motor run of 4 minutes at 100°C.
Po	..	Wallace plasticity
ASHT	..	Accelerated storage hardening test
SMR	..	Standard Malaysian Rubber
RRII	..	Rubber Research Institute of India
RRIM	..	Rubber Research Institute of Malaysia.

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LIST OF PUBLICATIONS ARISING OUT OF THIS THESIS

1. Oil extended natural rubber from latex, Journal of Polymer Materials (In press).

2. Latex stage compounding, an energy saving method in natural rubber processing. Plastics and Rubber processing and applications. (accepted for publication).

3. Processing of natural rubber in energy saving forms, with incorporation of peptisers. (under publication).

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