

PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements in the manufacture of raw natural rubber

We, SOCFIN COMPANY LIMITED, a Body Corporate duly organised under the laws of Malaya, of Bungsar Estate, Damansara Road, Kuala Lumpur, Malaya, 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 relates to the manufacture of raw natural rubber.

The amount of power required to masticate raw natural rubber prior to incorporation of compounding ingredients can be much reduced by adding so-called peptizing agents, which promote a rapid softening of the rubber on the mill. An even larger reduction in power consumption can be achieved by effecting this softening in the rubber largely at the time of preparation, by adding a peptizing agent to the rubber latex prior to coagulation, and drying the wet coagulum at temperatures in excess of 130°F, or at any other suitable temperature, until the required degree of softening has been achieved. Such a process as has been described for example in Specification No. 531,723 is, however, only applicable to initially liquid rubber latex.

30 A method has now been found whereby a peptizing agent can be incorporated into solid coagulum, obtained by natural coagulation of rubber latex, that is to say, into cuplump or bark scrap, or into rubber obtained by coagulation of rubber latex with chemicals such as formic, acetic, sulphuric or oxalic acid, or calcium chloride, prior to drying and despatch from the producing factory.

The method consists of disintegrating the coagulum into granules or crumbs, treating where necessary with a solution of a complexing agent to remove harmful metals such as copper and manganese, and spraying with a solution, or a water dispersion or an oil dispersion of a peptizing agent, if desired treating the sprayed granules or crumbs with steam according to the Specification of Application No. 32439/62 (Serial No. 964562) and then drying at temperatures at which the peptizing agent is sufficiently activated to effect softening in the time taken for drying.

The rubbers so obtained are softer than rubbers not prepared according to the invention and require less power for mastication than untreated rubbers.

In practice it has been found, however, that for bark scrap and other grades known to contain high concentration of manganese and other promoters of oxidation of raw rubbers, better quality rubbers are generally obtained if steps are first taken to reduce the concentration of manganese therein. This is achieved by soaking the raw materials in an aqueous solution of alkali and a known complexing agent such as ethylenediaminetetraacetic acid, prior to crepeing or scrap-washing.

The practice of the invention is illustrated by the following examples:—

EXAMPLE 1

Cup lumps and bark scrap were blended on a crepeing battery in the usual ratio for preparation of Estate Brown Crepe, until a 7 millimeter thick blanket was obtained; the bark scrap, which initially

[Price 4s. 6d.]

contained 80 parts per million manganese, having been soaked previously in water containing 0.15 w/v of sodium carbonate and 0.06% ethylenediaminetetraacetic acid, its manganese content being thereby reduced to 12 parts per million. This blanket was then fed to a Coste-Chevaleyre dicer consisting of a pair of grooved rolls which cut the blanket into filaments and rotary knives which sliced the latter into crumbs or granules of approximate dimensions $\frac{1}{4}'' \times \frac{1}{4}'' \times \frac{3}{8}''$.

The granules were then divided into several batches.

Batch A was dried directly at 140°F. to serve as a control.

Batch B was sprayed in a rotating drum, using a sprayer, such as a Four Oakes (Registered Trade Mark) sprayer, of the same type as employed for the spraying of insecticide dispersions with a dispersion made conveniently of:—

Renacit IV (Zinc thiopentachlorophenate) (1) ...	25 parts
Dutrex 32 (Rubber processing oil) (2) ...	75 parts
Carbon tetrachloride ...	50 parts

in such amounts that the final concentration of zinc thiopentachlorophenate in the dry rubber was 0.5%.

Batch C was sprayed in the same manner as batch B with a dispersion consisting of:—

Renacit IV (Zinc thiopentachlorophenate) ...	100 parts
Vultamol (Sodium salt of naphthalene sulphonic acid condensation products) (3) ...	5 parts
Vulkasil 30% (a 30% colloidal dispersion of silica) (4) ...	10 parts
Water ...	285 parts

in such amounts that the final concentration of zinc thiopentachlorophenate in the dry rubber was 0.5%.

Batches B and C were then treated with live steam according to the Specification of Application No. 32439/62 and dried at 140°F.

Batch D was sprayed with the same amount of the same dispersion as batch C, and dried directly at 140°F.

The Mooney values of these batches when dry were determined according to A.S.T.M. method D.927-55T and the results were:

Batch	A	B	C	D
Mooney value ML. 1+4 (212°F.)	82	58	52	54

(1) Bayer A.G. Registered Trade Mark.

(2) Shell Co. Ltd Registered Trade Mark.

(3) B.A.S.F.

(4) Bayer A.G.

The dry rubbers in batches B, C and D are obviously much softer than batch A, the control.

EXAMPLE 2

Cup lumps and bark scrap, the latter having been treated as in Example 1 to remove copper and manganese, were blended in a scrap washer and disintegrated on a Bauer attrition mill. The resulting granules were sprayed, with an ordinary spray gun, with 0.6% v/w of a solution of xylylmercaptan in an inert aromatic solvent; R.P.A. No. 3* is such a solution.

The granules were then dried at 140°F. and had, when dry, a Mooney value of 48, whereas untreated granules had a Mooney value of 78.

EXAMPLE 3

Rubber latex was coagulated with formic acid; the coagulum was milled on a crepeing battery, then disintegrated on a Coste-Chevaleyre dicer.

The granules were sprayed with a dispersion of:

Zinc thiopentachlorophenate	100 parts
Vultamol (1) ...	5 parts
Vulkasil (2) ...	10 parts
Water ...	285 parts

in such amounts that the final concentration of zinc thiopentachlorophenate in dry rubber was 0.35%.

The sprayed granules were then treated with steam according to the Specification of Application No. 32439/62 and subsequently dried in an air stream at 195°F.

When dry the Mooney value of the rubber was 38, whereas that of granules treated identically, but not sprayed, was 68.

Applicants have also found that the peptizing agent can be added during the preparation of the crepe, as a dispersion in mineral or vegetable oil, or as a masterbatch consisting of mineral or vegetable oil, peptizing agent and rubber. The peptizing agent is well distributed throughout the rubber to be peptized during subsequent crepeing. By selecting a suitable combination of peptizer and oil, a water-insoluble mixture is formed which has great affinity for rubber, and which will not be leached out during crepeing operations.

The process consists in adding either the mixture of peptizing agent with oil to rubber coagulum on the scrap-washer, or the masterbatch of oil, peptizing agent

* Dupont de Nemours.

(1) B.A.S.F.

(2) Bayer A.G.

and rubber on the scrap-washer or on the first roll of a standard estate crepeing battery, proceeding with crepeing operations as usual, putting the resulting crepe on trays or disintegrating it in a dicing machine, subjecting it to a steam-treatment according to British Patent Application No. 32439/62 (Serial No. 964562) and then drying it at a temperature sufficient to render the peptizing agent active.

The following examples illustrate the practice of the invention according to this procedure:—

EXAMPLE 4

Fifty grams of Renacit IV (Zinc thiopentachlorophenate supplied by Bayer A.G. and 50 grams of a non-staining rubber processing oil, supplied by The Shell Company, are blended into a smooth paste in a high-speed stirrer. This paste is added to 25 kg. of rubber-coagulum whilst the latter is being worked in the scrap-washer. The washed scrap is then creped as usual, until a blanket of about 2 cm. thickness is obtained. This blanket is laid on trays, subjected to live steam for five minutes, and then dried at 140°F.

The Mooney plasticity of the dry crepe was 45; crepe treated in the same manner, but without addition of peptizing agent, had a plasticity of 80. The dry rubber treated with peptizing agent according to the invention is obviously much softer.

EXAMPLE 5

Fifty grams of Renacit IV and 50 grams of rubber processing oil were blended as in Example 4, and then incorporated into 150 grams of wet crepe rubber by milling on a small crepeing roller. The resulting masterbatch was added to 25 kg. of scrap-washed rubber coagulum on the first pair of rollers of a crepeing battery and creped as usual. The resulting crepe was disintegrated on a Coste-Chavaleyre dicer, loaded on trays, steamed for five minutes at atmospheric pressure, and dried at 140°F. The dry crepe had a Mooney plasticity of 42.

EXAMPLE 6

Fifty grams of Renacit IV and 50 grams of palm oil were blended on a high speed stirrer, and then made into a masterbatch as in Example 5, subsequent operations being also as in Example 5. The Mooney plasticity was 37.

The invention comprehends not only the process for incorporating peptizing agent into rubber coagulum by the addition of mixtures of peptizing agent and mineral or vegetable oil on a scrap-washer, or crepeing rolls, or by the addition of masterbatches of mineral or vegetable oil, peptizing agent and rubber, followed by drying

at a temperature at which the peptizing agent is rendered active, but also the product obtained, which is considerably softer than ordinary crepe.

It will be appreciated that the invention also comprehends natural rubber resulting from the treatment of disintegrated coagulum which has undergone preliminary treatment with a complexing agent to remove harmful metals such as manganese when required, with peptizing agents either in solution, water or oil dispersion, followed by drying at temperatures which promote the action of the peptizing agent.

WHAT WE CLAIM IS:

1. A method of manufacturing natural rubber consisting in disintegrating coagulum into granules or crumbs, treating where necessary with a solution of complexing agent to remove harmful metals such as copper and manganese, and spraying with a solution, or a water dispersion or an oil dispersion of a peptizing agent, and then drying at temperatures at which the peptizing agent is sufficiently activated to effect softening in the time taken for drying.

2. A method of manufacturing natural rubber according to claim 1 wherein the sprayed granules or crumbs are treated with steam according to the Specification of co-pending Application No. 32439/62 (Serial No. 964562) prior to drying.

3. A method of manufacturing natural rubber according to either of the preceding claims 1 or 2 wherein manganese and other oxidation promoters are reduced by soaking the raw material in an aqueous solution of alkali and a complexing agent such as ethylenediaminetetraacetic acid prior to crepeing or scrap washing.

4. A modification of the method of manufacturing natural rubber according to the preceding claims wherein the peptizing agent is added during the preparation of crepe, as a dispersion in mineral or vegetable oil, or as a masterbatch consisting of mineral or vegetable oil, peptizing agent and rubber.

5. A method according to claim 4 wherein either the mixture of peptizing agent and oil, or the masterbatch is added to the rubber on the scrap washer or on the first roll of the crepeing battery.

6. A method of manufacturing natural rubber according to Example 1 substantially as described.

7. A method of manufacturing natural rubber according to Example 2 substantially as described.

8. A method of manufacturing natural rubber according to Example 3 substantially as described.

ethylenediaminetetraacetic

ENT
2108

A -

ENT
1008

chavaleyre

ENT
08

peptizing



and rubber on the scrap-washer or on the first roll of a standard estate crepeing battery, proceeding with crepeing operations as usual, putting the resulting crepe on trays or disintegrating it in a dicing machine, subjecting it to a steam-treatment according to British Patent Application No. 32439/62 (Serial No. 964562) and then drying it at a temperature sufficient to render the peptizing agent active.

The following examples illustrate the practice of the invention according to this procedure:—

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EXAMPLE 6

Fifty grams of Renacit IV and 50 grams of palm oil were blended on a high speed stirrer, and then made into a masterbatch as in Example 5, subsequent operations being also as in Example 5. The Mooney plasticity was 37.

The invention comprehends not only the process for incorporating peptizing agent into rubber coagulum by the addition of mixtures of peptizing agent and mineral or vegetable oil on a scrap-washer, or crepeing rolls, or by the addition of masterbatches of mineral or vegetable oil, peptizing agent and rubber, followed by drying

at a temperature at which the peptizing agent is rendered active, but also the product obtained, which is considerably softer than ordinary crepe.

It will be appreciated that the invention also comprehends natural rubber resulting from the treatment of disintegrated coagulum which has undergone preliminary treatment with a complexing agent to remove harmful metals such as manganese when required, with peptizing agents either in solution, water or oil dispersion, followed by drying at temperatures which promote the action of the peptizing agent.

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1. A method of manufacturing natural rubber consisting in disintegrating coagulum into granules or crumbs, treating where necessary with a solution of complexing agent to remove harmful metals such as copper and manganese, and spraying with a solution, or a water dispersion or an oil dispersion of a peptizing agent, and then drying at temperatures at which the peptizing agent is sufficiently activated to effect softening in the time taken for drying.

2. A method of manufacturing natural rubber according to claim 1 wherein the sprayed granules or crumbs are treated with steam according to the Specification of co-pending Application No. 32439/62 (Serial No. 964562) prior to drying.

3. A method of manufacturing natural rubber according to either of the preceding claims 1 or 2 wherein manganese and other oxidation promoters are reduced by soaking the raw material in an aqueous solution of alkali and a complexing agent such as ethylenediaminetetraacetic acid prior to crepeing or scrap washing.

4. A modification of the method of manufacturing natural rubber according to the preceding claims wherein the peptizing agent is added during the preparation of crepe, as a dispersion in mineral or vegetable oil, or as a masterbatch consisting of mineral or vegetable oil, peptizing agent and rubber.

5. A method according to claim 4 wherein either the mixture of peptizing agent and oil, or the masterbatch is added to the rubber on the scrap washer or on the first roll of the crepeing battery.

6. A method of manufacturing natural rubber according to Example 1 substantially as described.

7. A method of manufacturing natural rubber according to Example 2 substantially as described.

8. A method of manufacturing natural rubber according to Example 3 substantially as described.



ethylenediaminetetraacetic acid

chevalleyre

peptizing

9. A method of manufacturing natural rubber according to Example 4 substantially as described.

5 10. A method of manufacturing natural rubber according to Example 5 substantially as described.

11. A method of manufacturing natural rubber according to Example 6 substantially as described.

10 12. A method of manufacturing softened rubbers substantially as described.

13. Softened natural rubber when obtained by the method according to any one or more of the preceding claims substantially as described.

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