

PATENT SPECIFICATION

751,491

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COMPLETE SPECIFICATION.

Improvements in or relating to the Concentration of Aqueous Dispersions of Rubber.

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:—

10 This invention relates to a process for the concentration of aqueous dispersions of natural rubber.

15 It has already been proposed in British Patent Specification No. 478,406 to concentrate such dispersions by partly freezing the same and thereafter mechanically separating the liquid phase from the solid phase, or by completely freezing the said dispersions and subsequently allowing them partly to thaw before separating the phases.

20 The disadvantages of the aforescribed method is that the rubber is recovered in the form of a number of fractions of varying rubber contents of which only the most concentrated is of sufficiently high rubber content to be of any commercial value, whilst the more dilute or intermediate fractions have to be re-frozen and re-fractionated in order to render them useful, thereby necessitating a number of relatively expensive freezing or other operations.

25 The present invention aims at separating partially frozen aqueous dispersions of natural rubber in one operation, into only two fractions, one of which contains substantially all the rubber of the original dispersion in a very concentrated form, and the other a solid ice fraction containing substantially no rubber.

40 The present invention is based on the discovery that the concentration of rubber in

the liquid phase is determined by the temperature at which the rubber dispersion is partially frozen, and that if said dispersion be maintained substantially at that temperature whilst the separation of phases takes place, only two fractions are obtained, one of which is of high rubber content whilst the other consists of an ice fraction of very low rubber content.

Based on the foregoing discovery, the present invention broadly comprises subjecting a natural rubber dispersion to partial freezing at a temperature lying between -5 and -20° C. and whilst said rubber dispersion is partially frozen separating the frozen dispersion into the two aforescribed phases at substantially the same temperature as that at which freezing has initially taken place.

The separation can be effected for example by pressing, filtration or centrifuging, all carried out in suitably refrigerated apparatus.

In general, the amount of ice which separates from an aqueous dispersion of natural rubber increases progressively as the temperature is reduced. At first pure ice separates, but on further cooling, the residual ice crystals contain certain substances derived from the serum of the said dispersion. Finally, the dispersion may become unstable due to the precipitation and dehydration of an undue proportion of such substances in the serum as act as protective colloids.

When effecting the separation of the rubber dispersion by centrifuging, the rubber dispersion may first be subjected to partial freezing at a temperature of between -5 and -20° C. and the partially frozen dispersion thereupon introduced into a centrifuge refrigerated to a temperature of be-

tween -5 and -20° C. wherein the separation of the phases is effected.

Alternatively, pre-cooled rubber dispersion still in liquid form may be introduced at such a rate into a rotating centrifuge maintained at between -5 and -20° C., that the entering dispersion is substantially instantaneously partially frozen and thereupon separated into its component phases by centrifugal force.

An important advantage of the process of the present invention is that the stabiliser, such as ammonia, which may be added to the original aqueous dispersion of rubber passes mainly into the rubber-rich fraction, and that there are no intermediate fractions containing stabiliser from which it is difficult to recover the said stabiliser.

The hereindescribed process enables rubber concentrates of between 55 and 75% total solids content to be obtained.

The invention will be illustrated by the following comparative Examples viz.:

EXAMPLE I

Natural rubber latex preserved with ammonia was treated as in the process described in Specification 478,406 by freezing at a temperature of between -2° C. and -5° C. whilst stirring. The liquid fraction was then drawn off by suction, and the solid residue allowed to thaw partly before again drawing off the liquid fraction, so that three fractions were obtained. Of these fractions the most concentrated fraction was subjected to a similar process by freezing again and removing liquid fractions by suction. The products consisted of five fractions of the following rubber contents:

- Fraction 1—61.6%,
- Fraction 2—49.3%,
- Fraction 3—30.3%,
- Fraction 4—7.1%,
- Fraction 5—2.2%.

Only the first fraction, which contained 56.5% of the original total solids weight, had a sufficient rubber content for commercial use, while the remainder had to be reprocessed.

EXAMPLE II

Rubber latex preserved with ammonia was partially frozen at a temperature between -10° C. and -12° C. whilst stirring. The partially frozen latex was placed in a basket centrifuge refrigerated to a temperature of between -10° C. and -12° C. and separated by centrifugal action in one operation into concentrated latex of 70% total solids content and an ice fraction of 10% total latex solids content, 80% of the rubber in the original latex being recovered in the former fraction.

EXAMPLE III

13.4 kgs. of ammonia-preserved latex having a total solids content of 33% was partially frozen in a rotary ice-cream freezer to a temperature of -15° C. and whilst substantially at that temperature was centrifuged in a basket centrifuge refrigerated to substantially -15° C.

A concentrated latex of 65.0% total solids content was obtained, together with an ice fraction containing only 2.8% of the original total solids content in the latex.

EXAMPLE IV

652 grams of an ammonia-preserved rubber latex having a total solids content of 36% was partially frozen at a temperature of -19° C. in a rotary ice-cream freezer. The partially frozen latex was thereupon centrifuged in a basket centrifuge chilled to a temperature of -20° C. and was separated in one operation into a concentrated latex of a total solids content of 77.3% and an ice fraction.

EXAMPLE V

Ammonia preserved field latex having a total solids content of 33.2% was partially frozen in a refrigerator to a temperature of -12° C. and the partially frozen mass was placed in a plunger press chilled to a temperature of -12° C. and provided with a wire mesh filter of 120 mesh/inch gauge at one end of the cylinder.

Upon applying pressure to the plunger, a concentrated latex was forced out of the cylinder through the wire mesh filter, the total solids content of the concentrate being 71.3%.

EXAMPLE VI

Ammoniated field latex having a total solids content of 35.0% was partially frozen to a temperature of -12° C. and placed in a Buchner filter funnel chilled to a temperature of -12° C. and provided with cotton duck cloth filter. Upon applying vacuum to the Buchner flask, a concentrate of a total solids content of 60.2% was obtained.

What we claim is:—

1. A process of concentrating an aqueous dispersion of natural rubber, which comprises subjecting said dispersion to freezing at a temperature lying between -5 and -20° C. and, whilst said rubber dispersion is partially frozen, separating the partially frozen dispersion at substantially the same temperature as that at which the freezing was carried out, into a liquid fraction containing substantially all the rubber of the original dispersion in a highly concentrated form and a solid ice fraction containing substantially no rubber.

2. Process as claimed in Claim 1, in which the partially frozen dispersion is

tween -5 and -20° C. wherein the separation of the phases is effected.

Alternatively, pre-cooled rubber dispersion still in liquid form may be introduced at such a rate into a rotating centrifuge maintained at between -5 and -20° C., that the entering dispersion is substantially instantaneously partially frozen and thereupon separated into its component phases by centrifugal force.

An important advantage of the process of the present invention is that the stabiliser, such as ammonia, which may be added to the original aqueous dispersion of rubber passes mainly into the rubber-rich fraction, and that there are no intermediate fractions containing stabiliser from which it is difficult to recover the said stabiliser.

The hereindescribed process enables rubber concentrates of between 55 and 75% total solids content to be obtained.

The invention will be illustrated by the following comparative Examples viz.:

EXAMPLE I

Natural rubber latex preserved with ammonia was treated as in the process described in Specification 478,406 by freezing at a temperature of between -2° C. and -5° C. whilst stirring. The liquid fraction was then drawn off by suction, and the solid residue allowed to thaw partly before again drawing off the liquid fraction, so that three fractions were obtained. Of these fractions the most concentrated fraction was subjected to a similar process by freezing again and removing liquid fractions by suction. The products consisted of five fractions of the following rubber contents:

Fraction 1—61.6%,
 Fraction 2—49.3%,
 Fraction 3—30.3%,
 Fraction 4—7.1%,
 Fraction 5—2.2%.

Only the first fraction, which contained 56.5% of the original total solids weight, had a sufficient rubber content for commercial use, while the remainder had to be re-processed.

EXAMPLE II

Rubber latex preserved with ammonia was partially frozen at a temperature between -10° C. and -12° C. whilst stirring. The partially frozen latex was placed in a basket centrifuge refrigerated to a temperature of between -10° C. and -12° C. and separated by centrifugal action in one operation into concentrated latex of 70% total solids content and an ice fraction of 10% total latex solids content, 80% of the rubber in the original latex being recovered in the former fraction.

EXAMPLE III

13.4 kgs. of ammonia-preserved latex having a total solids content of 33% was partially frozen in a rotary ice-cream freezer to a temperature of -15° C. and whilst substantially at that temperature was centrifuged in a basket centrifuge refrigerated to substantially -15° C.

A concentrated latex of 65.0% total solids content was obtained, together with an ice fraction containing only 2.8% of the original total solids content in the latex.

EXAMPLE IV

652 grams of an ammonia-preserved rubber latex having a total solids content of 36% was partially frozen at a temperature of -19° C. in a rotary ice-cream freezer. The partially frozen latex was thereupon centrifuged in a basket centrifuge chilled to a temperature of -20° C. and was separated in one operation into a concentrated latex of a total solids content of 77.3% and an ice fraction.

EXAMPLE V

Ammonia preserved field latex having a total solids content of 33.2% was partially frozen in a refrigerator to a temperature of -12° C. and the partially frozen mass was placed in a plunger press chilled to a temperature of -12° C. and provided with a wire mesh filter of 120 mesh/inch gauge at one end of the cylinder.

Upon applying pressure to the plunger, a concentrated latex was forced out of the cylinder through the wire mesh filter, the total solids content of the concentrate being 71.3%.

EXAMPLE VI

Ammoniated field latex having a total solids content of 35.0% was partially frozen to a temperature of -12° C. and placed in a Buchner filter funnel chilled to a temperature of -12° C. and provided with cotton duck cloth filter. Upon applying vacuum to the Buchner flask, a concentrate of a total solids content of 60.2% was obtained.

What we claim is:—

1. A process of concentrating an aqueous dispersion of natural rubber, which comprises subjecting said dispersion to freezing at a temperature lying between -5 and -20° C. and, whilst said rubber dispersion is partially frozen, separating the partially frozen dispersion at substantially the same temperature as that at which the freezing was carried out, into a liquid fraction containing substantially all the rubber of the original dispersion in a highly concentrated form and a solid ice fraction containing substantially no rubber.

2. Process as claimed in Claim 1, in which the partially frozen dispersion is



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separated by pressing, filtration or centrifuging, carried out under refrigerating conditions.

3. Process as claimed in Claim 1 or 2, in which a pre-cooled aqueous dispersion in liquid form, of natural rubber, is introduced at such a rate into a rotating centrifuge maintained at a temperature between -5 and -20°C ., that the entering dispersion is substantially instantaneously partially frozen whereupon said partially frozen dispersion is subjected to centrifugal separation.

4. Process of concentrating aqueous dispersions of natural rubber, substantially as described with reference to the foregoing Examples. 15

5. Rubber concentrates whenever produced by the process claimed in any of the preceding claims. 20

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PROVISIONAL SPECIFICATION.

Improvements in or relating to the Concentration of Aqueous Dispersions of Rubber.

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 for the concentration of aqueous dispersions of rubber and rubber-like substances such as guttapercha, balata, jelutong and synthetic rubbers.

It has already been proposed in British Patent Specification No. 478,406 to concentrate such dispersions by partly freezing the same and thereafter mechanically separating the liquid phase from the solid phase, or by completely freezing the said dispersions and subsequently allowing them partly to thaw before separating the phases.

The disadvantage of the foregoing method is that the rubber is recovered in the form of a number of fractions of varying rubber contents of which only the most concentrated is of sufficiently high rubber content to be of any commercial value, whilst the more dilute or intermediate fractions have to be re-frozen and re-fractionated in order to render them useful, thereby necessitating a number of operations.

The present invention aims at separating aqueous dispersions of rubber in one operation, into only two fractions, one of which contains substantially all the rubber of the original dispersion in a very concentrated form, and the other a solid ice fraction containing substantially no rubber.

The present invention is based on the discovery that the concentration of rubber in the liquid phase is determined by the temperature at which the rubber dispersion is frozen, and that if said dispersions be maintained substantially at that temperature whilst the separation of phases takes place, only two fractions are obtained, one of

which is of high rubber content whilst the other consists of an ice fraction of very low rubber content. 65

Based on the foregoing discovery, the present invention broadly comprises subjecting a rubber dispersion to freezing at a temperature lying between -5 and -20°C . and subsequently or simultaneously with said freezing separating the frozen dispersion into the two aforescribed phases at the same temperature as or at a lower temperature than that at which freezing has initially taken place. 70

The separation can be effected for example by pressing, filtration or centrifuging, all carried out in suitably refrigerated apparatus. 75

In general, the amount of ice which separates from an aqueous dispersion of rubber and the like increases progressively as the temperature is reduced. At first pure ice separates, but on further cooling, the residual ice crystals contain certain substances derived from the serum of the said dispersion. Finally, the dispersion may become unstable due to the precipitation and dehydration of an undue proportion of such substances in the serum as act as protective colloids. 80

When effecting the separation of the rubber dispersion by centrifuging, the rubber dispersion may first be subjected to freezing at a temperature of between -5 and -20°C . and the frozen dispersion thereupon introduced into a centrifuge refrigerated to a temperature of between -5 and -20°C . wherein the separation of the phases is effected. 85

Alternatively, pre-cooled rubber dispersion still in liquid form may be introduced into a rotating centrifuge maintained at an extremely low temperature, such that the entering dispersion is substantially instantaneously frozen and thereupon separated 90

into its component phases by centrifugal force.

5 An important advantage of the process of the present invention is that the stabiliser, such as ammonia, which may be added to the original aqueous dispersion of rubber and the like passes mainly into the rubber-rich fraction, and that there are no fractions of intermediate stabiliser content from which it is difficult to recover the said stabiliser.

10 The hereindescribed process enables rubber concentrates of between 55 and 57% total solids content to be obtained.

15 The invention will be illustrated by the following comparative Examples viz.:

EXAMPLE I

20 Natural rubber latex preserved with ammonia was treated by the process described in Specification No. 478,406 by freezing at a temperature of between -2°C . and -5°C . whilst stirring. The liquid fraction was then drawn off by suction, and the solid residue allowed to thaw partly before again drawing off the liquid fraction, so that three fractions were obtained. Of these fractions the most concentrated fraction was subjected to a similar process by freezing again and removing liquid fractions by suction. The products consisted of five fractions of the following rubber contents:

Fraction 1—61.6%.
Fraction 2—49.3%.
Fraction 3—30.3%.
Fraction 4— 7.1%.
Fraction 5— 2.2%.

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Only the first fraction, which contained 56.5% of the original total solids weight, had a sufficient rubber content for commercial use, whilst the remainder had to be re-processed.

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EXAMPLE II

Rubber latex preserved with ammonia was frozen in accordance with the present invention at a temperature between -10°C . and -12°C . whilst stirring. The frozen latex was placed in a basket centrifuge refrigerated to a temperature of between -10°C . and -12°C . and separated by centrifugal action in one operation into concentrated latex of 70% total solids content and an ice fraction of 10% total solids content, 80% of the rubber in the original latex being recovered in the former fraction.

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