PATENT SPECIFICATION

612,443

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PROVISIONAL SPECIFICATION

Improvement in the Treatment of Natural Rubber Latices

I, JAN WILLEM VAN DALFSEN, of 10, Hertog, Govertkade, Delft, Holland, a Dutch subject, do hereby declare the nature of this invention to be as follows:—

The invention concerns an improvement in the treatment of natural rubber latices and may be applied in the manufacture of preserved latex, or of concentrated latex or of dry rubber with special properties, such as a low water absorption, a high plasticity and a white colour.

It is known that upon addition to fresh natural latex of only small amounts of acid, or of a salt of a polyvalent cation, a so called precoagulation follows. Only a fraction of the rubber coagulates and it is known that this fraction after milling and drying gives a yellow rubber having a higher acetone-extract, a higher protein content and generally, after vulcanization, a higher elasticity-modulus in comparison with the rubber obtained by a full coagulation with sufficient acid of the same original latex. At the other hand, the part of the latex which remained fluid after the formation of the precoagulum will give a rubber of white colour. All this is generally applied in the manufacture of sole-crepe. It is also known that latex, after removal of yellow precoagulum is especially suitable in the production of concentrated preserved latex by creaming or centrifuging methods.

The object of the present invention is to produce a method of fractionating latex by which both the yellow and the white fraction are obtained in a stable, fluid state.

According to the invention the pH of the fresh natural latex which is on the acid side, is raised so that the liquid becomes alkaline, and then a small amount of a salt solution with a mono-valent cation is added. The latex may be diluted, if desired. Then the latex mixture is allowed to stand for a short period, whereafter a separation into two main fractions becomes evident: a greyish-yellow fraction forming the underlayer, and a white fraction forming the upperlayer. Usually at least one thin layer, often bright yellow or orange, but sometimes green-yellow or bright green coloured, will become evident between the two main fractions. On standing the intersurfaces will move downwards.

The various layers are then separated in a known manner.

From the foregoing it is evident that the three following factors must be considered: pH of the latex, concentration of the mono-valent cation in the latex and the dilution of the latex.

A. pH OF THE LATEX.

The optimal pH generally will be about 7.5, but in order to secure the best results with a latex of unknown properties one has to try out the range of 6.8 up to 8.5.

B. CONCENTRATION OF THE SALT.

A concentration of the electrolyte of 0.1 grammol equivalents per litre will generally be in the neighbourhood of the optimum, but variations between 0.03 and 0.3 grammol equivalents per litre have been necessary. At a much higher concentration the fractionation becomes reversed. That is to say, the grey-yellow layer is formed on the top, the white fraction being under. In this instance the intersurface will move upwards with the time. Usually, when the yellow fraction is in contact with the air, a violet discoloration of the surface layer will manifest itself. Although the electrolyte concentration at which this reversed separation occurs, varies greatly with the various latices, the concentration range for every single latex is very small. Since, moreover, at these high concentrations the stability of the latex becomes effected, it is preferably to effect the fractionation in the low concentration range.

In between the first described separa-

tion and those of the reversed types there is a wide range of electrolyte concentration giving no separation at all.

The salts with the various monovalent kations are all workable; salts of bi- or polyvalent cations do not effect a fractionation in liquid layers.

C. Dilution of the Latex.

Dilution of the latex is not a necessary condition but often is convenient in order to ascertain separation in a short time.

In the event that dilution of the field latex is desirable it is preferred to make the latex slightly alkaline before the water is added, or to dilute with slightly alkaline water, since dilution with ordinary water—even distilled water—will often effect a microflocculation of the latex which will hamper the fractionation. It is possible of course, working with latex of known properties, to dilute with water containing the required amounts of ammonia, or alkali, and electrolyte.

Generally, at a pH of about 7.5 and with latex diluted with half of its volume of water, upon addition of NaCl, so that the salt concentration in the mixture is about 0.1 grammol per litre, the separation into two layers becomes evident after only 15—30 minutes and the interface will come to rest after 1—1½ hour. It is possible to accelerate the separation by centrifuging.

The optimal conditions of the fractionation are dependant on the nature of the latex; the age and the strain of the trees, the tapping method, the climate, the weather and even the type of soil may be of influence. These factors will determine the amount of the fractions obtainable under optimal conditions. The amount of the grey-yellow fraction generally is 10—20% of the total volume, but figures of 50% were observed in exceptional cases.

When the pH of the fractions is not raised after the separation, the yellow fraction will coagulate after some hours; the white fraction however is remarkably stable and may be kept for some days before coagulation sets in. During this period a decomposition of the non-rubber constituents takes place in consequence thereof, and so the white fraction may be easily manufactured into purified rubber having a low water absorption.

In the event it is desired to maintain the yellow fraction in liquid state for further use it is advisable to raise the pH directly after separation from the white fraction. The new process enables for the first time the manufacture of rubber articles from the yellow rubber fraction by latex processes, e.g., dipping because up to now that fraction was only known as precoagulum whereas in the method according to the invention it is available in the form of a yellow latex.

The white fraction may be used in the manufacture of sole crepe. As in the case of the white fraction obtained by removal of yellow precoagulum the white fraction obtained by the new process is especially adapted to creaming and centrifuging processes such in consequence of its low viscosity. Morover it has now been observed that it has a better temperature stability than the original latex and consequently it may be applied with advantage in all processes in which latex has to be heated.

Dated this 24th day of April, 1946.
POLLAK, MERCER & TENCH,
Chartered Patent Agents,
134, Cheapside, London, E.C.2,
Agents for the Applicant.

COMPLETE SPECIFICATION

Improvement in the Treatment of Natural Rubber Latices

I, Jan Willem Van Dalfsen, of 10, Hertog, Govertkade, Delft, Holland, a Dutch subject, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The invention concerns an improvement in the treatment of natural rubber latices and may be applied in the manufacture of preserved latex, or of concentrated latex or of dry rubber with special properties, such as a low water absorption, a high plasticity and a white colour.

It is known that upon addition to fresh natural latex of only small amounts of acid, e.g., 1/10 to 1/3 of the quantity of acid that would be required for total coagulation, or of a salt of a polyvalent cation, e.g., $CaCl_2$, a so called precoagulation follows. Only a fraction of the rubber coagulates and it is known that this fraction after milling and drying gives a yellow rubber having a higher acetone-extract (being in general resins), a higher protein content and generally, after vulcanization, a higher elasticity-modulus in comparison with the rubber obtained by a full coagulation with sufficient acid of the same original latex. On the other hand, the part of the latex which remained liquid after the forma-

tion and removing of the precoagulum will give a rubber of white colour..

It is also possible to obtain the yellow precoagulum by stirring diluted latex with a pH value of 6—8 during about 24 hours.

All this is generally applied in the manufacture of sole-crepe. It it also known that latex, after removal of yellow precoagulum is especially suitable in the production of concentrated preserved latex by creaming or centrifuging methods.

In all these cases the yellow fraction is obtained in a solid form.

The object of the present invention is a method of fractionating latex by which not only the white fraction but also the yellow fraction is obtained in a stable, fluid, state.

According to the invention the pH of the natural field rubber latex which is on the acid side (low pH), is raised, e.g., by adding ammonia so that the liquid becomes alkaline (pH 6.0—8.5) and then a small amount of a solution of a salt with a mono-valent cation is added. The latex may be diluted, if desired. Then the latex mixture is allowed to stand for a short period, whereafter a separation into two main fractions becomes evident; a greyish-yellow fraction forming the underlayer, and a white fraction forming the upperlayer. Usually at least one thin layer, often bright yellow or orange, but sometimes green-yellow or bright green coloured, will become evident between the two main fractions. The separation begins after about 15 minutes and is completed in about an hour. During that time the intersurfaces move downwards. The various layers are then separated in a known manner, e.g., decantation.

I am aware of the fact that the Specification No. 413,185 mentions a process of concentrating fresh latex by creaming, characterised in that in addition to the usual creaming agent a small quantity of water-soluble soap up to 0.5% is added to a latex. The present invention however does not make use of creaming agents.

Moreover I am aware of the fact that Patent Specification No. 586,830 discloses a process for the preparation of latex having a low protein-content, which comprises diluting the latex, adding to the diluted latex a capillary-active substance which is more strongly adsorbed by the rubber particles than the proteins, allowing the mixture to stand for some hours, and then separating the latex from the serum by creaming or centrifuging. The salts with monovalent cation, used according to the invention, are not capillary active.

From the foregoing it is evident that the three following factors must be considered: pH of the latex, concentration of the mono-valent cation in the latex and the dilution of the latex.

A. pH of the Latex.

The optimum pH generally will be about 7.5, but in order to secure the best results with a latex of unknown properties, it is necessary to try out the range of 6.0 up to 8.5.

B. Concentration of the Salt.

A concentration of electrolyte of 0.1 grammol-equivalents per litre will generally be in the neighbourhood of the optimum, but variations between 0.02 and 0.4 grammol equivalents per litre or even a little higher, dependent upon the type and age of the latex, have been necessary. At a much higher concentration, e.g., in the neighbourhood of 1.0 grammol per litre the fractionation becomes reversed. That is to say, the grey-yellow layer is formed on the top, the white fraction being under. In this instance the intersurface will move upwards with time. Usually when the yellow fraction is in contact with the air, a violet discoloration of the surface layer will manifest itself. Although the electrolyte concentration at which this reversed separation occurs, varies greatly with the various latices, the concentration range for every single latex is very small. Since, moreover, at these high concentrations the stability of the latex becomes ~~effected~~ affected, it is preferably to effect the fractionation in the low concentration range.

In between the first described separation and those of the reversed types there is a wide range of electrolyte concentration giving no separation at all.

The salts with the various mono-valent cations are all workable.

C. Dilution of the Latex.

Dilution of the latex is not a necessary condition but is often convenient, e.g., to a dry rubber content of 15—30%, in order to obtain separation in a short time.

In the event that dilution of the field latex is desirable it is preferred to make the latex slightly alkaline before the water is added, or to dilute with slightly alkaline water, since dilution with ordinary water—even distilled water—will often effect a microflocculation of the latex which will hamper the fractionation. It is possible of course, working with latex of known properties, to dilute with water containing the required amounts of ammonia, of alkali, and electrolyte.

Generally, at a pH of about 7.5 and with latex ~~of known properties, to dilute~~ diluted with half of its

volume of water, upon addition of NaCl, so that the salt concentration in the mixture is about 0.1 grammol per litre, the separation into two layers becomes evident after only 15—30 minutes and the intersurface will come to rest after 1—1½ hours. It is possible to accelerate the separation by centrifuging.

The optimum conditions of the fractionation are dependent on the nature of the latex; the age and the strain of the trees, the tapping method, the climate, the weather and even the type of soil may be of influence. These factors will determine the amount of the fractions obtainable under optimum conditions. The amount of the grey-yellow fraction generally is 10—20% of the total volume, but figures of 50% have been observed in exceptional cases.

When the pH of the fractions is not raised after the separation, the yellow fraction will coagulate after some hours; the white fraction, however, is remarkably stable and may be kept for some days before coagulation sets in. During this period a decomposition of the non-rubber constituents takes place, in consequence whereof the white fraction may be easily manufactured into purified rubber having a low water absorption.

In the event it is desired to maintain the yellow fraction in the liquid state for further use it is advisable to raise the pH directly after separation from the white fraction.

The new process enables for the first time the manufacture of rubber articles from the yellow rubber fraction by latex processes, e.g., dipping. Up to now that fraction was only known as a solid precoagulum whereas in the method according to the invention it is available in the form of a yellow latex.

The white fraction has a low water absorption. It may be used in the manufacture of sole-crepe. As in the case of the white fraction obtained by removal of yellow precoagulum the white fraction obtained by the new process is especially adapted for creaming and centrifuging processes in consequence of its low viscosity. Morover it has now been observed that it has a better temperature stability than the original latex and consequently it may be applied with advantage in all processes in which latex has to be heated.

EXAMPLE.

A small quantity ammonia was added to fresh field latex insufficient to raise the pH value to 7.5, after which the latex was diluted with half its volume of water; the pH of the diluted latex was then brought to a value of 7.5, by further addition of ammonia. After that 2 parts by volume of a 25% solution of common salt were added per 100 parts of volume of the mixture. About 15 minutes after this addition the separation became apparent, whereby the volume of the underlayer comprised 22% of the total volume; after 1 hour a sharp separation had taken place and a yellow fraction with a volume of 12% of the total volume was present. The upperlayer was subsequently siphoned off, and the underlayer was drawn off.

Having now particularly described and ascertained the nature of my said invention and in what manner the same is to be performed, I declare that what I claim is:—

1. A process for the separation of natural field rubber latex into fractions, whereby an alkaline substance such as ammonia is added to bring the pH value to between 6.0 and 8.5, characterised in that after the addition of the ammonia, a solution of a salt with a mono-valent cation is added and the mixture is allowed to stand till a separation into at least two layers becomes evident, namely a so-called "yellow" fraction and a so called "white" fraction whereafter the layers are separated in a known manner, e.g., by decantation.

2. A process as per claim 1, characterized in that the salt solution with the amount of mono-valent cation added is such that the concentration is 0.02 to 0.4 grammol per litre of mixture.

3. A process as per claim 1, thereby characterized, that the salt solution with the amount of mono-valent cation added is such that the concentration is about 1.0 grammol per litre of mixture.

4. A process as claimed in any of the preceding claims, in which the latex is diluted.

5. The latex fraction known as the "yellow" fraction when obtained in the fluid state by the process claimed in any of the preceding claims.

6. The latex fraction known as the "white" fraction when obtained in the fluid state by the process claimed in any of the preceding claims.

7. The "yellow" fraction as claimed in claim 5 when stabilized by raising its pH value after separation.

Dated the 17th day of February, 1947.

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volume of water, upon addition of NaCl, so that the salt concentration in the mixture is about 0.1 grammol per litre, the separation into two layers becomes evident after only 15—30 minutes and the intersurface will come to rest after 1—1½ hours. It is possible to accelerate the separation by centrifuging.

The optimum conditions of the fractionation are dependent on the nature of the latex; the age and the strain of the trees, the tapping method, the climate, the weather and even the type of soil may be of influence. These factors will determine the amount of the fractions obtainable under optimum conditions. The amount of the grey-yellow fraction generally is 10—20% of the total volume, but figures of 50% have been observed in exceptional cases.

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In the event it is desired to maintain the yellow fraction in the liquid state for further use it is advisable to raise the pH directly after separation from the white fraction.

The new process enables for the first time the manufacture of rubber articles from the yellow rubber fraction by latex processes, e.g., dipping. Up to now that fraction was only known as a solid precoagulum whereas in the method according to the invention it is available in the form of a yellow latex.

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1. A process for the separation of natural field rubber latex into fractions, whereby an alkaline substance such as ammonia is added to bring the pH value to between 6.0 and 8.5, characterised in that after the addition of the ammonia, a solution of a salt with a mono-valent cation is added and the mixture is allowed to stand till a separation into at least two layers becomes evident, namely a so called "yellow" fraction and a so called "white" fraction whereafter the layers are separated in a known manner, e.g., by decantation.

2. A process as per claim 1, characterized in that the salt solution with the amount of mono-valent cation added is such that the concentration is 0.02 to 0.4 grammol per litre of mixture.

3. A process as per claim 1, thereby characterized, that the salt solution with the amount of mono-valent cation added is such that the concentration is about 1.0 grammol per litre of mixture.

4. A process as claimed in any of the preceding claims, in which the latex is diluted.

5. The latex fraction known as the "yellow" fraction when obtained in the fluid state by the process claimed in any of the preceding claims.

6. The latex fraction known as the "white" fraction when obtained in the fluid state by the process claimed in any of the preceding claims.

7. The "yellow" fraction as claimed in claim 5 when stabilized by raising its pH value after separation.

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