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COMPLETE SPECIFICATION

Improvements in or relating to the Concentration and Purification of Aqueous Colloidal Dispersions

We, DUNLOP RUBBER COMPANY LIMITED, a British Company, of 1, Albany Street, London, N.W.1, and FREDERICK JAMES PATON, a British Subject, of Segil Estate, Tangkak, Johore, 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:—

This invention relates to a process and apparatus for concurrently concentrating and purifying aqueous colloidal dispersions.

It is known to concentrate aqueous colloidal dispersions by subjecting them to an electric potential difference while located between two or more vertical semi-permeable membranes, whereby the colloid particles migrate towards one of the electrodes used to set up the potential difference and form a colloid-rich region in the dispersion adjacent each membrane. As a result of the density difference thus produced between the parts of the dispersion adjacent to the membranes and the remainder of the dispersion, the colloid-rich portion moves vertically either upwards or downwards according to whether the specific gravity of the colloid particles is less or greater than that of the aqueous medium. Separation thus results into a colloid-rich layer and a colloid-impoverished layer. The electric potential gradient between the electrodes is usually of the order of 1—2 volts per cm. Although the rate of migration of colloid to the diaphragm would be increased by increasing the voltage gradient, the increased development of heat which would result in the dispersion, due to the increased current passed through it, has made it impracticable to accelerate the concentration process by so increasing the electric potential gradient. In Specification No. 505,753 there is described and claimed such a process which is made continuous by continuously supplying colloidal dispersion to the space or spaces between the membranes and continuously removing separately the colloid-rich layer and the colloid-impoverished layer.

The electrodes employed to set up the potential difference are normally in separate compartments which communicate with the dispersion through semi-permeable membranes, these compartments being filled with water. If the aqueous phase of the colloid dispersion also contains dissolved material some dialysis occurs in the initial stages of the process, i.e. dissolved material passes into the electrode compartments, but equilibrium conditions are soon reached and thereafter diminution in the content of dissolved material in the dispersion does not occur. The nature and concentration of the dissolved material in the aqueous medium are thus substantially those of the untreated dispersion.

The above described method of concentrating colloidal dispersions, generally referred to as electrodecantation, has been particularly useful for concentrating rubber latex, which contains dissolved material in the serum thereof. During the electrodecantation the latex separates into a rubber-rich upper layer and a rubber-impoverished lower layer. The latex frequently contains ammonia as a preservative and the electrode compartments are then filled with aqueous ammonia to prevent the ammonia content of the latex from decreasing.

It is an object of the present invention to provide an electrodecantation process whereby an aqueous colloidal dispersion containing dissolved material can be purified by removal of dissolved material from the aqueous medium and simultaneously concentrated. It is also an object of the invention to provide an

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apparatus for carrying out such a process.

According to the present invention a continuous process for concurrently concentrating and purifying an aqueous colloidal dispersion containing dissolved material comprises continuously supplying the colloidal dispersion to the space or spaces between a plurality of semi-permeable membranes spaced apart, maintaining an electric potential difference across said membranes to effect a separation of the dispersion into a colloid-rich layer and a colloid-impoverished layer, separately removing said layers from the space or spaces between said membranes in a continuous manner, and continuously removing dissolved substances from the aqueous dispersion between the membranes by dialysis.

According to a further feature of the invention, an apparatus for carrying out the above invention comprises a cell divided into an odd number of separate compartments by means of upright semi-permeable membranes, electrodes in the end compartments of the cell, means for feeding water through the electrode compartments and through alternate intermediate compartments, means for delivering colloidal dispersion to the remaining compartments and means for separately removing from each of said remaining compartments a colloid-rich layer and a colloid-impoverished layer.

By means of this invention the colloidal dispersion is both concentrated and purified by removal of dissolved impurities. A higher potential gradient across the electrodes can also be maintained without causing the temperature of the latex to rise unduly because the supply of water to effect dialysis also serves to cool the colloidal dispersion, and thus electrodecantation can be carried out more speedily.

The invention may be applied to the electrodecantation of the rubber latex and of other colloidal dispersions containing substances dissolved in the aqueous phase; these may be artificial dispersions of rubber, aqueous dispersions of synthetic rubber or synthetic resins, and aqueous dispersions of other colloids, e.g., casein or colloidal dispersions of dyestuffs.

Thus rubber latex can be concentrated and purified according to the invention in apparatus of known type comprising a cell having a latex compartment, in which are semi-permeable membranes spaced apart, and electrode compartments at each end of the cell each separated from the latex compartment by a semi-permeable membrane. The latex is concentrated in the latex compartment in the manner employed hitherto, and the electrolyte in each electrode compartment is continuously or intermittently changed during the concentration process with a frequency sufficient to prevent equal concentration of dissolved substances being set up in the liquids on each side of the semi-permeable membranes separating each electrode compartment from the latex compartment. As latex is supplied to the latter concentration and dialysis occur concurrently while latex is passing through the compartment, and a rubber-rich layer is recovered in which the serum is at least partly denuded of the dissolved substances originally present therein.

Preferably the process is carried out in the apparatus referred to above having a plurality of latex-containing compartments, each between two electrolyte compartments. When this apparatus is employed, water or a suitable electrolyte is placed in the electrode compartments and in alternate intermediate compartments and the latex to be purified is continuously supplied to each of the remaining compartments, the latex in any one compartment thus being separated on each side from electrolyte by a semi-permeable membrane. When a suitable electric potential difference is maintained between the electrode compartments dialysis occurs through the semi-permeable membranes, and the water or electrolyte in the compartments on both sides of those containing the colloidal dispersion is continuously or intermittently replaced so that conditions permitting the dialysis to occur are maintained throughout the process.

In either of the above forms of the invention the electrolyte fed to the electrode compartments may be water free from any dissolved substance. On the other hand, if it is desired to prevent any particular dissolved substance being removed from the dispersion the electrolyte fed into the compartments should be water containing such substance in a concentration equal to that in which it is present in the dispersion. Thus in treating an ammonia-containing latex, removal of ammonia from the latex may be prevented by employing as electrolyte water containing ammonia in the concentration in which it is present in the latex.

An apparatus according to the invention for concentrating natural rubber latex will now be described with reference to the accompanying drawings in which:—

Figure 1 is a sectional elevation of the apparatus,

Figure 2 is a plan of the apparatus.

The apparatus is built up of two elec-

trode boxes 11 and a large number of rectangular ebonite frames 1 alternating with rectangular frames 2, and between adjacent frames are sheets of regenerated cellulose 10. Soft rubber sealing rings may also be disposed between successive frames to render the unit water-tight. The electrode boxes and frames have around their outer peripheries a number of projecting perforated lugs 3. When the apparatus is assembled the perforations of the lugs are in register and form a passage through which rods 4 can be passed to clamp the frames together by nuts screwed thereon. The frames are disposed with their short sides vertical forming the side walls of the apparatus, and the long sides form respectively the top and bottom of the apparatus, and will be referred to as the top and bottom of the frames respectively. The frames 1 together with the regenerated cellulose sheets on either side define the water compartments of the apparatus, and frames 2 and the adjacent regenerated cellulose sheets define the latex compartments of the apparatus. Each frame 1 is provided in the middle of the top and bottom with holes 5, 6 respectively forming channels for water, and each frame 2 has in the top and bottom adjacent the centres thereof holes 7, 8 for the passage of concentrated latex and of serum respectively as will be described subsequently. The short sides of the frames 2 have also similar passages 9 by which latex to be concentrated can be delivered into the latex compartments as will be described subsequently. The apparatus also comprises a header 12 by means of which water can be delivered through pipes 13 and passages 5 to the compartments defined by frames 1, and a header 14 and pipes 15 by which water can be withdrawn from the compartments through passages 6. Two headers 16 are provided, one on each side of the apparatus, each having a plurality of pipes 17 by which latex to be concentrated can be delivered to the latex compartments through passages 9, header 18 and pipes 19 by which concentrated latex can be withdrawn from passages 7, and header 20 and pipes 21 by which serum can be withdrawn from passages 8. No regenerated cellulose sheet is provided between the end water compartment and the adjacent electrode box, and in each of the latter is a graphite electrode (not shown) having connections (not shown) by which the electrode can be connected to one terminal of a D.C. electric current supply.

When the apparatus is in operation ammonia-preserved latex is supplied to each of the latex compartments of the apparatus through the headers 16 and associated pipes 17 and passages 9, and water containing dissolved ammonia at the same concentration as in the latex is supplied through the header 12 and the pipes 13 to each of the water compartments of the apparatus. Water from the end pipes 13 also fills each electrode box 11. A suitable electric potential is then applied across the apparatus by connecting the electrodes to the source of direct current, and the supplies of ammonia-preserved latex and of ammonia-containing water respectively are adjusted so that when the apparatus is in steady operation a rubber-enriched latex having a diminished content of dissolved solids is withdrawn through the header 18 and serum is withdrawn through the header 20. Water containing the dissolved solids removed from the latex is withdrawn through the water outlet header 14, being replaced continuously by fresh ammonia-containing water introduced through the water inlet header 12. Because of the ammonia content of the water supplied, removal of ammonia from the latex by dialysis, with consequent loss of stability of the latex, is prevented.

The apparatus may be used for concentrating and purifying latex of any concentration up to concentrations of the order of 50% dry rubber content, and may be operated so as to produce a rubber-enriched latex containing as much as 70% dry rubber content. For example latex supplied to the apparatus may contain approximately 30% dry rubber content and a rubber-enriched latex with a diminished dissolved solids content and a dry rubber content of about 60% obtained from it. The potential difference applied across the electrodes should be such that the potential gradient across the apparatus is of the order of 2—4 volts per c.m.

Because a greater potential gradient can be employed than has been feasible hitherto, because of the cooling effect of the water circulating through the water compartments, electrodecantation proceeds more rapidly than in prior processes, and from latex of 30% dry rubber content a rubber-enriched latex containing about 60% dry rubber content can be produced at a rate of the order of 1.2 to 2.5 gallons per hour per square metre of membrane.

What we claim is:

1. A continuous process for concurrently concentrating and purifying an aqueous colloidal dispersion containing dissolved material which comprises continuously supplying the colloidal disper-

trode boxes 11 and a large number of rectangular ebonite frames 1 alternating with rectangular frames 2, and between adjacent frames are sheets of regenerated cellulose 10. Soft rubber sealing rings may also be disposed between successive frames to render the unit water-tight. The electrode boxes and frames have around their outer peripheries a number of projecting perforated lugs 3. When the apparatus is assembled the perforations of the lugs are in register and form a passage through which rods 4 can be passed to clamp the frames together by nuts screwed thereon. The frames are disposed with their short sides vertical forming the side walls of the apparatus, and the long sides form respectively the top and bottom of the apparatus, and will be referred to as the top and bottom of the frames respectively. The frames 1 together with the regenerated cellulose sheets on either side define the water compartments of the apparatus, and frames 2 and the adjacent regenerated cellulose sheets define the latex compartments of the apparatus. Each frame 1 is provided in the middle of the top and bottom with holes 5, 6 respectively forming channels for water, and each frame 2 has in the top and bottom adjacent the centres thereof holes 7, 8 for the passage of concentrated latex and of serum respectively as will be described subsequently. The short sides of the frames 2 have also similar passages 9 by which latex to be concentrated can be delivered into the latex compartments as will be described subsequently. The apparatus also comprises a header 12 by means of which water can be delivered through pipes 13 and passages 5 to the compartments defined by frames 1, and a header 14 and pipes 15 by which water can be withdrawn from the compartments through passages 6. Two headers 16 are provided, one on each side of the apparatus, each having a plurality of pipes 17 by which latex to be concentrated can be delivered to the latex compartments through passages 9, header 18 and pipes 19 by which concentrated latex can be withdrawn from passages 7, and header 20 and pipes 21 by which serum can be withdrawn from passages 8. No regenerated cellulose sheet is provided between the end water compartment and the adjacent electrode box, and in each of the latter is a graphite electrode (not shown) having connections (not shown) by which the electrode can be connected to one terminal of a D.C. electric current supply.

When the apparatus is in operation ammonia-preserved latex is supplied to each of the latex compartments of the apparatus through the headers 16 and associated pipes 17 and passages 9, and water containing dissolved ammonia at the same concentration as in the latex is supplied through the header 12 and the pipes 13 to each of the water compartments of the apparatus. Water from the end pipes 13 also fills each electrode box 11. A suitable electric potential is then applied across the apparatus by connecting the electrodes to the source of direct current, and the supplies of ammonia-preserved latex and of ammonia-containing water respectively are adjusted so that when the apparatus is in steady operation a rubber-enriched latex having a diminished content of dissolved solids is withdrawn through the header 18 and serum is withdrawn through the header 20. Water containing the dissolved solids removed from the latex is withdrawn through the water outlet header 14, being replaced continuously by fresh ammonia-containing water introduced through the water inlet header 12. Because of the ammonia content of the water supplied, removal of ammonia from the latex by dialysis, with consequent loss of stability of the latex, is prevented.

The apparatus may be used for concentrating and purifying latex of any concentration up to concentrations of the order of 50% dry rubber content, and may be operated so as to produce a rubber-enriched latex containing as much as 70% dry rubber content. For example latex supplied to the apparatus may contain approximately 30% dry rubber content and a rubber-enriched latex with a diminished dissolved solids content and a dry rubber content of about 60% obtained from it. The potential difference applied across the electrodes should be such that the potential gradient across the apparatus is of the order of 2—4 volts per c.m.

Because a greater potential gradient can be employed than has been feasible hitherto, because of the cooling effect of the water circulating through the water compartments, electrodecantation proceeds more rapidly than in prior processes, and from latex of 30% dry rubber content a rubber-enriched latex containing about 60% dry rubber content can be produced at a rate of the order of 1.2 to 2.5 gallons per hour per square metre of membrane.

What we claim is:

1. A continuous process for concurrently concentrating and purifying an aqueous colloidal dispersion containing dissolved material which comprises continuously supplying the colloidal disper-

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sion to the space or spaces between a plurality of semi-permeable membranes spaced apart, maintaining an electric potential difference across said membranes to effect a separation of the dispersion into a colloid-rich layer and a colloid-impoverished layer, separately removing said layers from the space or spaces between said membranes in a continuous manner, and continuously removing dissolved substances from the aqueous colloidal dispersion between the membranes by dialysis.

2. A process according to claim 1 which comprises feeding water through alternate compartments of a series defined by a plurality of upright spaced semi-permeable membranes, delivering the colloidal dispersion to each of the remaining compartments of the series, and separately removing from each of said remaining compartments a colloid-rich layer and a colloid-impoverished layer.

3. A process according to any of the preceding claims in which the aqueous colloidal dispersion is a natural rubber latex.

4. A process according to claim 1 for concurrently concentrating and purifying an ammonia-preserved natural-rubber latex which comprises circulating aqueous ammonia having the same content as the said latex through alternate compartments of a series defined by a plurality of upright spaced semi-permeable membranes, delivering the ammonia-preserved natural rubber latex to each of the remaining compartments of the series at a level intermediate the top and the bottom thereof, removing purified concentrated rubber latex from the upper part of each of said remaining compartments and removing serum from the lower part of each of said remaining compartments.

5. Apparatus for the continuous concentration and purification of aqueous colloidal dispersions containing dissolved material which comprises a cell divided into an odd number of separate compartments by means of spaced upright semi-permeable membranes, electrodes in the end compartments of the cell, means for feeding water through the electrode compartments and through alternate intermediate compartments, means for delivering an aqueous colloidal dispersion to the remaining compartments at a point between the top and the bottom thereof, and means for separately removing from each of said remaining compartments a colloid-rich layer and a colloid-impoverished layer.

6. Apparatus according to claim 5 in which the said cell is built up of a plurality of vertically disposed frames of non-conducting material and semi-permeable membranes extending between adjacent frames, the end frames and alternate intermediate frames having each an inlet for water and an outlet for water on opposite parts of the periphery of the frame, the remaining frames each having an inlet for colloidal dispersion at an intermediate level, an outlet for colloidal dispersion adjacent the lower part of the periphery of the frame and a further outlet for colloidal dispersion adjacent the upper part of the frame.

7. An apparatus for concurrently concentrating and purifying aqueous colloidal dispersions containing dissolved material substantially as hereinbefore described with reference to the accompanying drawings.

8. Aqueous colloid-rich dispersions whenever obtained by a process according to any of claims 1 to 4.

9. Aqueous colloid-rich dispersions whenever produced by an apparatus according to any of claims 5 to 7.

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Agent for the Applicants.

m.s.e.

PROVISIONAL SPECIFICATION

Improvements in or relating to the Concentration and Purification of Aqueous Colloidal Dispersions

We, DUNLOP RUBBER COMPANY LIMITED, a British Company, of 1, Albany Street, London, N.W.1, and FREDERICK JAMES PATON, a British Subject, of Segil Estate, Tangkak, Johore, Malaya, do hereby declare the nature of this invention to be as follows:—

This invention relates to a process and apparatus for concurrently concentrating and purifying aqueous colloidal dispersions.

It is known to concentrate aqueous colloidal dispersions by subjecting them to an electric potential difference while located between two or more vertical semi-permeable membranes, whereby the colloid particles migrate towards one face of each membrane and form a colloid-rich region in the dispersion. As a result of the density difference thus produced between the parts of the dispersion adjacent to the membranes and the

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remainder of the dispersion, the colloid-rich portion moves vertically either upwards or downwards, according to whether the specific gravity of the colloid particles is less or greater than the aqueous medium. Separation thus results into a colloid-rich layer and a colloid-impoverished layer. The process can be made continuous by continuously supplying colloidal dispersion to the space or spaces between the membranes and continuously removing separately the colloid-rich layer and the colloid-impoverished layer. The electrodes employed to set up the potential difference are normally in separate compartments which communicate with the dispersion through semi-permeable membranes, these compartments being filled with water. If the aqueous phase of the colloid dispersion also contains dissolved material some dialysis occurs in the initial stages of the process, i.e., dissolved material passes into the electrode compartments, but equilibrium conditions are soon achieved, and thereafter diminution in the content of dissolved material in the dispersion does not occur. The nature and concentration of the dissolved material in the aqueous medium are thus substantially those of the untreated dispersion.

The above described method of concentrating colloid dispersions generally referred to as electrodecantation, has been particularly useful for concentrating rubber latex, which contains dissolved material in the serum thereof. The rubber forms a rubber-rich upper layer and a rubber-impoverished lower layer.

The latex frequently contains ammonia as a preservative and the electrode compartments are then filled with aqueous ammonia to prevent the ammonia content from decreasing.

It is an object of the present invention to provide a method whereby an aqueous colloidal dispersion containing dissolved material can be concentrated by the above method and simultaneously purified by removal of dissolved material from the aqueous medium. It is also an object of the invention to provide an apparatus for carrying out such a process.

According to the present invention a continuous process for concurrently concentrating and purifying an aqueous colloidal dispersion containing dissolved material comprises continuously supplying the colloidal dispersion to the space or spaces between a plurality of semi-permeable membranes spaced apart, maintaining an electric potential difference across said membranes to effect a separation of the dispersion into a colloid-rich layer and a colloid-impoverished layer, separately removing said layers from the space or spaces between said membranes in a continuous manner, and continuously removing dissolved substances from the aqueous dispersion between the membranes by dialysis.

According to a further feature of the invention, an apparatus for carrying out the above invention comprises a cell divided into an odd number of separate compartments by means of upright semi-permeable membranes, electrodes in the end compartments of the cell, means for flowing an electrolyte through the electrode compartments and through the intermediate compartments alternate therewith, means for delivering colloidal dispersion to the remaining compartments and means for separately removing from each of said remaining compartments a colloid-rich layer and a colloid-impoverished layer.

Rubber latex can be concentrated and purified according to the invention in apparatus of known type comprising a cell having a latex compartment in which are a plurality of semi-permeable membranes spaced apart, and electrode compartments at each end of the cell each separated from the latex compartment by a semi-permeable membrane. The latex is concentrated in the latex compartment in the manner employed hitherto, and the electrolyte in each electrode compartment is continuously or intermittently changed during the concentration process with a frequency sufficient to prevent equal concentration of dissolved substances being set up in the liquids on each side of the semi-permeable membranes separating each electrode compartment from the latex compartment. As latex is supplied to the latter, concentration and dialysis occur concurrently while latex is passing through the compartment, and a rubber-rich layer is separated in which the serum is at least partly denuded of the dissolved substances originally present therein.

Preferably the process is carried out in the apparatus referred to above having a plurality of latex-containing compartments, each between two electrolyte compartments. When this apparatus is employed, a suitable electrolyte is placed in the electrode compartments and in the alternate compartments between them, and the latex to be purified is continuously supplied to each of the remaining compartments, the latex in any one compartment thus being separated on each side from electrolyte by a semi-permeable membrane. When a suitable electric potential is maintained between

the electrode compartments dialysis occurs through the semi-permeable membranes, and the liquid in the alternate compartments is continuously or intermittently replaced so that conditions permitting the dialysis to occur are maintained throughout the process.

In either of the above forms of the invention the electrolyte fed to the electrode compartments may be water free from any added soluble substances. On the other hand, if it is desired to prevent any particular dissolved substance being removed from the latex the electrolyte fed into the compartments should be water containing such substance in a concentration equal to that in which it is present in the latex. Thus in treating an ammonia-containing latex, removal of ammonia from the latex may be prevented by employing as electrolyte water containing ammonia in the concentration in which it is present in the latex.

The invention may be applied to both rubber latex and other colloid dispersions containing substances dissolved in the aqueous phase; these may be artificial dispersions of rubber, aqueous dispersions of synthetic rubbers or synthetic resins, and aqueous dispersions of other colloids, e.g., casein or colloid dispersions of dyestuffs.

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R. F. McKAY,
Acting for the Applicants.

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