

Oil Extension of Natural Rubber at Latex Stage

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A method is described for the manufacture of a compact oil-extended natural rubber masterbatch (OENRM) containing 75 parts of rubber and 25 parts of an inexpensive process oil. The rubber, which is of high technical quality, has both excellent shelf life and processing characteristics.

Inexpensive and commercially available rubber process oils can be simply added to natural rubber latex, either directly with high-speed stirring or in the form of a prior emulsion. The mixture when coagulated, milled and dried yields oil-extended natural rubber masterbatch (OENRM). In recent years (GURNEY, 1958; SEKHAR, 1965), the manufacture of such a rubber at the plantations met with qualified success mainly because of difficulties during drying and compaction when conventional procedures were employed. Manufacture of a compact masterbatch was resumed with the adoption of new presentation methods for converting the latex-oil coagulum to fast-drying crumb within a short period and for block baling and technical specification of dried crumb.

This paper describes a method of producing from latex an oil-extended rubber masterbatch containing up to 70 parts by weight of rubber and 30 parts by weight of oil, using the Heveacrub process (SEKHAR *et al.*, 1965). It also presents data obtained from laboratory evaluations in a 'Size K Intermix' involving:

- (a) a wide range of oil and carbon black levels;
- (b) the effect of the variation of curative levels;
- (c) a range of semi-reinforcing and reinforcing carbon blacks; and
- (d) a comparison of the masterbatch with dry-mixed compound.

Terminology

Natural rubber latex masterbatched with 75 parts by weight of rubber and 25 parts by weight of a naphthenic type of process oil and manufactured by the Heveacrub process has been provisionally called Heveacrub oil-extended natural rubber masterbatch (abbreviated Heveacrub OENRM). Masterbatches containing other oil levels are described appropriately.

METHOD OF MASTERBATCHING PROCESS OIL WITH NR LATEX

The process oil (as an emulsion) is blended with latex in the presence of a small amount of a crumbling agent and the mixture is co-precipitated with acid. The coagulum is converted to fine crumb by milling and is then dried at high temperature and compacted into bales. The details of the method are described below.

Preparation of Oil Emulsion

To 100 parts by weight of a process oil are added 40 parts by weight of water for an approximately 71.4 per cent aqueous emulsion and the mixture is agitated by a high-speed mixer. This is emulsified by the addition of 1 per cent oleic acid (based on oil) with about 0.05 per cent ammonia. Stable emulsions of aromatic, naphthenic and paraffinic process oils are successfully prepared by this method, e.g., Sundex 8125*, Circosol 4240*, Sun Par 150*, Dutrex 63†, Dutrex 66† and Flexon 766‡.

* Sun Oil Corporation, U.S.A

† Shell Company, Malaysia

‡ Esso Company, Malaysia

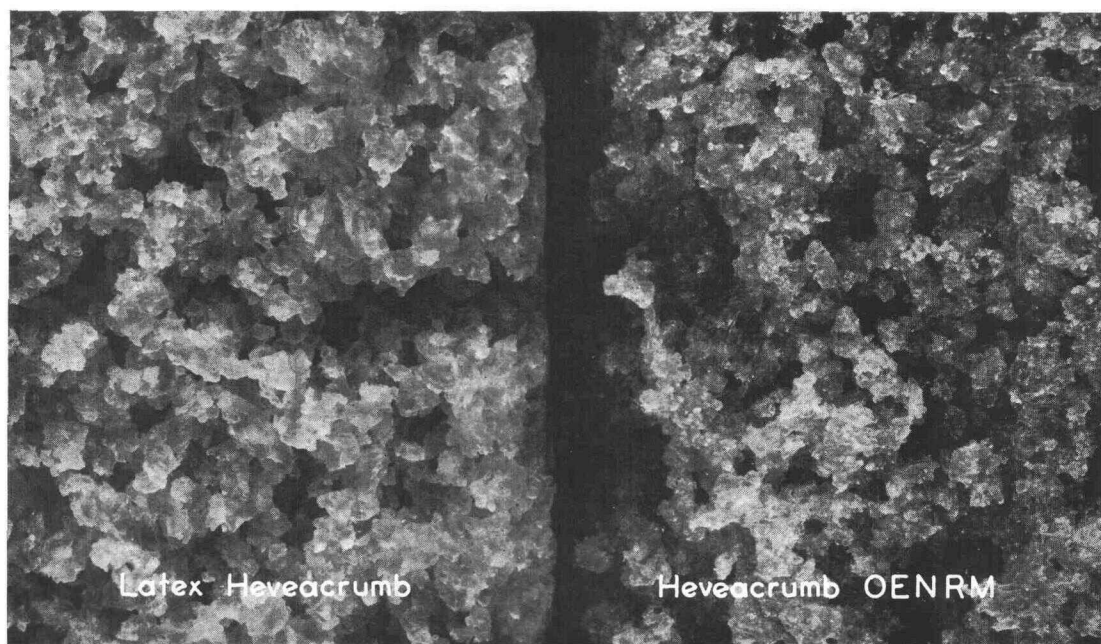


Figure 1. Comparison of the crumb from latex Heveacrumb and OENRM.

Latex

Lightly ammoniated undiluted field latex is used and it is properly sieved and bulked for uniform production. Ammoniation is required to maintain sufficient latex stability for a rapid d.r.c. to be determined, but it is kept at the minimum necessary, usually 0.05 per cent based on the weight of latex.

Crumbling Agent

About 1.33 per cent of castor oil or about 1.07 per cent of a castor oil-zinc stearate mixture based on the rubber content is used for crumbling and temporarily prevent the crumb from re-agglomerating during manual handling.

Blending of Masterbatch Ingredients

The crumbling agent is thoroughly dispersed into the lightly ammoniated latex. While the mixture is kept well stirred, the required amount of the oil emulsion is added to the latex which is then coagulated with 5 per cent formic acid to pH 5.

Treatment of Coagulum

Handling the wet coagulum after 8 hours' coagulation presents no difficulty. The coagulum is crumbled by passing through a series of crepeing rolls and the crumb which then results is similar in size to Heveacrumb containing no process oil (Figure 1).

Drying. The crumb is loaded into drying trays and dried at 100°C by a forced air-draught circulation at atmospheric pressure. A typical drying curve for a 4-inch depth loading in an experimental drier (SETHU, 1967) is shown in Figure 2. For comparison purposes, results of ordinary crumb without any process oil but produced from the same latex and dried at similar depths are also presented. The oil-extended crumb shows an initial moisture content of 62% compared to 38% for the un-extended crumb but, despite being subject to more plastic flow, a fairly short drying time is obtained (SETHU, 1968). However, on a commercial scale where the efficiency of drying is

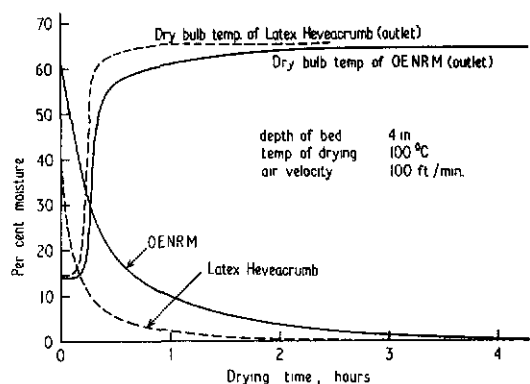


Figure 2. Drying curves for latex Heveacrumb and OENRM.

considerably less, the oil-extended crumb takes longer to dry.

Baling. Baling is carried out at a pressure and temperature slightly lower than for unextended crumb. Each bale is wrapped in polythene, labelled, placed in a multiwall paper bag and palletised.

Uniformity of production. Each production lot is carefully controlled at the factory by

using standardised methods of latex collection from characterised sources and adequate bulking and mixing facilities. Accuracy in the determination of the rapid d.r.c. of the latex as well as other volume measurements are also constantly maintained throughout. Although OENRM is a speciality rubber and is not marketed at present as Standard Malaysian Rubber, it is produced and tested to SMR specifications wherever applicable. In addition, each production lot is thoroughly checked for its acetone extract (indicating oil dispersion) and for its Mooney viscosity (indicating the efficiency of latex blending).

From experience with other rubbers, especially CV, OENRM manufactured from latex at different Malaysian factories is expected to have the following range of Mooney viscosity and acetone extract values:

Mooney viscosity, ML 1+4'	
at 100°C	30 to 45
Acetone extract, % wt	27 to 31

The wide range of these values is due to the multiplicity of latex sources at the different factories. Each of the latices has its own

TABLE 1. RESULTS OF SAMPLES TAKEN FROM ONE PRODUCTION LOT OF HEVEACRUMB OENRM

Sample No.	Acetone extract % wt	Mooney viscosity ML1+4' at 100°C	TC strain %	SMR tests						
				Dirt % wt	PRI % wt	Volatile matter % wt	Ash % wt	Nitrogen % wt	Copper p.p.m.	Manganese p.p.m.
1	27.97	34.5	142.0	0.010	93	0.25	0.30	0.34	1.0	1.2
2	27.14	34	147.8	0.010	94	0.34	0.24	0.35	1.0	1.8
3	27.29	36.5	141.0	0.020	88	0.33	0.34	0.36	1.0	1.8
4	28.10	34.5	143.4	0.019	97	0.41	0.39	0.34	1.0	1.0
5	27.81	35.5	141.0	0.010	97	0.33	0.38	0.33	1.0	1.0
6	28.19	34.5	138.6	0.006	88	0.32	0.25	0.31	1.0	1.7
7	27.89	37	134.8	0.012	85	0.30	0.25	0.32	1.0	2.2
8	27.99	37.5	140.2	0.014	89	0.31	0.25	0.30	1.0	1.8
9	28.32	37.5	143.5	0.005	86	0.45	0.25	0.31	1.0	2.4

characteristic value of viscosity and acetone extractable. For any given factory, however, the properties in practice fall within a much narrower range as illustrated in *Table 1*. These results were obtained from one typical production lot at the R.R.I.M. Experiment Station.

LABORATORY EVALUATIONS

Choice of Oil Type

Petroleum process oils are commercially divided into three main groups: aromatic, naphthenic and paraffinic depending on the hydrocarbon composition. Examples of each

TABLE 2. PHYSICAL PROPERTIES OBTAINED FROM OENRM WITH DIFFERENT OILS

Property	Oil (in order of decreasing aromatic nature)			
	Sundex 8125 (Aromatic)	Circosol 4240 (Naphthenic)	Dutrex 63 (Naphthenic)	Sun Par 150 (Paraffinic)
Saybolt viscosity at 100°C, S.U.S.	126	83	42	63
Specific gravity g/ml at 20°C	0.99	0.95	0.89	0.88
Viscosity gravity constant	0.946	0.890	0.835	0.805
Tensile strength, kg/cm ²	189	195	200	195
Elongation at break, %	571	565	559	561
Modulus at 300%, kg/cm ²	86	92	95	89
Hardness, IRHD	66.3	65.3	66.4	63.6
Tear, kg	27.0	25.0	26.8	25.0
Dunlop resilience, 21°C, %	53.0	55.0	58.0	56.0
De Mattia flex-cracking resistance, kc to Grade C	150	145	140	130
De Mattia cut growth resistance, kc to 4-8 mm	37.0	50.0	34.3	32.0
Goodrich flexometer heat build-up, °C	124	123	116	120
Dynamic compression set, %	44.0	44.0	29.4	37.0
Akron abrasion volume loss, cc/1000 rev	0.068	0.073	0.077	0.079
Viscosity of compound, ML 1+4' at 100°C	30	35	37	34½
Percentage retention on oven-ageing at 70°C for 14 days				
Tensile strength, kg/cm ²	88	86	77	85
Elongation at break, %	74	69	62	70
Modulus at 300%, kg/cm ²	142	139	142	138

Formulation:

Rubber + oil	100	Stearic acid	2
HAF black	50	Santocure CBS	0.1
Zinc oxide	5	PBN	1.0
		Sulphur	2.5

Mill-mixed in a 10-minute cycle and cured for 40 minutes at 140°C.

TABLE 3. MOONEY VISCOSITY RETENTION ON OVEN-AGEING OF DIFFERENT MASTERBATCHES CONTAINING 75 PARTS RUBBER AND 25 PARTS OIL

Oil	Percentage retention of Mooney viscosity on ageing time at 70°C, days						
	2	7	14	21	28	35	42
Sundex 8125 (aromatic)	93	90	75	55	41	fluid	fluid
Circosol 4240 (naphthenic)	98	84	66	59	43	35	fluid
Dutrex 63 (naphthenic)	108	107	95	82	76	66	62
Sun Par 150 (paraffinic)	104	102	97	84	78	67	63

group were studied with 75 parts by weight of rubber and 25 parts by weight of oil for each masterbatch. As shown in Table 2, the variation of the type of oil has no significant effect on the tensile properties of the unaged and oven-aged vulcanisates. In going from an aromatic oil to a paraffinic one, there is a marginal increase in resilience and a slight decrease in flex-cracking resistance as well as lower heat build-up. The laboratory abrasion loss for all four masterbatches is not distinguishable. In general, these results confirm that the petroleum oils recommended for oil extension are suitable showing little differences in the properties of the vulcanisates.

The shelf life of the unvulcanised oil-extended masterbatch is an important consideration for the choice of the oil. Aromatic oils are known generally to impart less thermal stability to unvulcanised masterbatches when compared to paraffinic oils. This is supported by experimental data shown in Table 3 of the masterbatches containing different oils. Nonetheless it is noted that the same masterbatches showed no change in their viscosities after nine months' storage in the Institute's laboratories under ambient conditions. The choice of the oil therefore rests mainly on its cost and availability at the plantation. Some petroleum oils of the paraffinic and naphthenic types have already established applications in Malaysia and one such grade, Dutrex 66—an 'equivalent' of a more well known grade, Circosol 4240—has been found suitable for the manufacture of an OENRM. The method of manufacture is capable of accommodating different types of oil, for example, Dutrex 63, being used for appli-

cations where non-staining and non-discolouring characteristics are required of the masterbatch.

Oil-Black Ratio

Another important consideration for the manufacture of an OENRM is its oil content so that the best all-round combination of properties is obtained. The effect of oil and carbon black levels was studied, using the simple mixing cycle A shown below and a 'Size K Intermix' with a starting temperature of 70°C and a rotor speed of 40 rev/min.

Mixing Cycle A

- 0 min — add rubber
- 2 min — add half black, zinc oxide
and stearic acid
- 4 min — add balance of black
- 7 min — dump

(Sulphur and accelerator were added on the mill at 70°C.)

The rubber used was Heveacrumb containing 0, 10, 20, 30 parts by weight of Dutrex 66. A wide range of mixes was studied, covering up to 30 per cent weight replacement of rubber by oil and black levels of 40 to 60 parts per hundred (rubber plus oil) with the following base formulation:

	Parts by weight
Rubber (+ oil)	100
Zinc oxide	5
Stearic acid	2
HAF* (Philblack O)	40–60
PBN	1
Santocure CBS	0.5
Sulphur	2.5

* HAF from this manufacturer resulted in particularly high hardness figures.

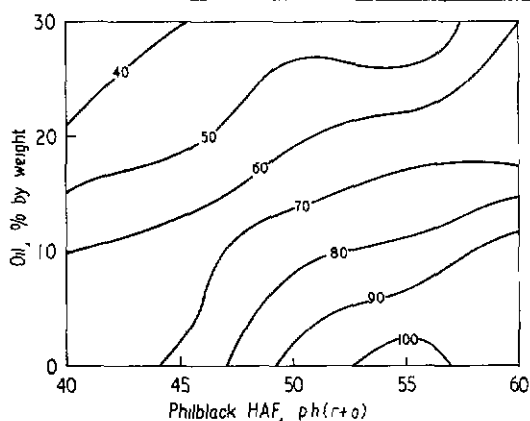


Figure 3. Mooney viscosity of compound, ML 1 + 4' at 100°C.

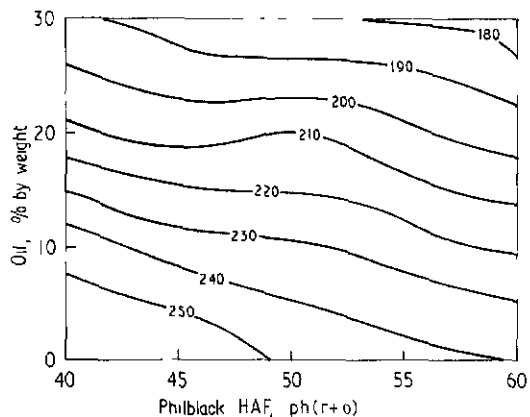


Figure 4. Tensile strength, kg/cm²

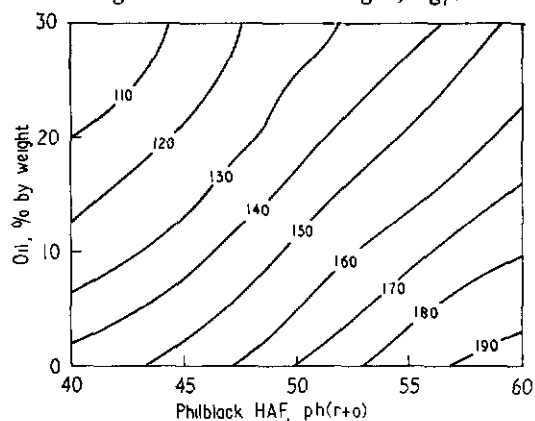


Figure 5. Modulus at 300%, kg/cm²

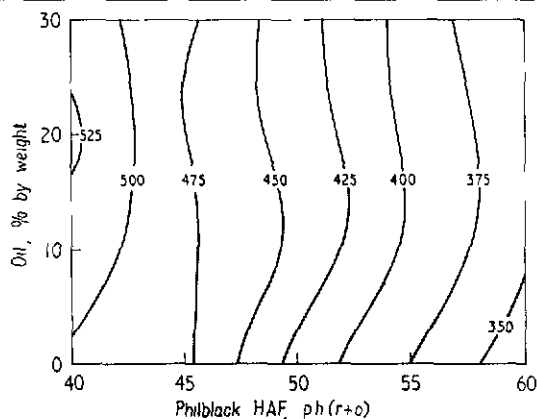


Figure 6. Elongation at break, %

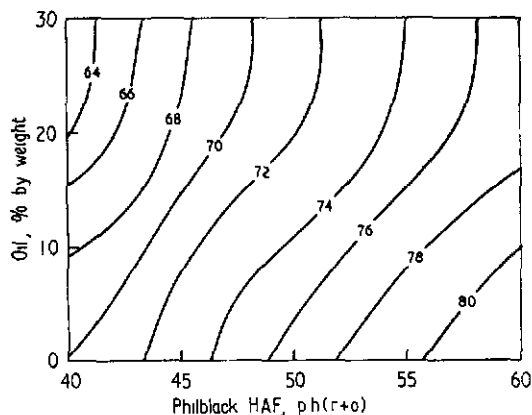


Figure 7. Hardness, IRHD

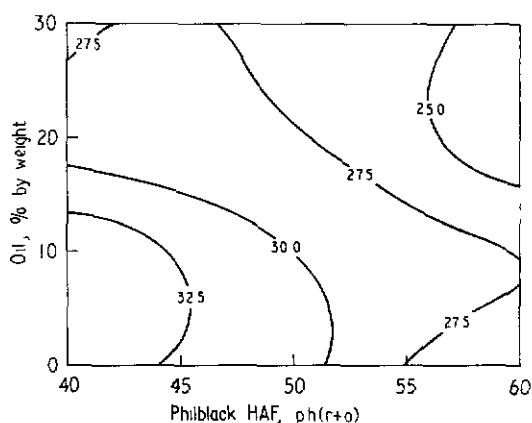


Figure 8. Tear strength, kg

Figures 3 to 8. Contour diagrams showing the change in properties for a range of black and oil levels.

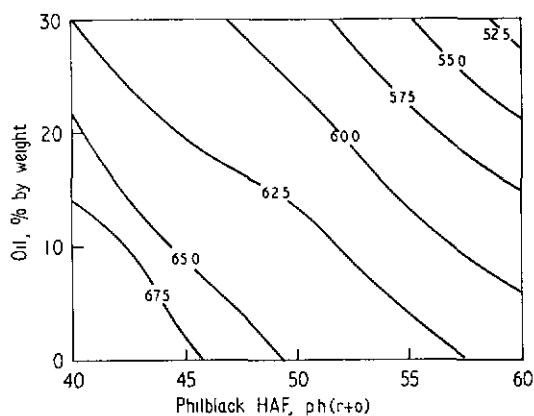


Figure 9. Dunlop resilience, %

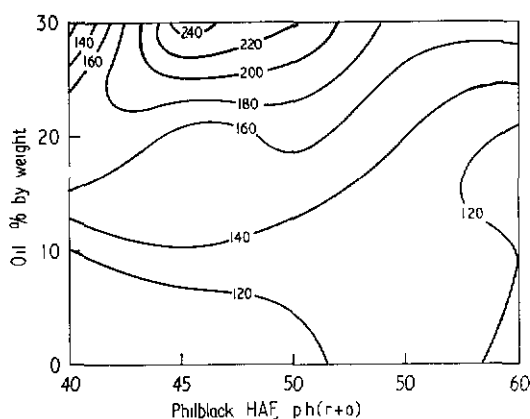


Figure 11. De Mattia flex-cracking, kc to Grade C.

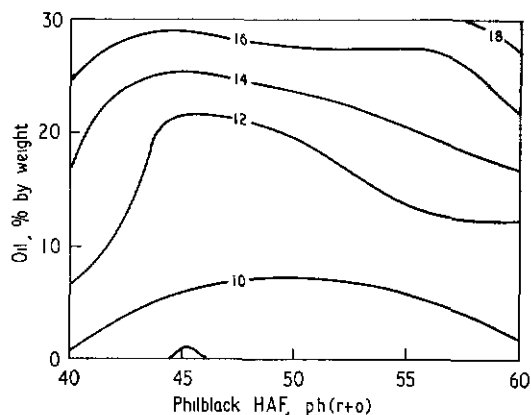


Figure 10. Compression set, %

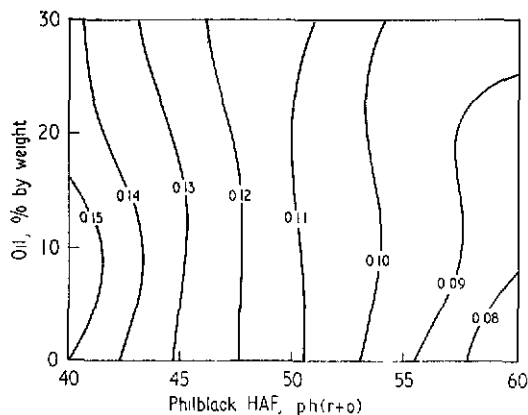


Figure 12. Akron abrasion resistance, vol. loss/1000 rev, cc.

Figures 9 to 12. Contour diagrams showing the change in properties for a range of black and oil levels.

The optimum cure was determined from hardness measurements and found to be 40 minutes at 140°C for all samples. The principal raw compound and vulcanisate properties are presented on contour diagrams (Figures 3 to 12). The effect of oil extension on the properties of black-loaded NR compounds is well known (MOORE *et al.*, 1965; SWIFT, 1967) and is confirmed in these results. In general, increasing oil results in a reduction in hardness, a fall in tensile properties and resilience and a deterioration of heat build-up and compression set

characteristics. Laboratory flex resistance and tear are, however little affected. It is noted that the contour diagrams do not show areas or 'troughs' of markedly inferior properties.

Resilience diminishes as expected with increasing carbon black and with increasing oil content. At 25 per cent oil extension and 55 parts of black, the resilience is reduced from 67 to 57%.

Akron abrasion resistance is little affected by oil extension though marginal improvements are seen with approximately 25 parts by weight

of oil. It is however very black-dependent and increases consistently with the black content. Tear resistance (crescent tear pieces) shows some scatter, but tends to be lower for the higher oil and black loadings. The flex-cracking resistance results obtained are very high. It appears that flex-cracking resistance reaches a maximum at about 45 parts of black and 30 parts of oil.

Summarising, the higher black levels give better abrasion resistance, higher compound viscosity and lower resilience. Tear and flex-cracking resistance are however reduced. A mix containing 75 parts of rubber, 25 parts of oil and 50 to 55 parts of carbon black represents a fair compromise between technical properties and cost.

Effect of Curative Levels

The compound and mixing cycle used in studying the effect of curative levels were as before, with the HAF black level fixed at 55 parts. Results are reported in the form of contour diagrams (Figures 13 to 21) with sulphur content against accelerator content as the axes. It is seen that when sulphur is increased to more than 2 parts, there is a rapid decrease in the scorch time.

Tensile strength, modulus, hardness and tear strength increase with the amount of sulphur and accelerator. Resilience shows an optimum value at approximately 0.6 of CBS and 2.25 of sulphur. Low accelerator levels give the best flex resistance and would thus be recommended in tyre applications where groove cracking is important. From the flex results a low level of accelerator is therefore recommended. When this finding is coupled with the scorch dependence on sulphur, the best compromise is probably 2 to 2.25 parts of sulphur with 0.4 to 0.5 parts of CBS as accelerator.

Effect of Type of Carbon Black

The same base compound, as previously described but with only 50 parts per hundred (r + o), was used to study the effect of carbon black type on the dispersion and properties of an OENRM compound.

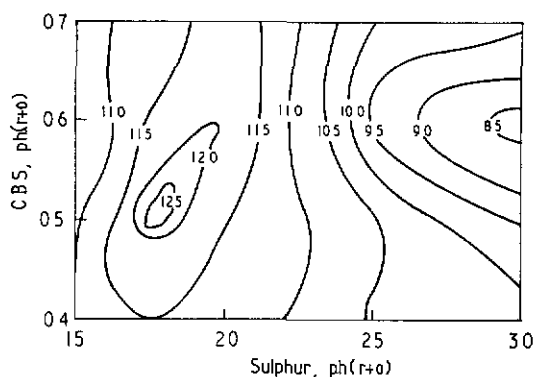


Figure 13. Mooney scorch time, min, to 5 point rise at 120°C.

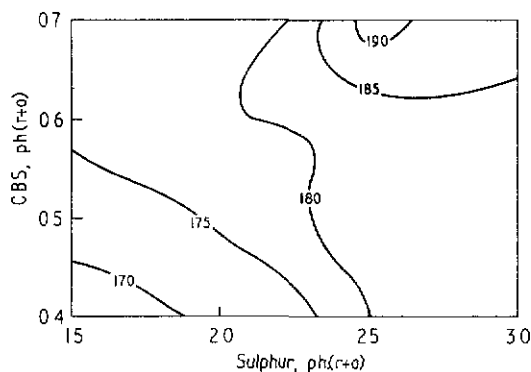


Figure 14. Tensile strength, kg/cm²

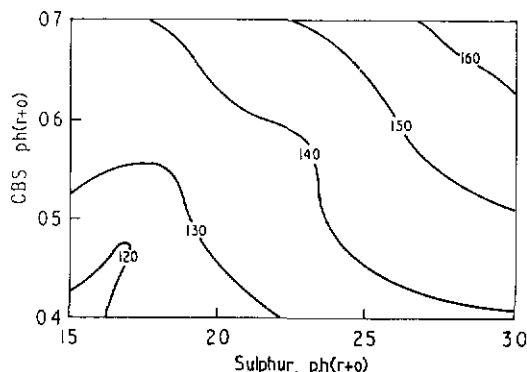


Figure 15. Modulus at 300%, kg/cm²

Figures 13 to 15. Contour diagrams showing the change in properties for a range of sulphur and accelerator levels.

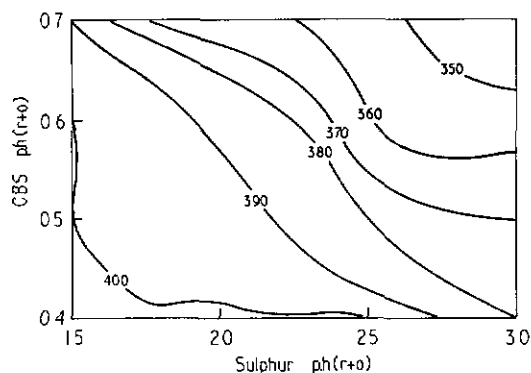


Figure 16. Elongation at break, %

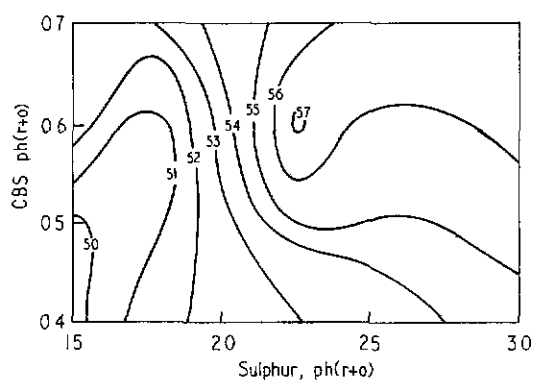


Figure 19. Dunlop resilience, %

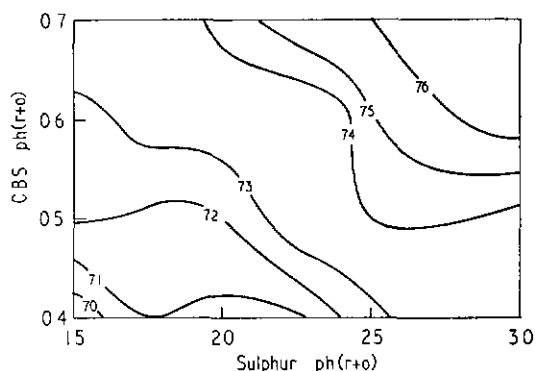


Figure 17. Hardness, IRHD

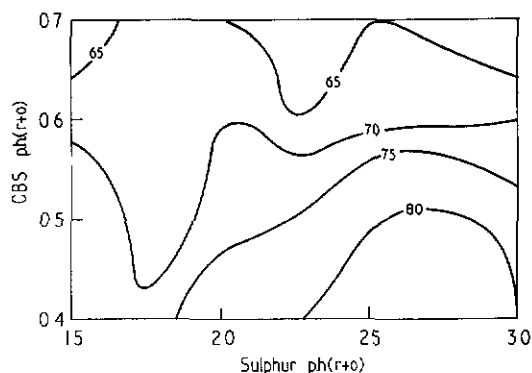


Figure 20. De Mattia flex cracking, kc to Grade C.

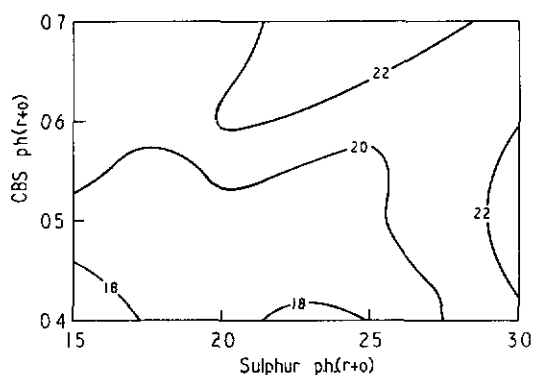


Figure 18. Tear strength, kg

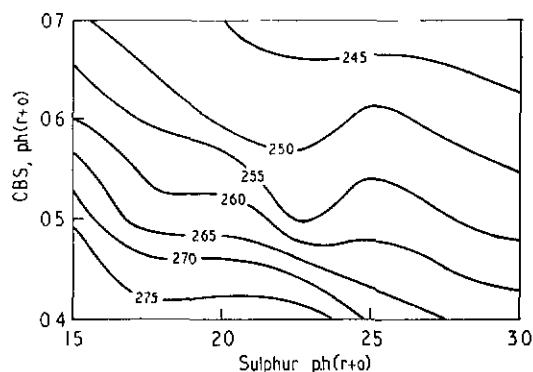


Figure 21. Heat build-up at 30 min, °F.

Figures 16 to 21. Contour diagrams showing the change in properties for a range of sulphur and accelerator levels.

Carbon black dispersion was assessed by visual examination of the surface appearance of the groove of a De Mattia flex-cracking test piece which is doubled. As seen from the photographs, Acarb SRF, Acarb FEF, United 65 SPF (high structure HAF), Acarb HAF and

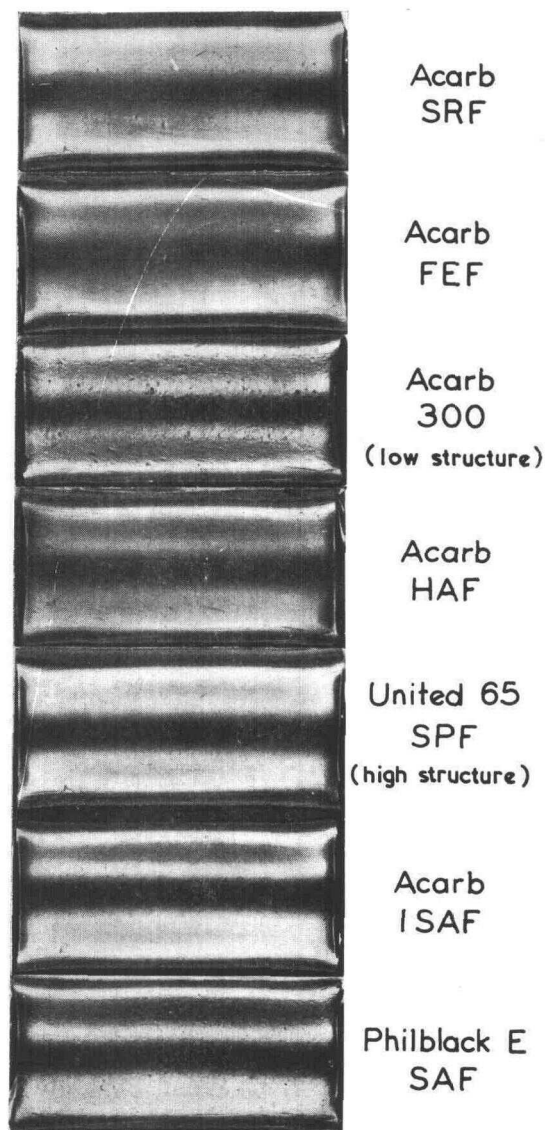


Figure 22. Bent flex pieces showing dispersion of black.

Acarb ISAF are well dispersed while Philblack E (SAF) and Acarb 300 (low structure HAF) are poorly dispersed.

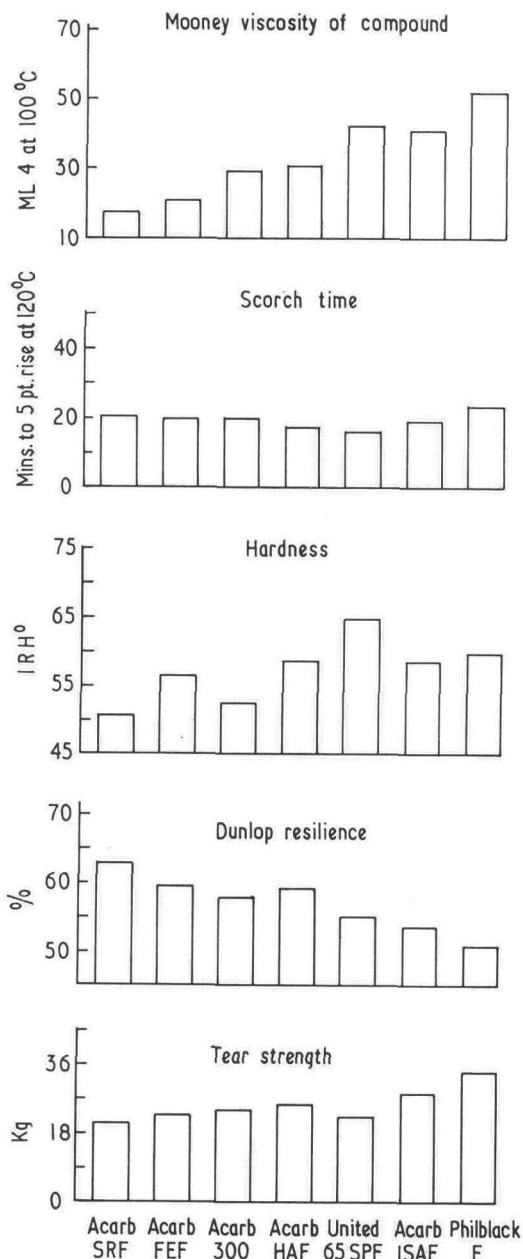


Figure 23. Histograms showing the physical properties obtained by compounding OENRM with 50 p.p.h. (r+o) of various blacks.

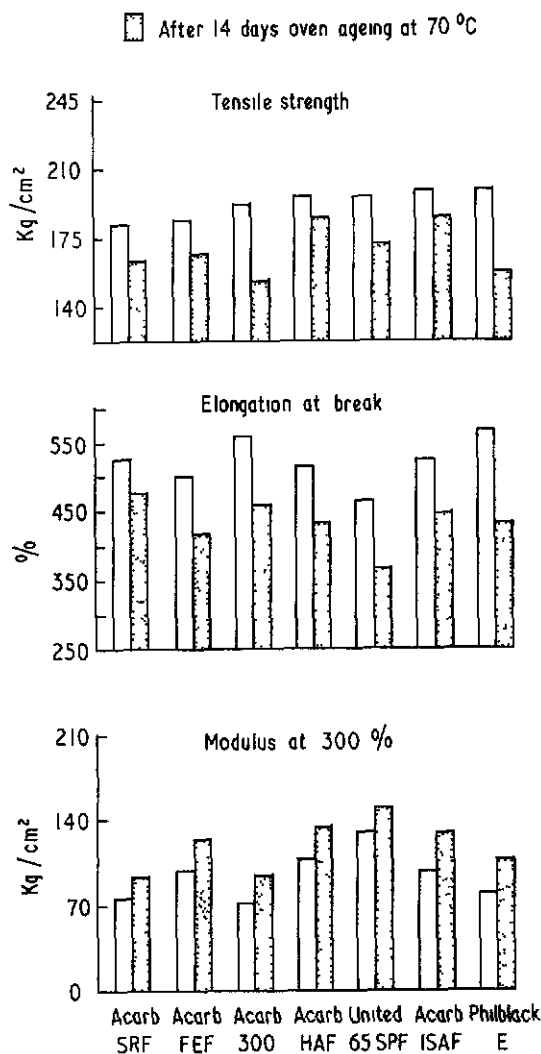


Figure 24. Histograms showing the physical properties obtained by compounding OENRM with 50 p.p.h. (r+o) of various blacks.

The results for compound and vulcanisate properties are shown in histograms (Figures 23 and 24).

At a level of 50 parts black (based on rubber + oil), compound viscosity is low for the more reinforcing or moderately reinforcing black but is satisfactory for HAF and other highly reinforcing types.

There is a slight decrease in tensile strength in changing from a highly reinforcing to a non-reinforcing black and from a high to a low structure black. The type of black in oil-extended compounds has a large effect on compound viscosity and vulcanisate hardness; a high structure HAF black, for example, gives a hardness 5 points higher than normal HAF. Tear strength shows the high values normally associated with natural rubber, but is highest for reinforcing black.

In general, OENRM gives good dispersion with easy processing for all but low structure and SAF blacks. If it is necessary to raise the compound viscosity or to raise vulcanisate hardness, a change to a black with higher structure will have the desired result.

Comparison between OENRM and Dry-mixed OENR

Simple mixing cycles have been developed for the dry-mixing of rubber, oil and carbon black directly together in a Banbury (MOORE *et al.*, 1965; SWIFT, 1967). These cycles can also be used for the OENRM with the possibility of even shortening them. OENRM was compared with the dry-mixed OENR at 25 per cent oil extension in the formulation indicated in Table 4. Cycles B and C were used for the OENRM and dry-mixed rubber respectively in the Banbury mixing (Model BC, 2 lb charge).

Mixing Cycle B

0 min — add carbon black, powders, and OENRM

2½ min — dump

(Sulphur and accelerator added on the mill at 70°C, cured 40 min at 140°C).

Mixing Cycle C

0 min — add carbon black, powders and rubber

1 min — add oil

3 min — dump

(Sulphur and accelerator added on the mill at 70°C, cured 40 min at 140°C).

Table 4 shows that the physical properties of these compounds are not significantly

TABLE 4. COMPARISON OF OENRM WITH DRY-MIXED OENR

Property	OENRM	Dry-mixed OENR
Tensile strength, kg/cm ²	204	220
Elongation at break, %	520	510
Modulus at 300%, kg/cm ²	96	105
Hardness, IRHD	58	60
Lupke resilience, 21°C, %	60	61.5
Akron abrasion loss, ml/500rev	0.035	0.035
De Mattia flex-cracking resistance, kc to Grade C.	205	181
Compound viscosity ML 1 + 4 at 100°C	55	52

Formulation:

Rubber	100
HAF	50
Zinc oxide	5
Stearic acid	2
Anti-oxidant 4010	1
CBS	0.4
Sulphur	2.0

different. However, OENRM may offer handling advantages for compounds containing fillers with poor oil absorption properties; it has greater flexibility in mixing cycles. Additionally OENRM can be mill-mixed and would be useful for manufacturers with limited mixing facilities.

PRODUCTION OF HEVEACRUMB OENR FROM MALAYSIAN PLANTATIONS

OENRM is at present produced in tonnage quantities sufficient for evaluation purposes, but it is not yet in large-scale commercial production for three reasons.

The first and most important reason is that the present prices of process oils available in West Malaysia (even though these have already found other definite markets) are higher than

those in consumer countries (though competitive prices are indicated when sufficiently large volumes are required). A re-appraisal by the oil suppliers would be opportune and any reduction in present prices would inevitably brighten the prospects for the masterbatch production. Secondly, the present design of deep-bed crumb driers is not entirely satisfactory for OENRM crumb despite its large surface area and consequently its short drying time. An unsatisfactory throughput of the OENRM is obtained because each drying tray can only be loaded to one-third its usual depth. Modification to a multilayer system, each of 4 inches, is considered more satisfactory (SETHU, 1968). Thirdly, there is the problem of tariff barriers imposed by consuming countries, principally U.S.A. and Japan. However, increasing demand from other countries which have more relaxed tariff rules, e.g., U.K., the Soviet Union, Eastern Europe, Germany and China can alleviate this problem to some extent.

CONCLUSIONS

An oil-extended natural rubber masterbatch containing 75 parts by weight of rubber and 25 parts by weight of a process oil can be manufactured at the plantations using the Heveacrumb process. The choice of the process oil is primarily dependent on price and availability; a naphthenic type is at present preferred in West Malaysia. However, masterbatches with other oil types can be manufactured without difficulty.

The OENRM has excellent processing characteristics and gives vulcanisate properties similar to those obtained by the dry-mix process.

The availability of an OENRM is largely governed by the cost of the process oil and any reduction in oil price would inevitably encourage its production. Tariff barriers in some consuming countries may retard its promotion, but the increasing demand of NR supplies by other countries with more liberal tariffs will undoubtedly offset this.

ACKNOWLEDGEMENT

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DISCUSSION

Chairman: Mr. J. E. Morris

Mr. H.J.M. Langshaw asked if it was possible to carry out oil-extension of natural rubber by the same method used in the synthetic rubber industry, *i.e.*, to determine the initial Mooney viscosity and then add adequate oil to obtain a particular value. Mr. Chin considered the processes to be fundamentally different: chains might be lengthened in SBR by modifying the polymerisation system, whereas natural rubber was restricted to cross-linking. The selection of latex sources to give suitable initial viscosities had been investigated. Mr. O'Connell noted that it was possible, but not necessarily preferable, to increase the Mooney viscosity by treatment with hydrazine hydrate to harden natural rubber. Dr. P.M. Sargo added that synthetic rubber with a narrow molecular weight distribution had a greater capacity for oil absorption than that with a broad molecular weight distribution.

Mr. A.H. Ritchie enquired about the relative merits of different oil-extension methods, in particular the Kualakep process. The Chairman and the authors agreed that such comparisons could not be made until the details of the Kualakep process were made available to the producers, who would be advised to study this aspect carefully. Mr. O'Connell added that soaking was an interesting stage because it allowed a wide choice of raw material; however, uniformity of the final product was the most important criterion.

Mr. Chin replied to Dr. A.A. Watson that the drying characteristics of oil-extended natural rubber in deep-bed driers were independent of the types of oil used so far.