PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improvements in or relating to Method of Producing Concentrated Hevea Rubber Latex

We, THE FIRESTONE TIRE & RUBBER COMPANY, a company incorporated under the laws of the State of Ohio, United States of America, of 1200, Firestone Parkway, Akron, Ohio, United States of America (Assignees of EAKIN MILTON GLYMPH), 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 the production of ammonia preserved Hevea rubber latices and more particularly to the production of such preserved latices which are to be shipped and/or stored in concentrated form prior to fabrication into articles of manufacture.

One reason for latex concentration stems from the great distances which normally separate rubber plantations and manufacturing centers. Lessening the bulk of material to be shipped, by concentrating to remove excess water, effects obvious economy. In addition, high solids latices such as those obtained by concentration are particularly adapted to certain manufacturing processes, e.g., dipping, coating, and the preparation of sponge rubber.

Methods of concentrating latex may be classified into two categories: (1) methods wherein purification is collaterally effected by a distinct separation into a rubber-rich layer and an essentially rubber-poor layer, the latter of which contains most of the non-rubber ingredients and may be separated from the former, e.g., centrifuging, creaming, electrodecantation, and the like, and (2) methods wherein no appreciable purification is effected, e.g., evaporation. For reasons which will become apparent, it is in conjunction with methods of concentrating of the first category that the present invention is concerned. For the purpose of simplification, therefore, the meaning of "concentration" as used hereinafter will be limited to include only said first category.

Many concentrated ammonia preserved latices, particularly those which have been shipped and/or stored for extended periods of time, contain a non-rubber sludge of undetermined origin—apparently formed after concentration—which renders the latices unsatisfactory for normal manufacturing purposes without expensive reprocessing, as for example, centrifugal clarification or filtering.

One means of obviating the difficulty (at the plantation) is to hold concentrated ammonia preserved latices in tanks or vats for extended periods of time (30 to 40 days) and then centrifugally clarify or filter them. Such procedures have the obvious disadvantage of being cumbersome and inefficient in that they not only are time consuming but are also wasteful of man-hours and equipment.

The primary object, therefore, of the present invention is to provide a method for producing concentrated ammonia preserved Hevea rubber latices which may be shipped and stored with substantially no subsequent sludge formation.

A further object is to provide an efficient and economical method of producing substantially sludge-free concentrated ammonia preserved Hevea rubber latices.

A still further object is to provide a method of producing concentrated ammonia preserved natural rubber latices having improved processing qualities.

According to the present invention a method of producing concentrated Hevea rubber latex comprises adding ammonia in an amount sufficient to produce a latex having a pH value of at least 8.5 and not

in excess of 11.0 and at least 0.035% and not more than 0.25% expressed as $PO_4\equiv$ based on the weight of latex of sodium, potassium or ammonium phosphate to Hevea rubber latex, the phosphate addition being insufficient to cause separation and thickening of the latex, and subsequently concentrating the latex by separating it into a rubber-rich layer and an essentially rubber-poor layer, the rubber-rich layer being a concentrated latex which can be shipped and stored with substantially no subsequent sludge formation, said concentrated latex having improved mechanical stability. Preferably, the soluble phosphate is added after the latex has been stabilized; however, the phosphate may be added before the latex is ammoniated. The phosphates may be primary, secondary or tertiary. Ammonium phosphates such as

$(NH_4)H_2PO_4$, $(NH_4)_2HPO_4$, and

$(NH_4)_3PO_4$ have been found to be particularly suitable; however, other soluble phosphates such as sodium and potassium phosphates are also operative and are also within the contemplation of the present invention.

The effectiveness of adding a soluble phosphate to latex, in accordance with the invention, has been found to vary directly with the amount of phosphate added. In order to more specifically define the invention, the soluble phosphate additions set out in the following examples and in the appended claims are given as percent phosphate expressed as $PO_4\equiv$ based on the weight of latex.

EXAMPLE 1.

A sample of ammoniated (0.3% NH_3 on the latex) Hevea rubber latex was divided into two test portions. To one portion 0.072% diammonium phosphate, expressed as $PO_4\equiv$ based on the weight of latex, was added. No phosphate was added to the other portion and it was retained as a control. After the two test portions had stood for about thirty minutes, they were centrifuged. The pH of each concentrate was adjusted to approximately 10.5 (as shown in Table I) by adding ammonia, and then each concentrate was bottled, capped and stored. The sludge which formed in the concentrates was collected on felt filters on the seventh, fourteenth and thirtieth days and reported as percent sludge on the weight of the latex of the original test portions. The following results were obtained.

TABLE I.

	Feed Latex		Concentrated Latex				
	Total solids	pH	Total solids	pH	% Sludge after 7 days	14 days	30 days
No Phosphate added	35.2	9.87	64.6	10.49	.061	.418	.229
Phosphate added	35.1	9.74	64.3	10.45	.044	.040	.036

A substantially sludge-free concentrated latex suitable for normal latex processes was produced by the phosphate treatment, whereas the untreated latex contained a substantial amount of sludge and was unsatisfactory without subsequent reprocessing.

EXAMPLE 2.

Ammonia preserved (containing 0.3% NH_3 on the latex, pH 9.95) field latex, having a total solids content of 43.5 was divided into several samples. To these samples diammonium phosphate was added in amounts varying from 0.000 to 0.720% expressed as $PO_4\equiv$ on the latex (0.00 to 1.00% $(NH_4)_2HPO_4$ on the latex). The samples were allowed to stand overnight and were then centrifuged. The ammonia (NH_3) concentrations of the centrifuged samples were adjusted to 0.70%, resulting in a pH of 10.4, and the samples were stored. The weight and volume of sludge were determined both on the 21st and 30th days. Mechanical stabilities were also determined on both of the above days. Determinations of KOH numbers were, however, made on the 30th day only.

The weight determinations for sludge were made according to the following procedure. A test portion of a sample was diluted 1:1 with 0.5% ammonia water. Forty grams of the diluted latex was weighed into a 50 ml. conical centrifuge tube and centrifuged in a Type C International Centrifuge (Supplier: International Equipment Co., 352, Western Ave., Boston, Mass.) at maximum speed using a head arrangement giving a relative centrifugal force of

1400 for 30 minutes or until a clear liquid zone appeared above the sludge, which was packed in the bottom of the tube. The thick latex paste was then removed from the top of the tube; the liquid latex was siphoned off; and the tube was then filled with an alcohol-ammonia water wash solution and recentrifuged. Washing and recentrifuging were repeated until the supernatant liquid above the packed sludge was clear. After washing, the wet sludge was transferred quantitatively to a weighed flask and dried overnight at 70° C. The amount of sludge is reported in Table II as weight percent based on the original undiluted latex.

The volume determinations for sludge were made in a somewhat similar manner. A test portion of a sample was diluted 1:1 with 0.5% ammonia water and placed in a centrifuge tube having a graduated capillary tube welded on the bottom. The test portion was then centrifuged in a Type C International Centrifuge for 30 minutes at 2000 RPM. Sludge packed in the graduated capillary tube, and its volume was read off directly. The volume of sludge is reported in Table II as volume percent on the original undiluted latex.

TABLE II

Sample	A	B	C	D	E	F	G	H
$\%PO_4\equiv$ on latex	0.000	0.036	0.072	0.144	0.216	0.288	0.360	0.720
Total solids	65.7	64.4	64.7	64.4	64.1	63.3	62.2	61.8
Stored for 21 Days								
Vol. % Sludge	1.000	0.620	0.048	0.061	0.039	0.037	0.048	0.024
Wt. % Sludge	0.155	0.085	0.023	0.023	0.010	0.016	0.026	0.020
Mech. Stability	605	805	760	820	690	650	640	455
Stored for 30 Days								
Vol. % Sludge	1.090	0.630	0.061	0.050	—	0.073	0.048	0.049
Wt. % Sludge	0.133	0.091	0.027	0.030	0.034	0.026	0.018	0.025
Mech. Stability	850	945	1075	925	855	610	695	285
KOH Number	0.56	0.56	0.60	0.67	0.73	0.79	0.88	1.39

The sludge formed in the untreated sample was considerable and was sufficient to render the sample undesirable as such for the manufacturing processes in which latex is normally utilized. On the other hand, improvement was quite marked with the addition of even relatively small amounts of phosphate in accordance with the invention. The addition of only 0.036% phosphate expressed as $PO_4\equiv$ based on the weight of latex, reduced sludge formation by about one-half. Seventy-two thousandths percent reduced the amount of sludge formed to a negligible figure. Greater concentrations of phosphate also reduced the sludge formation but not to a substantially smaller amount.

An additional surprising result of practicing the invention is the increased mechanical stability obtained by adding phosphates up to about 0.25% expressed as $PO_4\equiv$. It should be noted that within this range i.e., at least 0.035% and not more than 0.25% the KOH number is not essentially affected. The practice of the invention, therefore, not only produces essentially sludge-free latices but also produces latices with improved processing properties. It will be appreciated that in Table II of Example 2, only samples B through E lie within the scope of the invention and the other samples in which the amount of phosphate added is outside the range of the invention are given for purposes of comparison.

EXAMPLE 3.

Two samples of fresh latex were taken from each of several different areas of a large Hevea plantation and ammoniated to 0.30% ammonia. Two hundred and forty eight thousandths percent (0.248%) of dihydrogen ammonium phosphate expressed as $PO_4\equiv$ (0.3% $(NH_4)H_2PO_4$ on the latex) was added to one of each pair of samples; the other sample was not treated with phosphate and served as a control. All of the samples were then allowed to stand overnight. They were then centrifuged, ammoniated to 0.70% ammonia, and stored for thirty days in glass bottles. The volumes of sludge formed were then determined according to the procedure set out in Example 2 and are reported below in Table III as volume percent on original undiluted latex. (It should be noted that, as shown in Table II, volume percent and weight percent show essentially the same behaviour. Though the volume percent is not as accurate quantitatively, it is, nevertheless, sufficiently indicative to demonstrate the effectiveness of the invention in reducing the formation of sludge in concentrated latices to a negligible amount).

TABLE III.

Volume Percent of Sludge

Area No.	No phosphate	Phosphate added
1	.784	.037
2	.735	.050
3	.145	.024
4	.079	.025
5	.218	.073
6	.092	.025
7	.919	.049
8	1.110	.024
9	.802	.025
10	1.052	.025
11	.968	.085
12	.579	.099
13	.036	.024

Sludge formation in different concentrated latices varies considerably, as is indicated in Table III. The geographic origin of the latex is apparently an important factor. Nevertheless, soluble phosphates retard sludge formation after concentration regardless of the sludge forming propensity of the initial latex. As is shown in Table III, all of the latices treated were substantially improved.

What we claim is:—

1. A method of producing concentrated Hevea rubber latex which comprises adding ammonia in an amount sufficient to produce a latex having a pH value of at least 8.5 and not in excess of 11.0 and at least 0.035% and not more than 0.25% expressed as $PO_4\equiv$ based on the weight of latex of sodium, potassium or ammonium phosphate to Hevea rubber latex, the phosphate addition being insufficient to cause separation and thickening of the latex, and subsequently concentrating the latex by separating it into a rubber-rich layer and an essentially rubber-poor layer, the rubber-rich layer being a concentrated latex which can be shipped and stored with substantially no subsequent sludge formation, said concentrated latex having improved mechanical stability.

2. A method according to claim 1, in which the phosphate is added to the latex after said latex has been stabilized by the addition of ammonia.

3. A method according to any one of the preceding claims, in which the treated latex is concentrated by centrifuging.

4. A method of producing concentrated Hevea rubber latex substantially as hereinbefore described.

5. Concentrated Hevea rubber latex produced in accordance with the method of any of the preceding claims.

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