

PATENT SPECIFICATION

926,162

NO DRAWINGS.

926,162



Date of filing Complete Specification: Jan. 19, 1962.

Application Date: Jan. 23, 1961. No. 2636/61.

Complete Specification Published: May 15, 1963.

Index at Acceptance:—Class 70, Q5X.

International Classification:—C08c.

COMPLETE SPECIFICATION.

Improvements in or relating to the Preservation of Rubber Latexes.

We, THE NATURAL RUBBER PRODUCERS' RESEARCH ASSOCIATION, a British Body Corporate, of 19 Buckingham Street, Adelphi, London, W.C.2 (formerly of 19 Fenchurch Street, London, E.C.3), do hereby declare the invention (a communication from DEREK HENRY TAYSUM, a British Subject, of The Rubber Research Institute of Malaya, Kuala Lumpur, Malaya), 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 is for improvements in or relating to the preservation of rubber latexes.

In recent years much effort has been devoted to a search for alternative preservatives to ammonia for the natural rubber latex industry. This effort has resulted in the discovery, and the successful industrial use, of such preservatives as formaldehyde, sodium pentachlorophenate, zinc diethyl dithiocarbamate and ammonium borate. The major spheres of use of these preservative materials have been in field latex in the case of formaldehyde and in latex concentrates, containing reduced amounts of ammonia, e.g. 0.2% by weight of the latex in the case of the latter three compounds.

Preservatives which are at present in use for latex which is to be made into sheet or crepe are not effective for storage periods in excess of 24 hours. If ammonia is used in sufficient quantity to keep the latex for several days, additional acid is required for the coagulation of the latex for the production of sheet or crepe and it is difficult to prepare sheet or crepe of good appearance. Hence, there is a great need for a satisfactory preservative which would be capable of keeping latex in good condition for several days and

which would not increase the pH of the latex.

Further, up to the present, the compounds used in the preservation of latex have not been easy to employ in the sterilisation of tapping cups although formaldehyde has been used in the fumigation of factory plant—largely for storage of latex concentrates.

This invention is based on the discovery that ethylene oxide is of great value as a general sterilant for latex. Ethylene oxide produces little or no pH change when added to rubber latexes thus facilitating its generalised use.

According to the present invention, therefore, there is provided a method for the preservation of fresh field latex and latex concentrate, which comprises adding to the latex a small proportion of ethylene oxide, with or without a small proportion of ammonia.

It is now well known that latex tapped from trees under entirely sterile conditions (including the sterilisation of contaminated latex vessels) is a relatively stable material. In contrast to the coagulation due to bacterial action in field latex obtained under normal conditions which occurs within 8 to 14 hours after tapping, sterile latex remains liquid for at least six days and under certain conditions for up to 34 days. Unfortunately, ethylene oxide cannot be used in contact with the tree, since although large volumes of sterile latex are obtained thereby, the ethylene oxide itself proves toxic to the tree and necrosis of the bark ensues. However, ethylene oxide can be introduced into latex as soon as collection from trees has been completed: i.e. it can be added to latex either at the field collecting station or upon reception at the factory. Suitable amounts of ethylene

[Price 4s. 6d.]

oxide range from 0.05% by volume upwards, preferably from 0.1 to 1.0%.

Field latex containing normal levels of viable bacteria (4×10^7 per ml.) is subject to rapid bacterial decay. Within four hours of bulking in the absence of preservative, field latex has undergone sufficient bacterial fermentation to make the manufacture of bubble-free sheet difficult and its volatile fatty acid content (hereinafter referred to as VFA number) has risen rapidly to about 0.10 units. Similarly, delays in the collection and transport of field latex may make the preparation of high quality latex concentrate exceedingly difficult. The addition of 0.5% by volume of ethylene oxide, either as gas or in the form of an aqueous solution thereof, brings about a considerable reduction in the bacterial count; following this addition of ethylene oxide the latex remains liquid for at

least sixty hours before a slow flocculation, followed by coagulation, occurs. Only a small pH change occurs during this time, -0.2 units at maximum. The importance of this preservation at a pH virtually identical to that of fresh latex is most marked in connection with sheet and crepe manufacture. Using 0.5% of ethylene oxide, it is possible to transport liquid field latex having a substantially neutral pH to a central sheeting and creping plant and without any addition of ammonia.

Table 1 compares bacterial counts on latex used to prepare sheet rubber after 3.5 hours storage in the absence of preservative with 3.5, 24 and 48 hours storage in the presence of 0.5% by volume of ethylene oxide. It is clear that the viable bacterial counts on latex are lower with ethylene oxide even after prolonged storage.

TABLE 1.

BACTERIAL COUNTS ON LATEX IN SHEET MANUFACTURE.

Latex description	Viable bacterial count on latex (bacteria/ml.)	V.F.A. number
Immediately on reception—no preservative	5.6×10^6	0.018
Stored for 3.5 hours—no preservative	7.38×10^7	0.031
With ethylene oxide 0.5% by volume—stored for 3.5 hours	1.82×10^6	0.022
Stored for 24 hours—no preservative	1.9×10^9	0.10
With ethylene oxide 0.5% by volume stored for 24 hours	9.4×10^4	0.028
With ethylene oxide 0.5% by volume stored for 48 hours	2.23×10^5	0.032

Small amounts of ammonia may also be added to field latex if desired but the figures given above show that in many cases this will not be necessary. As a rule amounts of 0.01—0.05% by volume are all that are likely to be needed.

In the manufacture of pale crepe rubber one requirement is that the carotenoid-bearing (yellow) particles of latex should not be disrupted by the preservative. If this disruption were to occur, pigmented material would be distributed throughout the latex which cannot be removed by partial coagulation. In this respect we have found that up to 24 hours after the addition of 0.5% by volume of ethylene oxide there is no destruction of carotenoid-bearing Frey-Wyssling particles.

Flocculation of carotenoid-bearing particles in the latices treated with ethylene oxide seems rather easier than in controls. Thus it appears that ethylene oxide will prove a suitable preservative for latex intended for sheet and crepe rubber manufacture. There is no detectable difference between the amount of acid required for the coagulation of latices treated with ethylene oxide and of control latices containing no preservatives.

Turning now to the preservation of latex destined for the manufacture of latex concentrates, the main properties which require to be considered are fluidity (freedom from thickening, flocculation and coagulation), KOH Number, V.F.A. Number and mechanical stability.

The amount of ethylene oxide required

depends upon the time of storage. One per cent by volume of ethylene oxide effectively preserves badly contaminated field latex (V.F.A. 0.075) for at least three days; at the
5 end of this period the V.F.A. Number had only risen slightly to 0.087. Using from 0.1% to 0.3% by volume of ethylene oxide, bulk field latex is preserved for one day; 0.4% by volume of ethylene oxide preserves
10 for two days, 0.5% for three days, and 0.6% to 0.8% for five days without appreciable rise of the V.F.A. Number.

Two latex concentrates each containing 0.2% of ammonia and containing 0.5% and 1.0% ethylene oxide have been compared
15 with concentrates containing 0.2% ammonia and a normal low ammonia-ammonium borate preservative. In Table 2 are given the results of tests up to one month's stor-
20 age.

TABLE 2.
THE PRESERVATION OF LOW AMMONIA LATEX CONCENTRATES BY ETHYLENE OXIDE.

Description of preservative system for latex concentrate	pH				KOH No.				V.F.A. No.				Mech Stability				Bacterial Counts /ml.			
	Storage in days				Storage in days				Storage in days				Storage in days				Storage in days			
	3	10	20	30	3	10	20	30	3	10	20	30	3	10	20	30	3	10	20	30
(Preserved Control)																				
0.2% Ammonia	9.68	9.45	9.38	9.22	0.70	0.76	0.79	0.89	0.010	0.015	0.015	0.016	43	155	205	206	2.5	<10	6	8
0.2% Boric acid																				
0.03% Lauric acid																				
0.2% Ammonia																				
0.5% Ethylene oxide	9.80	9.66	9.58	9.42	0.39	0.40	0.44	0.53	0.011	0.012	0.013	0.013	37	66	69	75	<10	<10	1.25	1
0.2% Ammonia																				
1% Ethylene oxide	9.70	9.55	9.46	9.28	0.30	0.32	0.36	0.47	0.010	0.011	0.014	0.013	29	39	45	48	<10	1×10^2	<10	12
(Unpreserved Control)																				
0.2% Ammonia	9.62	9.25	8.98	8.72	0.62	0.84	1.04	1.25	0.128	0.315	0.316	0.335	30	31	35	42	$>10^6$	8.9×10^7	1.61×10^5	4.87×10^5

From Table 2 it will be seen that the addition of ethylene oxide to concentrated ammonia latices containing as little as 0.2% by volume of ammonia has preserved the latices. The only drawback is the rather low mechanical stability. It has been found that this can be rectified by the addition of a small amount of ammonium laurate to the preserved concentrated latex.

With respect to the amount of ammonia which may be present in concentrated latices in accordance with this invention this may range from as little as 0.05% to as much as 0.75%: amounts of 0.15 to 0.3% are preferred.

Instead of using ammonium laurate to increase the mechanical stability of the latices there may be used small amounts of the ammonium salts of other fatty acids e.g. oleic acid as well as synthetic surface active agents. As a rule not more than 0.05% of lauric acid (converted to the ammonium salt prior to addition) is added to the latices. In Table 3 which follows there is illustrated the effect of adding 0.03% of lauric acid as ammonium laurate upon the properties of a typical concentrated rubber latex containing ethylene oxide: typical controls are also given.

TABLE 3.
THE EFFECT OF AMMONIUM LAURATE ON ETHYLENE OXIDE PRESERVATION.

Description of concentrate preservative	pH			KOH No.			V.F.A. No.			Mech. Stability			Bacterial Counts /ml.		
	Storage in days			Storage in days			Storage in days			Storage in days			Storage in days		
	1	15	30	1	15	30	1	15	30	1	15	30	1	15	30
.7% Ammonia	10.56	10.40	10.36	0.34	0.43	0.49	0.010	0.011	0.008	48	392	671	1×10^3	10	0.75
.2% Ammonia .5% Ethylene oxide	9.82	9.70	9.65	0.35	0.43	0.48	0.012	0.016	0.012	36	142	174	1	10	2.6
.2% Ammonia .5% Ethylene oxide .03% Lauric acid	9.82	9.68	9.62	0.36	0.43	0.49	0.011	0.012	0.011	45	164	698	10	10	10
.2% Ammonia (Control)	9.80	9.65	8.98	0.36	0.85	1.15	0.014	0.247	0.35	44	60	145	3.2×10^4	8.42×10^5	1.4×10^6

It will be seen from the above table that after 30 days storage a concentrated latex containing only 0.2% of ammonia, 0.05% of lauric acid and 0.5% by volume of ethylene oxide had a mechanical stability of 698 seconds : this is regarded as satisfactory.

Rubber latices preserved with ethylene oxide in accordance with the present invention can be used in normal latex manufacturing processes without modifications in the processing.

WHAT WE CLAIM IS:—

1. A process for the preservation of a rubber latex which comprises adding to fresh field latex or to a latex concentrate at least 0.05% of its volume of ethylene oxide.
2. A process according to Claim 1 in which the amount of ethylene oxide added is 0.1 to 1.0% by volume.
3. A process according to either of Claims 1 or 2 in which ammonia is also added to the latex or latex concentrate.
4. A process according to Claim 3 in which 0.15 to 0.3% by volume of ammonia is added to a concentrated rubber latex.

5. A process according to any of the preceding claims in which the mechanical stability of the latex is improved by the addition of a small amount of a surface/active agent.

6. A process according to Claim 5 in which the surface active agent is an ammonium salt of a fatty acid.

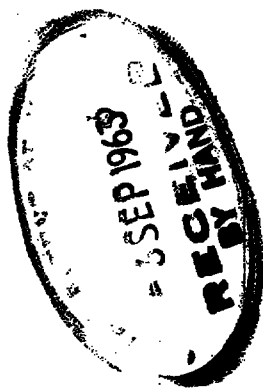
7. A process according to Claim 6 in which the fatty acid is lauric or oleic acid.

8. A process for the preservation of a rubber latex according to Claim 1 and substantially as hereinbefore described.

9. A rubber latex which has been preserved by the presence therein of at least 0.05% of its volume of ethylene oxide.

STEVENS, LANGNER, PARRY
& ROLLINSON,
Chartered Patent Agents,
Agents for the Applicants.

Abingdon: Printed for Her Majesty's Stationery Office, by Burgess & Son (Abingdon), Ltd.—1963
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2,
from which copies may be obtained.





5 Rubber latices preserved with ethylene oxide in accordance with the present invention can be used in normal latex manufacturing processes without modifications in the processing.

WHAT WE CLAIM IS:—

10 1. A process for the preservation of a rubber latex which comprises adding to fresh field latex or to a latex concentrate at least 0.05% of its volume of ethylene oxide.

2. A process according to Claim 1 in which the amount of ethylene oxide added is 0.1 to 1.0% by volume.

15 3. A process according to either of Claims 1 or 2 in which ammonia is also added to the latex or latex concentrate.

4. A process according to Claim 3 in which 0.15 to 0.3% by volume of ammonia is added to a concentrated rubber latex.

5. A process according to any of the preceding claims in which the mechanical stability of the latex is improved by the addition of a small amount of a surface/active agent. 20

6. A process according to Claim 5 in which the surface active agent is an ammonium salt of a fatty acid. 25

7. A process according to Claim 6 in which the fatty acid is lauric or oleic acid.

8. A process for the preservation of a rubber latex according to Claim 1 and substantially as hereinbefore described. 30

9. A rubber latex which has been preserved by the presence therein of at least 0.05% of its volume of ethylene oxide. 35

STEVENS, LANGNER, PARRY
& ROLLINSON,
Chartered Patent Agents,
Agents for the Applicants. SC

Abingdon: Printed for Her Majesty's Stationery Office, by Burgess & Son (Abingdon), Ltd.—1963
Published at The Patent Office, 25, Southampton Buildings, London, W.C.2,
from which copies may be obtained.

