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(54) A METHOD OF EXTENDING A NATURAL RUBBER WITH AN OIL.

(71) We, KUALA LUMPUR-KEPONG AMALGAMATED LIMITED, a Company incorporated in the United Kingdom, of 71 St. Mary Axe, London, E.C.3, 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:—

The present invention relates to a method of extending a natural rubber with an oil.

Although it has been shown that natural rubber extended with oil possesses unique and valuable properties, the manufacture of oil extended natural rubber has not flourished to any great extent due to the difficulties which have been encountered in known extension processes.

There are at present two known methods of incorporating oil as a softener or an extender into natural rubber. A first method is one in which an emulsion of the extender oil is mixed into the rubber latex prior to coagulation, so that on coagulation the oil is co-coagulated with the rubber. A second method is one in which oil is milled into dry rubber which has been masticated and made receptive to oil addition either on an open roller mill or in an internal mixing machine.

The method of adding an emulsion of the extender oil to the latex prior to coagulation offers several difficult problems. Firstly, it is necessary to use a rapid coagulation, which calls for excessive use of acid, in order to avoid separation of the oil and rubber phases prior to coagulation. Secondly, the coagulum formed, which even at such low extensions as 10 to 15% becomes too soft to pass through a conventional sheeting machine, is difficult to handle. Finally, the greatest difficulty is encountered in drying the sheets of extended and coagulated latex. Oil extended rubber made by this method has extremely poor "wet strength" in the undried state and will not hang on conventional drying trolleys as sheet or crepe, and packs down when made into crumb rubber

to such an extent as to prevent the flow of air, which is required in the drying process, from passing through it.

The method of adding oil to rubber which has been softened by mastication on a mill, although often used, is an expensive one since the power consumed in the breakdown of the dry rubber is considerable. Manufacturers are loath, therefore, to use this method as the cost may well outweigh the advantages of cheapness gained by incorporating a lower cost oil into the rubber. The cost of this method of oil extension puts natural rubber at a distinct disadvantage compared with ready made oil extended synthetic products.

When it is desired to oil extend any of the "lower grade" rubbers (i.e. rubbers made from the coagulum which has formed in tapping cups by fermentation and which arrive at the processing factory already in a coagulated condition) only the latter method, that of mill incorporation, may at present be used.

In a recently developed rubber processing technique rubber coagulum is comminuted to form what is commonly called a crumb, the crumb is dried, and the loose aggregates of particles from the dryer are subsequently pressed into blocks for transportation. As mentioned above where extension has been attempted prior to coagulation the coagulum is not entirely satisfactory for processing by this technique.

We have now found that substantially any grade of natural rubber may be extended with an oil in a relatively inexpensive manner by comminuting and drying a natural rubber coagulum and thereafter bringing the thus formed loose aggregate of rubber particles into intimate contact with a compatible extender oil.

Accordingly the present invention provides a method of extending a natural rubber with an oil, which method comprises comminuting and drying a natural rubber coagulum and thereafter bringing the thus formed loose aggregate of rubber particles

[Price 5s. 0d. (25p)]

SPECIFICATION AMENDED - SEE ATTACHED SLIP

into intimate contact with a compatible oil, the proportion of oil and the period of contact being such as to bring about the desired degree of extension. After extension the rubber may be pressed to a solid block for transportation.

By suitable selection of the proportion of oil used and the period of contact the method of the invention may be used to produce any degree of extension up to 100%.

Preferably the rubber is extended with the oil at an elevated temperature e.g. at from 50°C. to 100°C. This may conveniently be achieved by treating raw rubber by the method of the invention as soon as possible after drying.

In general raw rubber coagulum, of any grade, may be produced in divided form by any of the conventional comminuting methods, e.g. crumbing, pelletising, granulating or grinding. After the product has been dried and is still in an open structure form, that is unpressed, and usually and preferably unbroken, it is brought into intimate contact with the oil with which it is to be extended, and after a suitable period of time, which depends on the amount of extension required, the oil will be completely absorbed in the rubber particles, e.g. crumbs, which can then be pressed into a block for shipment in a conventional manner.

The oil may be brought into contact with the rubber by any suitable method, for example, the divided rubber, e.g. the rubber crumb, may be placed in perforated containers, the volume of which is calculated such that the containers hold a block of oil extended rubber of predetermined weight at a predetermined density. The weight of crumb calculated to be required as the rubber portion of the predetermined weight of oil extending rubber is placed on the container which is then immersed in the extending oil. Absorption of the oil takes place quite rapidly, particularly if the crumb is fresh from the dryers and still retains an elevated temperature. 48 hours has been found to be ample time when 100% extension is required. After removal from the container the oil extended rubber is ready for packing.

In addition non-perforated containers of predetermined size may be used in exactly the same manner as described above for perforated containers with the exception that in this case the oil is poured into the containers either before the crumb is put in or afterwards.

Where less than 50% extension is required for example, where the required extension is 25% or 33%, simple contacting as described above may not produce an extended rubber which has a consistent concentration

of oil throughout. If this is the case the extended rubber may not have the desired characteristics, for example, it may not have the desired appearance. Should simple contacting not produce an extended rubber in which the concentration of oil throughout is of the desired consistency, further processing may be necessary. For example, the extended rubber may conveniently and economically be further processed by the use of a blending mill.

Any rubber processing aid which is miscible with the oil used in the extension either in fluid or fine particle form, may be incorporated in the rubber/oil mixture by using the extender oil as a vehicle.

In addition one or more rubber compounding ingredients which are miscible with the oil used in the extension either in fluid or fine particle form, may also be incorporated in the rubber/oil mixture by using the extender oil as a vehicle.

The invention thus includes a composition e.g. a composition for use in the manufacture of tyre treads, comprising a natural rubber extended with an oil by the method of the invention with one or more rubber compounding ingredients.

Following is a description by way of example of methods in accordance with the invention.

EXAMPLE 1.

A quantity of natural rubber field latex was coagulated and processed by a method of comminution commonly known as "Pelletising". A block of crumb weighing 37.5 lbs. was removed from the drying tray and placed in a square metal container whilst still hot. To this was added 37.5 lbs. of a naphthenic mineral oil sold under the brand name of "Caltex Process Oil No. 150", by pouring the oil on to the rubber crumb.

After 24 hours the treated rubber was removed from the container and was found to be an homogeneous mixture from which the oil could not be removed without resort to solvent extraction.

EXAMPLE 2.

A quantity of natural rubber coagulum of the type known as "cup lump" i.e. a latex rubber which has coagulated whilst still in the tapping cup affixed to the rubber tree, was prepared by a crumbing method known under the name of the "mechano-crumbing process". On removal from the drying tray a block of crumb 37.5 lbs. in weight was placed in a square metal container whilst still hot. To this was added 37.5 lbs. of an aromatic mineral oil sold under the trade name of "Shell Dutrex R", by pouring the oil on to the rubber crumb. (The words "Shell Dutrex" are registered Trade Marks.)

After a lapse of 36 hours the treated rubber was discharged from the container directly on to a Cellophane (registered Trade Mark) sheet, wrapped, and packed directly into a cardboard carton for shipment. No oil exuded from the treated rubber, and oil could not be removed from the rubber without resort to solvent extraction.

EXAMPLE 3.

- 10 A quantity of natural rubber coagulum of the type known as "tree lace" i.e. latex rubber which has dried on the tapping cup of the rubber tree after latex flow has finished, was processed to crumb in a conventional manner and after removal from the drying tray a block weighing 37.5 lbs. was placed in a metal container whilst still hot. To this was added 37.5 lbs. of a naphthenic mineral oil sold under the brand name of "Shell Dutrex 65", by pouring the oil on to the rubber crumb.

After a lapse of 24 hours the treated

rubber was removed from the container and found to be an homogeneous mixture which could be wrapped in Cellophane (registered Trade Mark) for shipment. The oil could not be removed from the rubber without resort to solvent extraction.

The following Examples illustrate the utilization of oil extended rubber made by the method of the invention in the production of compositions for the manufacture of tyre treads.

EXAMPLE 4.

To compare the mixing cycles for oil extended rubber made by the method of the invention with two methods of mixing unmasticated natural rubber currently recommended by the Natural Rubber Producers Research Association and to show the time saved by using the oil extended rubber made by the method of the invention, the following composition was mixed in a high speed internal mixer:

Ingredients	Parts by Weight	
	(a)	(b) & (b ¹)
Oil extended rubber (50%/50%) by weight mixture) made by the method of the invention	100	—
Unmasticated natural rubber	—	50
Mineral oil	—	50
Zinc Oxide	5	5
Stearic Acid	2	2
Antioxidant	1	1
Carbon black	60	60
Accelerator	0.5	0.5
Sulphur	2.0	2.0

Comparative mixing cycles were as follows:—

(a) *Mixing of oil extended rubber*

- 60 0.0 minutes: Load oil extended rubber and all compounding ingredients.
1.5 Minutes: Dump on to sheeting mill.

(b) *Conventional mixing method recommended by NRPA.*

- 65 0.0 minutes: Load unmasticated rubber and all compounding ingredients with the exception of the mineral oil.
70 1.0 minutes: Load all oil.
2.5 minutes: Dump on to sheeting mill.

(b¹) *Alternative mixing method recommended by NRPA.*

- 75 0.0 minutes: Load unmasticated rubber
1.0 minutes: Load all powders and three quarters of the oil.
2.0 minutes: Load remainder of oil.
3.0 minutes: Dump on to sheeting mill.

EXAMPLE 5.

An oil extended rubber was produced by the method of the invention containing 15 lbs. of rubber and 10 lbs. of naphthenic oil was formulated as a typical motor car tyre tread composition containing the ingredients itemized as (a) in Table 1 below on an open two roll conventional mixing mill as follows:—

The oil extended rubber was added to an open two roll conventional mixing mill and all of the other ingredients were added as soon as the rubber had formed a band on the rollers. Mixing was completed in 10 minutes, and the composition had a Mooney viscosity (ML-4) of 33. The mixture was then sheeted off and fed to a conventional tyre tread extruder and a preformed tread strip obtained.

Visual examination showed carbon black dispersion to be good.

The preformed tyre tread strip was used to "retread" a worn motor tyre casing by moulding and vulcanising on to the pre-

pared carcass of the tyre. No fault could be found with the finished tyre, mould flow being good and no voids apparent in the moulded tyre tread pattern. There was no evidence of tread separation from the carcass.

An attempt was made to duplicate the above procedure except that oil extension was carried out during the mixing process after mastication of the rubber and the composition contained the ingredients itemized as (b) in Table 1 below.

Using this conventional method of oil extension on an open mill, i.e. by first masticating the raw rubber on to the mill to break it down to a consistency sufficient

to make it receptive to oil addition, the full amount of oil could only be added with difficulty due to the oxidative breakdown of the rubber having considerably increased the plasticity of the rubber and completion of compounding took three times as long as for the compounding which started with oil extended rubber.

The completed mix was of paste like consistency having Mooney viscosity (ML-4) below 18, and could only be extruded to a preformed tyre tread moulding strip with great difficulty. The preformed strip was not considered suitable for tyre retreading due to the poor moulding qualities, and softness.

TABLE 1.

Ingredients for oil extended tyre tread.						Parts by Weight.	
						(a)	(b)
Oil extended masterbatch	60% rubber	40% oil	...	...	...	100	
Ribbed Smoked Sheet Rubber	...	...	...	...	...	—	60
Napthenic Oil	...	...	...	...	...	—	40
Zinc Oxide	...	...	...	...	...	3	3
Stearic Acid	...	...	...	...	...	1	1
Accelerator	...	...	...	...	...	0.5	0.5
High Abrasion Furnace Black	...	...	...	...	...	55	55
Antioxidant	...	...	...	...	...	1.5	1.5
Sulphur	...	...	...	...	...	2	2

It will be appreciated from the above Examples that the method of the invention may be used to produce oil extended rubber from any type of natural rubber which is receptive to oil incorporation, i.e. substantially all grades of natural rubbers, which have been processed by any of the known comminution methods. In addition the method has the advantage that:

(a) No mechanical breakdown of the rubber is required subsequent to its production as a dry product, thus providing considerable power cost saving during manufacture to finished goods compounding.

(b) Oxidative breakdown attendant upon mechanical breakdown of the rubber in former dry rubber processes of oil extension is eliminated, thus imparting superior properties to the product.

WHAT WE CLAIM IS:—

1. A method of extending a natural rubber with an oil, which method comprises comminuting and drying a natural rubber coagulum and thereafter bringing the thus formed loose aggregate of rubber particles into intimate contact with a compatible oil,

the proportion of oil and the period of contact being such as to bring about the desired degree of extension.

2. A method as claimed in claim 1 when carried out at a temperature of from 50°C. to 100°C.

3. A method as claimed in claim 1 or claim 2, wherein one or more rubber compounding ingredients are incorporated in the rubber by using the oil as a vehicle for the ingredients.

4. A method as claimed in claim 1 and substantially as hereinbefore described specifically.

5. A method as claimed in claim 1 and substantially as hereinbefore described with reference to any one of Examples 1 to 3.

6. A method as claimed in claim 3 and substantially as hereinbefore described with reference to Example 4 or Example 5.

7. A natural rubber when extended with an oil by a method as claimed in any one of claims 1, 2, 4 or 5.

8. A composition comprising a natural rubber as claimed in claim 7 and one or more rubber compounding ingredients.

9. A composition as claimed in claim 8

pared carcass of the tyre. No fault could be found with the finished tyre, mould flow being good and no voids apparent in the moulded tyre tread pattern. There was no evidence of tread separation from the carcass.

An attempt was made to duplicate the above procedure except that oil extension was carried out during the mixing process after mastication of the rubber and the composition contained the ingredients itemized as (b) in Table 1 below.

Using this conventional method of oil extension on an open mill, i.e. by first masticating the raw rubber on to the mill to break it down to a consistency sufficient

to make it receptive to oil addition, the full amount of oil could only be added with difficulty due to the oxidative breakdown of the rubber having considerably increased the plasticity of the rubber and completion of compounding took three times as long as for the compounding which started with oil extended rubber.

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Zinc Oxide	...	...	...	...	...	...	...	3	3
Stearic Acid	...	...	...	...	...	...	...	1	1
Accelerator	...	...	...	...	...	...	...	0.5	0.5
High Abrasion Furnace Black	...	...	...	...	...	...	...	55	55
Antioxidant	...	...	...	...	...	...	...	1.5	1.5
Sulphur	...	...	...	...	...	...	...	2	2

It will be appreciated from the above Examples that the method of the invention may be used to produce oil extended rubber from any type of natural rubber which is receptive to oil incorporation, i.e. substantially all grades of natural rubbers, which have been processed by any of the known comminution methods. In addition the method has the advantage that:

(a) No mechanical breakdown of the rubber is required subsequent to its production as a dry product, thus providing considerable power cost saving during manufacture to finished goods compounding.

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the proportion of oil and the period of contact being such as to bring about the desired degree of extension.

2. A method as claimed in claim 1 when carried out at a temperature of from 50°C. to 100°C.

3. A method as claimed in claim 1 or claim 2, wherein one or more rubber compounding ingredients are incorporated in the rubber by using the oil as a vehicle for the ingredients.

4. A method as claimed in claim 1 and substantially as hereinbefore described specifically.

5. A method as claimed in claim 1 and substantially as hereinbefore described with reference to any one of Examples 1 to 3.

6. A method as claimed in claim 3 and substantially as hereinbefore described with reference to Example 4 or Example 5.

7. A natural rubber when extended with an oil by a method as claimed in any one of claims 1, 2, 4 or 5.

8. A composition comprising a natural rubber as claimed in claim 7 and one or more rubber compounding ingredients.

9. A composition as claimed in claim 8



when made by a method as claimed in claim
3 or claim 6.
10. A rubber tyre when retreaded with
a composition as claimed in claim 8 or
5 claim 9.

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