PATENT SPECIFICATION 743,139

Inventor :—WILLIAM HENRY BAGNALL.

Date of filing Complete Specification : June 9, 1954.

Application Date : July 2, 1953. No. 18394/53.

Complete Specification Published : Jan. 11, 1956.

Index at Acceptance :—**Class 70**, E(6A : 17 : 18 : 19).

COMPLETE SPECIFICATION.

Process of Treating Dilute Aqueous Rubber Dispersions.

We, REVERTEX LIMITED, formerly of King William Street House, Arthur Street, London, E.C.4, now of 51 to 55 Strand, London, W.C.2, a British Company, 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 :—

This invention relates to a process of treating dilute aqueous rubber dispersions and more particularly to the treatment of dilute aqueous rubber dispersions obtained in the centrifuging and creaming treatments of rubber latices.

In our Specification No. 553,249, we have described a process of treating rubber latex skim which comprises flocculating the rubber content of the skim with a suitable flocculating agent, thereupon filtering the whole, leaving the flocculated rubber as residue, washing the flocculated rubber in order to remove non-rubber constituents and impurities therefrom and thereafter converting the washed flocculated rubber into an aqueous dispersion by means of water containing an alkaline dispersing agent, such as caustic alkali or ammonia, to form an alkaline latex.

It has now been discovered that when the skim has been flocculated by an acid flocculating agent and the aqueous non-rubber constituents and impurities have been removed by filtration or settling, it is possible, by adding a non-ionogenic surface-active agent to the flocculated rubber, to redisperse said flocculated rubber whilst still acid and without previous treatment with alkali, to form a stable acid latex.

Based on the foregoing discovery, the present invention provides a process of treating dilute aqueous rubber dispersions and especially rubber latex skim, which comprises flocculating the rubber content of the dilute dispersion with a flocculating agent, separating the aqueous non-rubber constituents and impurities from the flocculated dispersion leaving the flocculated rubber as residue, thereupon adding a non-ionogenic surface-active agent to the flocculated rubber while it is still acid thereby redispersing said flocculated rubber to form a stable acid latex.

The non-ionogenic surface-active agent employed in carrying out the present invention should be one which does not attach the rubber and which is at the same time unaffected by the acid present in the flocculated skim.

The surface-active agents preferably employed in carrying out the invention are condensation products of polyethylene glycols with long chain organic carboxylic acids, or with long chain organic alcohols or with long chain organic amines or with long chain organic amides or alkyl phenols.

Examples of surface-active agents of the foregoing kind are :—

A condensation product of a long chain fatty alcohol and ethylene oxide, known under the Registered Trade Mark " Lubrol W " ;

A polyethylene glycol ether of octyl phenol known under the Registered Trade Mark " Lissapol N " ; and

A condensation product of ethylene oxide and cetyl alcohol known under the Registered Trade Mark " Vulcastab LW " (all manufactured by Imperial Chemical Industries Limited) ;

A condensation product of oleyl alcohol and ethylene oxide known under the Trade Name " Emulphor O " (manufactured by General Dyestuff Corp., 435, Hudson St., New York, N.Y.) ;

A fatty acid ester of polyethylene glycol, known under the Trade Name " Ethofat " ;

A di-polyoxyethylene fatty alkyl amine known under the Registered Trade Mark " Ethomeen " ; and

[*Price* 3*s.* 0*d.*]

An N,N-polyoxyethylene fatty acid amine known under the Registered Trade Mark "Ethomids" (all manufactured by Armour & Co., 1355 W. 31st Street, Chicago, Ill.); and

A polyethylene ether of N-dodecyl diethanolamine known under the Trade Name "Stabiliser 12NR50" (manufactured by Oxirane Limited).

The amount of non-ionogenic surface-active agent employed may advantageously be between 0.5 and 5% by weight calculated on the total solids content of the latex and preferably between 0.5 and 2% of such total solids content.

For the purpose of the present invention it is immaterial whether a very small amount of a strong acid or larger amounts of a weak acid are employed to bring the latex to the *p*H required for flocculation. The acids employed may be sulphuric acid, hydrochloric acid and the lower carboxylic acids e.g. formic or acetic acid. However, the nature of the acids employed and/or their concentration should be such that they do not attack either the rubber or the surface-active agent.

The acid latex produced on redispersion of the flocculated rubber has a concentration and stability suitable for enabling it to be treated with acidic agents, for example, for chemically modifying the rubber contained therein. Thus, for example, the resultant latex can be treated with concentrated sulphuric acid to yield a cyclised latex or it may be reacted with hydrochloric acid either in the form of a gas or concentrated solution to produce a rubber hydrochloride in the form of a latex. Alternatively, the latex can be treated with chlorine with or without previous treatment with hydrochloric acid to produce a chlorinated rubber. In addition, the resultant latex can be treated with sulphur dioxide to form a rubber-sulphur dioxide adduct or with acidic oxidants to produce resinified rubbers. Finally, the latex can be added to acid dispersions of rubber, modified rubber, or polymers.

The acid latex dispersion produced in accordance with the present invention can, if desired, be subsequently rendered alkaline, for example, by any suitable fixed alkali or ammonia.

The invention will be illustrated by the following Examples :—

EXAMPLE I.

1 litre of a skim latex, resulting from the centrifugal concentration of vulcanised natural rubber latex, and having a total solids content of 8.2% and a dry rubber content of 3.9%, was flocculated by the addition of sufficient 25% sulphuric acid solution to bring the mixture to a *p*H of 3.6.

The flocculated rubber was filtered, washed and drained. Upon stirring in 0.6 gms. of the non-ionogenic stabiliser, known under the Registered Trade Mark "Lubrol W," the flocs dispersed to a concentrated latex having a total solids content of 21% and a slightly acid reaction, which latex was found to withstand the addition of concentrated sulphuric acid without coagulating.

EXAMPLE II.

1 litre of a skim latex resulting from the centrifugal concentration of vulcanised natural rubber latex and having a total solids content of 8.2% was flocculated by the addition of 10% sulphuric acid solution to reduce the *p*H to 4. After filtering, washing and allowing to drain, the flocculated rubber had a volume of 385 mls., being of a cheese-like consistency.

Upon stirring in 0.5 gm. of Ethomeen 1860 a fluid concentrated latex of 21% total solids content was obtained which was stable to additions of concentrated sulphuric acid.

What we claim is :—

(1) Process of treating dilute aqueous rubber dispersions and especially rubber latex skim, which comprises flocculating the rubber content of the dilute dispersion with a flocculating agent, separating the aqueous non-rubber constituents and impurities from the flocculated dispersion leaving the flocculated rubber as residue, thereupon adding a non-ionogenic surface-active agent to the flocculated rubber while it is still acid thereby redispersing said flocculated rubber to form a stable acid latex.

(2) Process as claimed in Claim 1, in which the surface-active agent employed is a condensation product of polyethylene glycol with a long chain carboxylic acid or with a long chain alcohol, or with a long chain organic amine or with a long chain organic amide or with a long chain alkyl phenol.

(3) Process as claimed in Claim 1 or 2, in which the amount of non-ionogenic surface-active agent employed lies between 0.5 and 5% by weight calculated on the total solids content of the rubber dispersion and preferably between 0.5 and 2% of said total solids content.

(4) Process of treating dilute aqueous rubber dispersions, substantially as described with reference to the foregoing Examples.

W. H. A. THIEMANN,
Prestige House,
14 to 18 Holborn, London, E.C.1.
Agent for the Applicants.

An N,N-polyoxyethylene fatty acid amine known under the Registered Trade Mark "Ethomids" (all manufactured by Armour & Co., 1355 W. 31st Street, Chicago, Ill.); and

A polyethylene ether of N-dodecyl diethanolamine known under the Trade Name "Stabiliser 12NR50" (manufactured by Oxirane Limited).

The amount of non-ionogenic surface-active agent employed may advantageously be between 0.5 and 5% by weight calculated on the total solids content of the latex and preferably between 0.5 and 2% of such total solids content.

For the purpose of the present invention it is immaterial whether a very small amount of a strong acid or larger amounts of a weak acid are employed to bring the latex to the *p*H required for flocculation. The acids employed may be sulphuric acid, hydrochloric acid and the lower carboxylic acids e.g. formic or acetic acid. However, the nature of the acids employed and/or their concentration should be such that they do not attack either the rubber or the surface-active agent.

The acid latex produced on redispersion of the flocculated rubber has a concentration and stability suitable for enabling it to be treated with acidic agents, for example, for chemically modifying the rubber contained therein. Thus, for example, the resultant latex can be treated with concentrated sulphuric acid to yield a cyclised latex or it may be reacted with hydrochloric acid either in the form of a gas or concentrated solution to produce a rubber hydrochloride in the form of a latex. Alternatively, the latex can be treated with chlorine with or without previous treatment with hydrochloric acid to produce a chlorinated rubber. In addition, the resultant latex can be treated with sulphur dioxide to form a rubber-sulphur dioxide adduct or with acidic oxidants to produce resinified rubbers. Finally, the latex can be added to acid dispersions of rubber, modified rubber, or polymers.

The acid latex dispersion produced in accordance with the present invention can, if desired, be subsequently rendered alkaline, for example, by any suitable fixed alkali or ammonia.

The invention will be illustrated by the following Examples:—

EXAMPLE I.

1 litre of a skim latex, resulting from the centrifugal concentration of vulcanised natural rubber latex, and having a total solids content of 8.2% and a dry rubber content of 3.9%, was flocculated by the addition of sufficient 25% sulphuric acid solution to bring the mixture to a *p*H of 3.6.

The flocculated rubber was filtered, washed and drained. Upon stirring in 0.6 gms. of the non-ionogenic stabiliser, known under the Registered Trade Mark "Lubrol W," the flocs dispersed to a concentrated latex having a total solids content of 21% and a slightly acid reaction, which latex was found to withstand the addition of concentrated sulphuric acid without coagulating.

EXAMPLE II.

1 litre of a skim latex resulting from the centrifugal concentration of vulcanised natural rubber latex and having a total solids content of 8.2% was flocculated by the addition of 10% sulphuric acid solution to reduce the *p*H to 4. After filtering, washing and allowing to drain, the flocculated rubber had a volume of 385 mls., being of a cheese-like consistency.

Upon stirring in 0.5 gm. of Ethomeen 1860 a fluid concentrated latex of 21% total solids content was obtained which was stable to additions of concentrated sulphuric acid.

What we claim is:—

(1) Process of treating dilute aqueous rubber dispersions and especially rubber latex skim, which comprises flocculating the rubber content of the dilute dispersion with a flocculating agent, separating the aqueous non-rubber constituents and impurities from the flocculated dispersion leaving the flocculated rubber as residue, thereupon adding a non-ionogenic surface-active agent to the flocculated rubber while it is still acid thereby redispersing said flocculated rubber to form a stable acid latex.

(2) Process as claimed in Claim 1, in which the surface-active agent employed is a condensation product of polyethylene glycol with a long chain carboxylic acid or with a long chain alcohol, or with a long chain organic amine or with a long chain organic amide or with a long chain alkyl phenol.

(3) Process as claimed in Claim 1 or 2, in which the amount of non-ionogenic surface-active agent employed lies between 0.5 and 5% by weight calculated on the total solids content of the rubber dispersion and preferably between 0.5 and 2% of said total solids content.

(4) Process of treating dilute aqueous rubber dispersions, substantially as described with reference to the foregoing Examples.

W. H. A. THIEMANN,
Prestige House,
14 to 18 Holborn, London, E.C.1,
Agent for the Applicants.

PROVISIONAL SPECIFICATION.

Process of Treating Dilute Aqueous Rubber Dispersions.

We, REVERTEX LIMITED, of King William Street House, Arthur Street, London, E.C.4, a British Company, do hereby declare this invention to be described in the following statement :—

This invention relates to a process of treating dilute aqueous rubber dispersions and more particularly to the treatment of dilute aqueous rubber dispersions obtained in the centrifuging and creaming treatments of rubber latices.

In our Specification No. 553,249, we have described a process of treating skim rubber which comprises flocculating the rubber content of the skim with a suitable flocculating agent, thereupon filtering the whole, leaving the flocculated rubber as residue, washing the flocculated rubber in order to remove non-rubber constituents and impurities therefrom and thereafter converting the washed flocculated rubber into an aqueous dispersion by means of water containing an alkaline dispersing agent, such as caustic alkali or ammonia, to form an alkaline latex.

It has now been discovered that when the skim has been flocculated by an acid flocculating agent and the aqueous non-rubber constituents and impurities have been removed by filteration or settling, it is possible, by adding a non-ionogenic surface-active agent to the flocculated rubber, to redisperse said flocculated rubber whilst still acid and without previous treatment with alkali, to form a stable acid latex.

The non-ionogenic surface active agent employed in carrying out the present invention should be one which does not attack the rubber and which is at the same time unaffected by the acid present in the flocculated skim.

The surface-active agents preferably employed in carrying out the invention are condensation products of polyethylene glycols with long chain organic carboxylic acids, or with long chain organic alcohols or with long chain organic amines or with long chain organic amides.

Examples of surface-active agents of the foregoing kind are known under the Registered Trade Mark " Lubrol W " (manufactured by Imperial Chemical Industries Limited) and the Trade Name " Ethofats " (manufactured by Armour & Company).

The acid latex produced on redispersion of the flocculated rubber has a concentration and stability suitable for enabling it to be treated with acidic agents, for example, for chemically modifying the rubber contained therein. Thus, for example, the resultant latex can be treated with concentrated sulphuric acid to yield a cyclised latex or it may be reacted with hydrochloric acid either in the form of a gas or concentrated solution to produce a rubber hydrochloride in the form of a latex. Alternatively, the latex can be treated with chlorine with or without previous treatment with hydrochloric acid to produce a chlorinated rubber. In addition, the resultant latex can be treated with sulphur dioxide to form a rubber-sulphur dioxide adduct or with acidic oxidants to produce resinified rubbers. Finally, the latex can be added to acid dispersions of rubber, modified rubber, or polymers.

The acid latex dispersion produced in accordance with the present invention can, if desired, be subsequently rendered alkaline, for example, by any suitable fixed alkali or ammonia.

W. H. A. THIEMANN,
Prestige House,
14–18 Holborn, London, E.C.1.
Agent for the Applicants.

Abingdon : Printed for Her Majesty's Stationery Office, by Burgess & Son (Abingdon), Ltd.—1955.
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2,
from which copies may be obtained.