

# PATENT SPECIFICATION

DRAWINGS ATTACHED

880.739



Date of filing Complete Specification June 30, 1958.

Application Date March 28, 1957.

No. 10188/57.

Complete Specification Published Oct. 25, 1961.

Index at acceptance:—Class 70, E6(A:G).

International Classification:—C08c.

## COMPLETE SPECIFICATION

### Improvements in the Preparation of Superior Processing Rubbers

We, THE NATURAL RUBBER PRODUCERS' RESEARCH ASSOCIATION, (formerly known as The British Rubber Producers' Research Association), a Body Corporate duly organised under the Laws of Great Britain, of 19, Buckingham Street, Adelphi, London, W.C.2, (formerly of 19, Fenchurch Street, London, E.C.3.), do hereby declare this invention (which invention has been communicated to us by BALACHANDRA CHAKKINGAL SEKHAR, a citizen of the Federation of Malaya and WILLIAM GEORGE WREN, a British Subject both of the Rubber Research Institute of Malaya, Kuala Lumpur, Malaya), for which we pray that a patent may be granted to us to be particularly described in and by the following statement:—

This invention relates to the manufacture of rubbers and more especially superior processing rubbers.

In the manufacture of superior processing rubbers, hereinafter called S.P. rubbers according to copending Patent Application No. 7770/54 (Serial No. 803,013), vulcanized latex is mixed with ordinary latex in the correct proportions the mixture being coagulated and machined in the usual way to give sheet or crepe rubber so that the dry rubber obtained contains 20 parts of vulcanized to 80 parts of unvulcanized raw rubber. In practice, the process has been limited to certain grades of rubber, viz. those equivalent to No. 1 smoked sheet, (or air-dried sheet) and No. 1 pale crepe. So far it has not been practicable to produce lower grades of rubber having S.P. characteristics.

According to the present invention, superior processing characteristics can now be imparted to a wide range of synthetic and natural rubbers including lower grades of natural rubber, by blending them in suitable proportions with a masterbatch containing a high proportion of vulcanized rubber particles embedded in a matrix of unvulcanized rubber

prepared by mixing a high proportion of vulcanized latex with field latex followed by coagulation in the customary manner.

The masterbatch can be dried to a continuous sheet or to a crumb, according to the proportion of vulcanized rubber present. Products containing 95 parts of vulcanized to 5 parts of raw rubber have been satisfactorily prepared; they are obtained in the form of a crumb. The preferred proportion of vulcanized rubber for masterbatch preparation is however from 80 to 90 parts of vulcanized rubber in 100 parts of masterbatch which can then be machined to a coherent sheet if required.

Owing to the fact that the continuous phase in these masterbatches consists of unvulcanized raw rubber, they can be readily diluted with further quantities of raw rubber or with compatible types of synthetic rubber to give mixtures containing the required proportion of vulcanized particles. The proportion usually preferred for superior processing properties is approximately 80 parts of unvulcanized raw rubber to approximately 20 parts of vulcanized rubber particles. The mixing can be accomplished in two ways, the first employing the wet coagulum of masterbatch, the second utilising masterbatch in the form of the dry crepe, sheet or crumb.

In the wet process, the masterbatch coagulum is mixed in the correct proportions with further raw rubber also in the form of coagulum or scrap, using a creping battery. This process can be usefully employed on estates in the production of S.P. brown crepes from scrap rubber and confers excellent S.P. properties on the finished crepe. It can similarly be employed in remilling factories, when the masterbatch coagulum is mixed in an appropriate proportion with scrap rubbers to produce S.P. remilled blankets or crepes, again having excellent S.P. characteristics.

In the second method, the dry masterbatch

[Price 3s. 6d.]

Price 4s 6d.

is mixed with any grade of dry raw rubber, natural or compatible synthetic, employing open roller mills or internal mixers, and confers on the product excellent S.P. characteristics. This process is envisaged as being carried out in factories producing rubber goods and involves marketing the S.P. masterbatch as sheet, crepe or crumb.

The test that is employed to show superior processing characteristics is to extrude a compounded rubber tread mix through an orifice of known dimensions at a constant temperature and worm speed and measure the increase in dimensions of the extruded material over that of the orifice.

#### EXAMPLE 1.

The following is an example of the preparation of superior processing rubber by the wet process and of the reduction in extrusion swell when it is made into a tyre tread compound. 20 parts by weight of unvulcanized rubber in latex form were mixed with 80 parts of vulcanized rubber also in latex form and prepared by using an accelerated sulphur recipe and coagulated in the usual manner by the addition of formic acid. The required proportion of the resulting wet crumb was then blended with wet scrap rubber on the crepeing battery. This blending was carried out without altering the normal operation involved in producing remilled crepes from wet scrap. The proportion of wet scrap required to give a final blend of 20 parts vulcanized rubber and 80 parts of unvulcanized rubber was calculated from the estimated dry rubber content of the wet scrap. The extrusion swell of the resulting dry superior processing remilled crepe was 18.7 p.c. compared with 32.5 p.c. for the remilled crepe control. When the proportion of vulcanized rubber particles in the concentrate was 90% and this more concentrated masterbatch was mixed with wet scrap rubber on the crepeing battery so that the final proportion of vulcanized rubber particles in the mixture was 20 p.c., the extrusion swell of the superior processing mixture was 19.2 p.c.

#### EXAMPLE 2.

The following is an example of the preparation of superior processing rubber by the dry process and of the reduction in extrusion swell when it is made into a tyre tread compound. 10 parts of unvulcanized rubber in latex form were mixed with 90 parts of vulcanized rubber also in latex form prepared as in Example 1. The mixture was coagulated in the usual way by addition of acid. The coagulated crumb was then passed through the crepeing mill once to remove excess water and dried. The drying process usually took 24 hours at room temperature i.e. 28–30° C.

6.67 lb. of the dry masterbatch was blended on open rolls with 23.33 lb. of smoked sheet for 20 minutes. This corresponds approxi-

mately to 20% of vulcanized rubber particles in the mixture. The extrusion swell of the superior processing blend was 14.8% compared with 30.2% for the smoked sheet control.

In the above examples when the proportion of vulcanized particles in the masterbatch was 95% and this more concentrated masterbatch was mixed in the dry state with dry smoked sheet on open rolls so that the final proportion of vulcanized rubber particles in the mixture was 20% the extrusion swell of the resulting superior processing sheet was 13.8%.

Similarly when a dry masterbatch containing 90% vulcanized particles was mixed on open rolls with dry remilled blanket crepe so that the final proportion of vulcanized rubber particles in the mixture was 20%, the extrusion swell of the resulting superior processing remilled blanket crepe was 14.3% compared with 33% for the control remilled blanket crepe. The improvement in extrusion swell was determined in a carbon black tread compound. An alternative method of assessing superior processing properties is by means of a mill shrinkage test on the uncompounded rubber. The test is carried out on a two-roll laboratory mill in the following manner:—

|                               |   |   |       |     |
|-------------------------------|---|---|-------|-----|
| Diameter of rolls (in.)       | - | - | 6     | 95  |
| Length of rolls (in.)         | - | - | 12    |     |
| Friction ratio                | - | - | 1.4   |     |
| Surface speed—front (ft./min) | - | - | 33.7  |     |
| Surface speed—back (ft./min)  | - | - | 47.1  |     |
| Temperature of rolls (°C.)    | - | - | 70    | 100 |
| Weight of rubber (gm)         | - | - | 200   |     |
| Distance between guides (in.) | - | - | 9     |     |
| Nip width at start (in.)      | - | - | 0.030 |     |
| Nip width at finish (in.)     | - | - | 0.070 |     |

Mill for 4 minutes during which the rubber is cut and folded over 5 times from one side and 5 times from the other side, two successive cuts being from opposite sides. During the next half minute open the rolls in stages from 0.030 to 0.070 in. nip.

Then over a period of half minute while the rolls are still revolving cut a centre strip 4½ in. wide using two mill cutting knives fixed at the required distance by a metal bar.

Stop the rolls; cut across, restart rolls and take out the centre strip. Lay on a smooth chalked surface. After 24 hours interval, measure length L cm.

$$\text{Shrinkage} = \frac{47.8 - L}{47.8} \text{ per cent.}$$

(circumference of 6 in. dia. roll = 47.8 cm).

Reduction in mill shrinkage gives a measure of the superior processing properties conferred by the masterbatch.

The effectiveness of dry masterbatch in reducing mill shrinkage is clearly shown by Fig. 1 in the accompanying drawing in which the mill shrinkage is plotted against the vulcanized component in mixtures of natural rubber (RSS) with increasing proportions of the dry 90/10 concentrate of Example 2.

#### EXAMPLE 3.

Mill shrinkage tests were similarly carried out with low temperature styrene-butadiene copolymer with addition of the masterbatch described in Example 2 on the one hand and styrene-butadiene copolymer lightly cross-linked with di-vinyl benzene on the other, a material commonly used as a processing aid for styrene-butadiene rubbers. These materials were added in the ratio of 30 parts by weight to 100 of copolymer. The addition of masterbatch and of cross-linked styrene-butadiene copolymer was quickly effected at the beginning of the test without the need to increase the time of milling. The average value of four mill shrinkage tests with the low temperature styrene-butadiene copolymer was 42%. The average shrinkage in two tests with the cross-linked styrene-butadiene copolymer addition was 40%, and of two tests with the masterbatch addition was 26%.

#### EXAMPLE 4.

Tests were similarly carried out with high temperature styrene-butadiene copolymer alone and with respective additions of masterbatch and of the cross-linked styrene-butadiene copolymer in the ratio of 30 parts copolymer.

The average shrinkage in three tests with high temperature styrene-butadiene copolymer was 64%; in four tests with cross-linked styrene-butadiene copolymer addition it was 61%; in two tests with masterbatch addition it was 32%.

#### WHAT WE CLAIM IS:—

1. A method of imparting superior processing characteristics to synthetic and natural rubbers and particularly lower grades

of natural rubber by blending them in suitable proportions with a masterbatch containing a high proportion of vulcanized rubber particles embedded in a matrix of unvulcanized rubber, prepared by mixing a high proportion of vulcanized latex with natural latex, followed by coagulation in a customary manner.

2. A method of imparting superior processing characteristics to rubbers according to Claim 1 wherein the masterbatch contains 80% to 90% vulcanized rubber and can be machined to a coherent sheet.

3. A method of imparting superior processing characteristics to rubbers according to either of the preceding claims wherein the masterbatch is diluted with further quantities of raw rubber or with compatible types of synthetic rubber so as to contain approximately 80% unvulcanized raw rubber.

4. A method of imparting superior processing characteristics to rubbers substantially as described with reference to Example 1.

5. A method of imparting superior processing characteristics to rubbers substantially as described with reference to Example 2.

6. A method of imparting superior processing characteristics to rubbers substantially as described with reference to Example 3.

7. A method of imparting superior processing characteristics to rubber substantially as described with reference to Example 4.

8. A method according to any of Claims 1 to 3 wherein the masterbatch is mixed with wet raw rubber.

9. A method according to any of Claims 1 to 3 wherein the masterbatch in the form of dry crepe, sheet or crumb is mixed with dry raw rubber.

10. Superior processing rubber when prepared according to Claims 1 to 7 substantially as described.

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rubbers and more especially superior processing rubbers.

In the manufacture of superior processing hereafter called S.P. rubbers according to co-pending Patent Application No. 7770/54 (Serial No. 803,013) vulcanized latex is mixed with ordinary latex in the correct proportions, the mixture being coagulated and machined in the usual way to give sheet or crepe rubber, the dry rubber obtained containing 20 parts of vulcanized to 80 parts of raw rubber. In practice, the process has been limited to certain grades of rubber, viz. those equivalent to No.



The effectiveness of dry masterbatch in reducing mill shrinkage is clearly shown by Fig. 1 in the accompanying drawing in which the mill shrinkage is plotted against the vulcanized component in mixtures of natural rubber (RSS) with increasing proportions of the dry 90/10 concentrate of Example 2.

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of natural rubber by blending them in suitable proportions with a masterbatch containing a high proportion of vulcanized rubber particles embedded in a matrix of unvulcanized rubber prepared by mixing a high proportion of vulcanized latex with natural latex, followed by coagulation in a customary manner.

2. A method of imparting superior processing characteristics to rubbers according to Claim 1 wherein the masterbatch contains 80% to 90% vulcanized rubber and can be machined to a coherent sheet.

3. A method of imparting superior processing characteristics to rubbers according to either of the preceding claims wherein the masterbatch is diluted with further quantities of raw rubber or with compatible types of synthetic rubber so as to contain approximately 80% unvulcanized raw rubber.

4. A method of imparting superior processing characteristics to rubbers substantially as described with reference to Example 1.

5. A method of imparting superior processing characteristics to rubbers substantially as described with reference to Example 2.

6. A method of imparting superior processing characteristics to rubbers substantially as described with reference to Example 3.

7. A method of imparting superior processing characteristics to rubber substantially as described with reference to Example 4.

8. A method according to any of Claims 1 to 3 wherein the masterbatch is mixed with wet raw rubber.

9. A method according to any of Claims 1 to 3 wherein the masterbatch in the form of dry crepe, sheet or crumb is mixed with dry raw rubber.

10. Superior processing rubber when prepared according to Claims 1 to 7 substantially as described.

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1 smoked sheet, (or air-dried sheet) and No. 1 pale crepe. So far it has not been practicable to produce lower grades of rubber having S.P. characteristics.

5 According to the present invention, superior processing characteristics can now be imparted to a wide range of natural and synthetic rubbers including lower grades of natural rubber, by mixing high proportions of vul-  
10 canized latex with field latex, followed by coagulation in the customary manner, to produce a masterbatch comprising particles of vulcanized rubber embedded in a matrix of raw rubber.

15 The material can be dried to a continuous sheet or to a crumb, according to the proportion of vulcanized rubber present. Products containing up to 95 parts of vulcanized to 5 parts of raw rubber have been satisfactorily prepared; they are obtained in the form of  
20 a crumb. The preferred proportion of vulcanized rubber for masterbatch preparation is however 90 parts to 10 parts of raw rubber.

It is further envisaged that to cheapen cost  
25 of production, the addition of oil or vulcanized oil as liquid, solid or emulsion, could be employed, thus producing an S.P. lower grade material at approximately the same cost as the normal lower grade.

30 Owing to the fact that the continuous phase in these masterbatches consists of raw rubber, the materials can be readily diluted with other forms of raw rubber, natural or synthetic, to give S.P. rubbers containing the required proportion of 20 parts vulcanized to 80 parts raw  
35 rubber. The mixing can be accomplished in two ways, the first employing the wet co-

agulum of masterbatch, the second utilizing the dry crepe, sheet or crumb.

In the wet process, the masterbatch co-  
40 agulum is mixed in the correct proportions with further raw rubber also in the form of coagulum or scrap, using a creping battery. This process can be usefully employed on  
45 estates in the production of brown crepes from scrap rubber and confers excellent S.P. properties on the finished crepe. It can similarly be employed in remilling factories, when the masterbatch coagulum is mixed in an appropriate proportion with scrap rubbers to pro-  
50 duce remilled blankets or crepes, again having excellent S.P. characteristics.

In the second method, the dry masterbatch is mixed with any grade of dry rubber, natural or synthetic, employing open roller mills or  
55 Banbury mixers, and confers on the product excellent S.P. characteristics. This process is envisaged as being carried out in factories producing rubber goods and involves market-  
60 ing the S.P. masterbatch as sheet, crepe or crumb.

The test that is employed to show superior processing characteristics is to extrude a compound rubber tread mix through an orifice of  
65 known dimensions at a constant temperature and measure the increase in dimensions of the extruded material over that of the orifice. A material will be superior processing if extru-  
70 sion swell % is less than 70% of that of ordinary rubber.

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