

Singapore (1)

1.122.463



PATENT SPECIFICATION

NO DRAWINGS

1.122.463

Inventor: BALACHANDRA CHAKKINGAL SEKHAH

Date of filing Complete Specification: 10 Nov., 1966.

Application Date: 16 Nov., 1965.

No. 48702/65.

Complete Specification Published: 7 Aug., 1968.

© Crown Copyright 1968.

Index at acceptance:—C3 E(1D, 1X)

Int. Cl.:—C 08 c 1/14, C 08 c 3/00

COMPLETE SPECIFICATION

Improvements in or relating to the preparation of Lower Grade Natural Rubbers and Skim Rubbers

5 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 relates to the treatment of natural rubber derived from unvulcanised skim rubbers (a by-product of latex centrifugation), and the lower grade rubbers, viz. cup lumps (the coagulum formed in the tapping cup after normal collection) and tree lace (the tree exudate that coagulates on the tapping cut).

15 In the preparation of dry raw natural rubber in the plantation for export, a number of source materials are used. These can be broadly divided into latex grades, lower grades and skim rubbers. Rubbers derived from normal natural rubber latex possess excellent ageing characteristics and present no problem during drying, storage or use. On the other hand, rubbers from the lower grades, i.e. cup lumps and tree lace, vary in ageing behaviour depending on several factors such as (a) exposure to sunlight, (b) period of storage prior to processing, (c) contamination e.g. with trace metals. 20 With skim rubbers, the ageing behaviour is very poor and degradation can occur during drying and subsequent use.

25 Recent developments in new processing methods have made it necessary to dry raw natural rubbers at elevated temperatures for (c) contamination e.g. with trace metals. With ordinary latex rubbers such processes, involving drying temperatures of 100°C., have had no detrimental effect on their ageing behaviour. On the other hand, the lower grade rubbers and particularly skim rubbers are prone to appreciable oxidation under similar conditions. Such rubbers are prone to oxidation and also show poor properties in the vulcanised 30

state particularly when submitted to the usual ageing tests. 45

Oxidation of natural rubber in any form can be caused by several factors such as (1) exposure to oxygen in the latex state, (2) presence of non-rubbers prone to oxidation, (3) exposure to sunlight and (4) metallic contamination. Methods for the prevention of air oxidation and the activation by metallic contamination are known for normal natural rubber. The former can be prevented by the addition of tetraethylene pentamine, while the latter can be overcome with metal chelating agents, such as ethylenediamine tetra-acetic acid. However, neither of these methods when applied to skim rubbers or lower grade rubbers have satisfactorily improved their ageing characteristics to levels deemed necessary for certain uses particularly where dynamic properties are important. We have now discovered that these rubbers can be greatly improved by treatment with a solution of oxalic acid. 50 55 60 65

According to the present invention therefore there is provided a method of improving the raw ageing characteristics of lower grade rubbers and skim rubber which comprises bringing said rubbers into intimate contact with oxalic acid in aqueous solution in an amount preferably ranging from 0.01 to 1.0% of the solid oxalic acid based on dry rubber content by weight. The rubbers may be in finely divided form i.e. in the form of a latex or in dry comminuted form. The solid rubbers are treated by being left in contact with the oxalic acid for several hours but the skim rubber latex may be coagulated by the use of oxalic acid and the coagulum so produced then granulated to produce a rubber of improved ageing characteristics. 70 75 80

The manner in which the invention may be carried into effect is illustrated by the following examples in which the percentage figures quoted are calculated by weight. 85

[Price 4s. 6d.]

With economy in each and every processing.



EXAMPLE I

5 Fresh cup lumps were collected and cut up into small pieces. This was then divided into lots and a number of the lots were allowed to soak in solutions of oxalic acid ranging in concentration from 0.2 to 0.5%, for a period of 12 hours. The remaining lots were the control lots which were allowed to soak in water for the same period. ~~the~~ the samples were 10 subsequently granulated, using the crumbling

process described in British Patent Specification No. 1075235 and dried at 100°C. in 3 hours. The resulting dry rubbers were then subjected to an ageing test. The test consists in heating the rubber of fixed thickness (by milling) at a temperature of 140°C. for 30 minutes. The plasticity is measured before and after ageing. The percentage of the plasticity retained is a measure of the oxidisability of the rubber

Treatment	Plasticity		Plasticity Retention Index $\frac{B}{A} \times 100$
	Before Ageing (A)	After Ageing (B)	
Nil	43	32	74
0.2% Oxalic Acid	35	34	97
0.5% Oxalic Acid	32	32	100

EXAMPLE II

Tree lace was taken and submitted to exactly

the same procedure as in Example I. The results obtained were as follows:—

Treatment	Plasticity		Plasticity Retention Index $\frac{B}{A} \times 100$
	Before Ageing (A)	After Ageing (B)	
Nil	39	15	38
0.2% Oxalic Acid	39	18	46
0.5% Oxalic Acid	40	22	55

EXAMPLE III

30 Skim latex was divided into portions and the following treatments were carried out on the separate portions:—

- (a) Coagulate with formic acid in the usual manner, granulate and dry at 100°C.
 (b) Coagulate with oxalic acid, granulate and dry at 100°C.
 35 (c) Coagulate with formic acid, granulate,

the granules were then soaked in 0.2% aqueous oxalic acid solution for 12 hours, wash and dry at 100°C.

(d) Add ethylenediamine tetra-acetic acid (0.2% on rubber), coagulate with formic acid, granulate and dry at 100°C.

All the 4 samples were then submitted to the ageing test as in Examples I and II. The results obtained were as follows:—



aqueous

Portion	Plasticity		Plasticity Retention Index B ($\frac{B}{A} \times 100$)
	Before Ageing (A)	After Ageing (B)	
a	54	5	9
b	59	48	81
c	53	48	91
d	57	42	74

5 The present invention thus provides a satisfactory method for improving the raw ageing characteristics of skim rubbers and lower grade natural rubbers. The method is suitable for natural rubber cup lumps, tree lace and particularly for rubbers derived from skim latex and can be applied to the rubber whether in the form of a latex, a wet coagulum or in dry comminuted form.

10 The plasticity measurements were carried out with the aid of the Wallace type plastimeter described in the Transactions of the Institute of the Rubber Industry Volume 29, No. 4 (August 1953) on page 186.

15 **WHAT WE CLAIM IS:—**

20 1. A method of improving the raw ageing characteristics of lower grade rubbers and skim rubbers which comprises bringing said rubbers into intimate contact with oxalic acid in aqueous solution.

2. A method according to claim 1 wherein the rubber is in a finely divided form.

25 3. A method according to claim 2 wherein the rubber is in the form of dry comminuted rubber.

4. A method according to claim 1 wherein the rubber is in the form of a wet coagulum.

5. A method according to claim 4 wherein the coagulum is produced by the addition of an acid to skim rubber. 30

6. A method according to any of the preceding claims wherein the rubber is soaked in the oxalic acid solution for a period of several hours. 35

7. A method according to claim 2 wherein a skim rubber latex is coagulated by the addition of an aqueous solution of oxalic acid.

8. A method according to any of the preceding claims wherein the oxalic acid is employed in an amount of from 0.01% to 1.0% of the solid oxalic acid by weight of the dry rubber content of the rubber undergoing treatment. 40

9. The method for improving the characteristics of skim rubber and lower grade rubbers by treatment with oxalic acid substantially as described in any of the Examples. 45

10. Lower grade rubbers and skim rubbers when treated by the method claimed in any of the preceding claims. 50

STEVENS, LANGNER, 
PARRY & ROLLINSON,
Chartered Patent Agents,
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1968.
Published by the Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.

*1 economy in cost
and 1.5 times
more?*

Portion	Plasticity		Plasticity Retention Index $\frac{B}{A} \times 100$
	Before Ageing (A)	After Ageing (B)	
a	54	5	9
b	59	48	81
c	53	48	91
d	57	42	74

The present invention thus provides a satisfactory method for improving the raw ageing characteristics of skim rubbers and lower grade natural rubbers. The method is suitable for natural rubber cup lumps, tree lace and particularly for rubbers derived from skim latex and can be applied to the rubber whether in the form of a latex, a wet coagulum or in dry comminuted form.

The plasticity measurements were carried out with the aid of the Wallace type plastimeter described in the Transactions of the Institute of the Rubber Industry Volume 29, No. 4 (August 1953) on page 186.

WHAT WE CLAIM IS:—

1. A method of improving the raw ageing characteristics of lower grade rubbers and skim rubbers which comprises bringing said rubbers into intimate contact with oxalic acid in aqueous solution.

2. A method according to claim 1 wherein the rubber is in a finely divided form.

3. A method according to claim 2 wherein the rubber is in the form of dry comminuted rubber.

4. A method according to claim 1 wherein the rubber is in the form of a wet coagulum.

5. A method according to claim 4 wherein the coagulum is produced by the addition of an acid to skim rubber.

6. A method according to any of the preceding claims wherein the rubber is soaked in the oxalic acid solution for a period of several hours.

7. A method according to claim 2 wherein a skim rubber latex is coagulated by the addition of an aqueous solution of oxalic acid.

8. A method according to any of the preceding claims wherein the oxalic acid is employed in an amount of from 0.01% to 1.0% of the solid oxalic acid by weight of the dry rubber content of the rubber undergoing treatment.

9. The method for improving the characteristics of skim rubber and lower grade rubbers by treatment with oxalic acid substantially as described in any of the Examples.

10. Lower grade rubbers and skim rubbers when treated by the method claimed in any of the preceding claims.

STEVENS, LANGNER,
PARRY & ROLLINSON,
Chartered Patent Agents,
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1968.
Published by the Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.

