

PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements in or relating to the processing of Natural Rubber

We, THE BOARD OF THE RUBBER RESEARCH INSTITUTE OF MALAYA, of 3rd Mile Ampang Road, Kuala Lumpur, Malaya, a Body Corporate of Malaysia, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention is for improvements in or relating to the processing of natural rubber and has for an object to facilitate the production of rubber in crumbled form.

15 Many attempts have been made to process natural rubber latex and scrap rubber in forms other than as sheets or crepes. Such attempts include mechanical breakdown, flocculation and crumbling. Both flocculation and crumbling in the past have meant adulterating rubber with chemicals which cause deterioration in its ageing and properties. Mechanical breakdown necessitates new machinery and has other difficulties in particle size in relation to drying. There is therefore a need for a simple and effective means of crumbling rubber without conferring undue deterioration in properties. The present invention offers one such method.

20 The basis of our method is our discovery that when a compatible oil, such as a rubber extender oil, is added to wet rubber, whether normal or scrap rubber, on a crepeing mill, the rubber crumbles. However, in the case of very highly compatible oils, e.g. those of the aromatic type, the addition of oil as such on the crepeing mill produces only a transient crumbling effect and, in general, the direct addition of oil to rubber on the mill is a messy operation and only a limited control can be exerted on either the quantity of oil incorporated or the uniformity of incorporation. We have now found that the oil can be added conveniently in the form of a masterbatch with latex, fresh or vulcanised.

According to the present invention there is provided a method of processing natural rubber which comprises milling a masterbatch comprising a wet coagulum of natural or synthetic rubber latex having a compatible oil dispersed therein with wet natural rubber in the form of a coagulum or solid mass, the milling being continued at least until the rubber crumbles.

It is desirable to dry the resultant crumbled rubber and this is readily achieved by passing heated air through a bed of the rubber crumbs. The masterbatch may be readily obtained by emulsifying or dispersing the oil in the latex, which may be wholly fresh or vulcanised latex or a mixture thereof, and thereafter coagulating the mixture of oil emulsion or dispersion and latex.

It will be appreciated that the invention includes the use of such compatible oils as have a greasy consistency at normal ambient temperatures and the term "oil" as used herein is to be interpreted accordingly.

We have discovered that the higher the percentage of oil in the masterbatch, the lower is the minimum concentration of oil required in the final rubber mix to cause effective crumbling. The maximum amount of oil that could be incorporated in the latex to form a masterbatch is limited because too high a concentration makes the masterbatch too sticky to handle conveniently but it has been ascertained that a masterbatch containing up to 90% by weight of oil incorporated therein may be handled without undue difficulty. Using such a masterbatch, the proportion necessary to cause satisfactory crumbling of the wet latex coagula, crepe, sheet or scrap rubber is normally only about 1 part of masterbatch to 22 parts of the wet rubber on the basis of dry weights, depending on the particular oil used. The particles of such crumbled rubbers are fine in size, thereby

enabling drying to be effected at a faster rate. In the case of the naphthenic type extender oil sold under the trade name "Circosol" 596, the concentrations of oil required in the final

product to achieve effective crumbling, using a masterbatch containing 90% by weight of the oil, are shown in the following Table.

TABLE I

Concentration of oil in % by weight on final rubber	Ratio of masterbatch to coagula (dry wts.)	Appearance of final rubber
1	1:89	crepe
2	1:44 $\frac{1}{2}$	brittle crepe
3	1:29 $\frac{2}{3}$	brittle crepe
4	1:22 $\frac{1}{4}$	crumb
5	1:17 $\frac{4}{5}$	fine crumb
6	1:14 $\frac{5}{6}$	fine crumb
7	1:12 $\frac{5}{7}$	fine crumb
8	1:11 $\frac{1}{8}$	fine crumb

10 The oil incorporation to the levels necessary to achieve effective crumbling of the rubber, e.g. in the case of "Circosol" 596 from about 4 to 10% by weight, does not greatly affect the physical properties of the final rubber, although the small amount of oil incorporated tends to reduce the Mooney viscosity of the rubber and improve its processing characteristics. Further, by replacing fresh latex in the masterbatch with vulcanised latex, either wholly or in part, the extrusion characteristics and the appearance of the final dry crumbs can be improved. The crumbled rubbers were submitted to drying by forcing hot air at 100° C. through a bed four inches thick. Drying under such conditions took only 2 hours. Thus, 15 the present invention provides an effective means of producing natural rubber in crumbled form. This is of importance not only from the point of view of drying rate but also in producing a form of rubber which can be easily specified technically. The method is of additional advantage in that latex in which the compatible oil is dispersed may be at field dry rubber content. Such a method allows incorporation of desirable chemicals when it is 20 necessary without loss due to washing out by dilution during coagulation.

40 Examples of other compatible oils which may be employed are aromatic type oils, such as the oil sold under the trade name "Dutrex" R and aliphatic type oils such as palm oil ("Dutrex" is a Registered Trade Mark.) The levels of oil necessary to produce the desired crumbling effect will depend on the type of

oil selected but in general the concentration of oil in the final rubber will be in the range of from 3% to 10% by weight.

The following Examples will serve to illustrate the manner in which the crumbling of rubber may be carried into effect in accordance with the present invention.

EXAMPLE 1.

90 gms. of "Circosol" 596 oil were emulsified with 0.9 gm. oleic acid and sufficient aqueous ammonia added to form the emulsion. This emulsion was then mixed with natural rubber latex containing 10 gms. of rubber on a dry weight basis. The mixture was then coagulated using formic acid. The oil/rubber masterbatch so prepared was divided into five equal portions. Fresh natural rubber latex was then divided into five unequal portions containing the following quantities of rubber:

- (a) latex containing 300 gms. rubber
- (b) latex containing 400 gms. rubber
- (c) latex containing 500 gms. rubber
- (d) latex containing 600 gms. rubber
- (e) latex containing 700 gms. rubber

These portions (a) to (e) were then separately coagulated and to each of the coagulated portions one portion of the masterbatch was added on a crepeing mill. In each case the mixture was passed through the crepeing mill battery six times. The nature of the final rubber after drying in each case is given below.

	Drying Time
(a) Fine crumb	2 hours
(b) Fine crumb	"
(c) Crumb-brittle crepe	"
(d) Brittle crepe	"
(e) Brittle crepe	5 hours

EXAMPLE 2.

100 gms. of a masterbatch containing 80 parts of a "Circosol" 596 oil was prepared as in Example 1 and divided into five equal portions. Five unequal portions of fresh latex containing the following quantities of rubber were coagulated separately.

- (a) 300 gms.
(b) 400 gms.

- (c) 500 gms.
(d) 600 gms.
(e) 700 gms.

One portion of the masterbatch was added to each of these coagulated portions (a) to (e) on a crepeing mill and passed through the crepeing battery six times. The nature of the product obtained after drying is given below.

	Drying Time
(a) Fine crumb	2 hours
(b) Crumb/brittle crepe	2 hours
(c) Brittle crepe	4 hours
(e) Crepe	12 hours

EXAMPLE 3.

The procedure described in Example 1 was repeated except that the masterbatch was mixed with wet scrap rubber instead of with coagulated latex in exactly the same proportions. The results obtained were closely similar to those given in Example 1.

The present invention thus provides a method by which natural rubber, ordinary or scrap grade, can be produced in a crumbled form which facilitates drying and constitutes a better presentation of the rubber. The physical properties of such crumbled rubbers are little different from those made from conventional rubbers. This method is equally applicable when vulcanised latex is used in place of some or all of the fresh latex to form the masterbatch. By using vulcanised latex either

as a complete replacement or partial replacement of fresh latex, the tacky nature of the crumbled rubber can be improved. This, although not absolutely necessary, may be attractive in certain instances.

A further series of samples of crumbled rubber containing "Circosol" 596 were formulated for the purpose of determining the effect on the physical properties of the rubber of the presence of the oil. Three batches of crumbled rubber were prepared, containing 5%, 8% and 10% by weight respectively of "Circosol" 596, by mixing a latex rubber masterbatch containing 90% by weight of the oil with the appropriate quantity of rubber on the mill. Each batch was compounded according to the following recipe and vulcanised:—

Rubber	=	100 parts by weight
Stearic acid	=	0.5 parts by weight
Mercaptobenzthiazole	=	0.5 " " "
Zinc oxide	=	6.0 " " "
Sulphur	=	3.5 " " "

A control crepe, containing no oil, was compounded according to the same recipe and vulcanised. The physical properties of the result-

ing vulcanised rubbers are given in the following Table: —

TABLE II

% by weight of oil in rubber	Tensile Strength lbs/sq.in.	Elongation at break %	Modulus 300% lbs/sq.in.	Hardness BS°	Resilience %
Control	2731	849	213	40.5	85.5
5%	2077	882	157	36.3	82.8
8%	2134	890	157	35.0	84.0
10%	2006	892	157	34.0	81.7

10 By using larger amounts of a compatible oil in the form of the oil/rubber masterbatches as above described, we can increase the amount of oil incorporated into the natural latex coagula or scrap rubber and thereby improve the processing characteristics of the rubber and at the same time can produce oil-extended rubbers containing any desired amount of oil 15 without encountering the difficulty in drying the oil-extended rubbers produced direct from rubber latices which has been experienced in the past when oil, or ~~dry/oil~~ rubber masterbatches had been used. The use of masterbatches prepared from vulcanised latices impart to the final dry, crumbled rubber superior processing properties for example in extrusion. 20

Utilising such a method we have found that 25 oil to a level as high as 40% could be incorporated without too great a difficulty in drying, the normal range being from 10% to 40% by weight of oil based on the dry rubber content. This further aspect of our present invention is illustrated in the following example. 30

EXAMPLE 4.

The following masterbatches were prepared by mixing latex, vulcanised or unvulcanised, with a rubber extender oil in an emulsion form 35 using ammonium oleate or tetraethylenepentamine oleate for emulsification. The mixture was coagulated using formic acid.



dry oil/rubber

	Oil	Rubber	Vulcanised Rubber
(a)	90	10	—
(b)	90	—	10
(c)	90	5	5
(d)	80	20	—
(e)	80	10	10
(f)	80	5	15
(g)	80	0	20

These masterbatches were then mixed on the crepeing mill with fresh wet natural latex coagula to produce a final concentration of 25% by weight of oil based on dry rubber content. In all cases at the end of 10 passes through the crepeing battery, the mass crumbled into fine particle size. These were then submitted to drying. All samples dried in 2 days at 140° F. The samples containing vulcanised rubber showed less tackiness and dried faster. Preliminary evaluation of rubbers incorporated with oil utilising this method showed that the physical properties are as good as those of oil-extended rubbers obtained by other methods of incorporating rubber extender oils into natural rubber. The process is equally effective with scrap grades of rubber.

WHAT WE CLAIM IS:—

1. A method of processing natural rubber which comprises milling a masterbatch comprising a wet coagulum of natural or synthetic rubber latex having a compatible oil dispersed therein with wet natural rubber in the form of a coagulum or solid mass, the milling being continued at least until the rubber crumbles.

2. A method according to claim 1 wherein the masterbatch is prepared by emulsifying or dispersing the oil in the latex and thereafter coagulating the resultant mixture of oil emulsion or dispersion and latex.

3. A method according to claim 1 or claim 2 wherein the natural or synthetic rubber latex is a vulcanised latex or a mixture of vulcanised and unvulcanised latices.

4. A method according to any one of the

preceding claims wherein the masterbatch contains up to 90% by weight of the oil.

5. A method according to any one of the preceding claims wherein the crumbled rubber is dried by passing heated air through a bed of the rubber crumbs.

6. A method according to any one of the preceding claims for the production of oil-extended rubber in crumbled form wherein the masterbatch is employed in an amount such that the final product contains oil in an amount of up to 40% by weight.

7. A method according to any one of claims 1 to 5 for the production of crumbled rubber wherein the masterbatch is employed in an amount such that the final product contains oil in an amount of from 3 to 10% by weight.

8. The method for the production of crumbled rubber substantially as described with reference to Examples 1, 2 or 3.

9. The method for the production of oil-extended rubber in crumbled form substantially as described with reference to Example 4.

10. Crumbled rubber when produced by the method claimed in any one of claims 1 to 5, 7 and 8.

11. Oil-extended rubber in crumbled form when produced by the method claimed in any one of claims 1 to 6 and 9.

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	Oil	Rubber	Vulcanised Rubber
(a)	90	10	—
(b)	90	—	10
(c)	90	5	5
(d)	80	20	—
(e)	80	10	10
(f)	80	5	15
(g)	80	0	20

5 These masterbatches were then mixed on the crepeing mill with fresh wet natural latex coagula to produce a final concentration of 25% by weight of oil based on dry rubber content. In all cases at the end of 10 passes through the crepeing battery, the mass crumbled into fine particle size. These were then submitted to drying. All samples dried in 2 days at 140° F. The samples containing vulcanised rubber showed less tackiness and dried faster. Preliminary evaluation of rubbers incorporated with oil utilising this method showed that the physical properties are as good as those of oil-extended rubbers obtained by other methods of incorporating rubber extender oils into natural rubber. The process is equally effective with scrap grades of rubber.

WHAT WE CLAIM IS:—

20 1. A method of processing natural rubber which comprises milling a masterbatch comprising a wet coagulum of natural or synthetic rubber latex having a compatible oil dispersed therein with wet natural rubber in the form of a coagulum or solid mass, the milling being continued at least until the rubber crumbles.

25 2. A method according to claim 1 wherein the masterbatch is prepared by emulsifying or dispersing the oil in the latex and thereafter coagulating the resultant mixture of oil emulsion or dispersion and latex.

30 3. A method according to claim 1 or claim 2 wherein the natural or synthetic rubber latex is a vulcanised latex or a mixture of vulcanised and unvulcanised latices.

35 4. A method according to any one of the

preceding claims wherein the masterbatch contains up to 90% by weight of the oil.

5. A method according to any one of the preceding claims wherein the crumbled rubber is dried by passing heated air through a bed of the rubber crumbs. 40

6. A method according to any one of the preceding claims for the production of oil-extended rubber in crumbled form wherein the masterbatch is employed in an amount such that the final product contains oil in an amount of up to 40% by weight. 45

7. A method according to any one of claims 1 to 5 for the production of crumbled rubber wherein the masterbatch is employed in an amount such that the final product contains oil in an amount of from 3 to 10% by weight. 50

8. The method for the production of crumbled rubber substantially as described with reference to Examples 1, 2 or 3. 55

9. The method for the production of oil-extended rubber in crumbled form substantially as described with reference to Example 4. 60

10. Crumbled rubber when produced by the method claimed in any one of claims 1 to 5, 7 and 8.

11. Oil-extended rubber in crumbled form when produced by the method claimed in any one of claims 1 to 6 and 9. 65

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