PRELIMINARY OBSERVATIONS ON THE COAGULATION OF ALKALINE LATEX

BY

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Introduction

Various alternatives to the usual acetic and formic acid coagulants have been proposed for the coagulation of Hevea latex in estate practice, generally with the object of reducing production costs. A few have been favourably received and used to a small extent, but most have been rejected for various reasons, and acetic and formic acids are still almost universally used.

Satisfactory coagulation of latex in an alkaline medium is considerably more difficult than it is in the slightly acid condition in which coagulation normally takes place. The production of good sheet rubber from ammonia-preserved, creamed, or centrifugal skim latex by simple acid coagulation is very difficult, and in most cases impossible, as the coagulum is only suitable for crepe manufacture. This may be of no importance in the case of ammoniated or creamed concentrated latex as it is seldom necessary to coagulate either. With centrifugal skim latex containing up to 15 per cent dry rubber, or even more, depending on the type and efficiency of the centrifuge, the disposal of the skim becomes a problem. With the possibility of concentration by centrifugal means showing still further expansion, an alternative coagulant which will enable sheet rubber to be made from the skim becomes valuable, provided its effect on the rubber is not deleterious and the cost is reasonable.

The coagulation of latex in an alkaline medium is therefore under investigation with particular reference to the production of a coagulum which can be made into sheet rubber. Coagulation of the following types of latex has been subjected to a preliminary study:

(a) centrifugal skim latex

(b) creamed concentrate

(c) preserved field latex.

In all cases the preservative used has been ammonia, and coagulation in the presence of other alkalis has not yet been investigated. Although the work is incomplete it is considered that the preliminary results are sufficiently interesting to be placed on record.

Survey of Literature

In recent years, as a result of the use of preserved latex for industrial purposes both as concentrate and as normal latex, a considerable amount of information on coagulation has accumulated. Many of the methods described have particular application to various types of manufacturing processes in which compounded latex is often used. Reference is only made here to the literature concerning those methods which are of interest in connection with the problem under investigation.

R. G. Fullerton (1) discussing the coagulation of ammoniated latex observes that it is more sensitive to acid than is fresh latex, and that the first few drops of acid produce local clotting. R. G. James (2) says it is practically impossible to reproduce with preserved latex the slow type of acid coagulation used on the plantation. Investigations on the properties of the rubber prepared from ammoniated latex by acid coagulation have been made by O. de Vries and his co-workers (3). In all cases however the coagulum was creped. It was observed that the amount of acid required for coagulation was considerably in excess of that required to neutralize the ammonia present and to coagulate the same quantity of fresh latex. Similar observations were made at the Imperial Institute during investigations on crepe rubber prepared from ammoniated latex (4).

In the earlier days of the plantation rubber industry a considerable amount of work was done by Henri, Vernet and others on the use of salts for the coagulation of fresh latex, and it was observed that the effectiveness of the salt is largely determined by the valency of the positive ion. R. G. James states that the precipitation of the rubber particles by salt ions can take place in acid, neutral, or alkaline solution. The effect of the added salt solutions becomes increasingly greater as the pH of the latex approaches the isoelectric point. P. Scholz (5) working on the coagulating coefficient of a number of electrolytes, found that they are rather similar for fresh and ammoniated latex, especially after neutralization of the ammonia with sulphuric acid. The latex used in these experiments was very weak, being diluted to 3.5 per cent dry rubber content.

Coagulation is generally considered to be the coalescence of the flocculent rubber particles, first formed by the action of the coagulant, into a coherent clot of coagulum. With ammoniated latex this flocculent condition of the particles is produced without further coalescence, when certain of the heavy metal salts are used as coagulants. Aluminium chloride and sulphate, which give normal coagulation with fresh latex, produce only a flocculation with ammoniated latex. C. C. Loomis and H. E. Stump (6) working with latex of 30—38 per cent dry rubber content preserved with 0.5 per cent ammonia, describe the creaming effect obtained with aluminium sulphate and salts such as zinc and lead acetates. The cream consists of small flocculent particles which are redispersed on stirring. This method of semi-coagulation is used in certain industrial processes where a discontinuous coagulum is required.

For certain manufacturing processes it is necessary to produce from compounded normal or compounded concentrated latex mixes, a continuous type of coagulum, similar in many respects to that produced in the coagulation of fresh latex. The ease with which the coagulant can be distributed and its coagulating speed are obviously important. A delayed-action type of coagulant, such as sodium silicofluoride, is often used in these circumstances. Sodium silicofluoride has been advocated as an alternative coagulant for the coagulation of fresh latex (7—10) and is said to be better in some respects than acids. In the manufacturing processes in which it is used, the speed of coagulation can be controlled by the degree of alkalinity in the mix or by the addition of some powder such as zinc oxide (11).

The delayed coagulating action of sodium silicofluoride with highly compounded concentrated latex mixes, was recently used during investigations on the use of latex mixes for rubber roadways (12).

There is, however, considerable difference between the coagulation of compounded latex and that of an uncompounded and dilute latex with which this investigation is mainly concerned. With technical mixes, coagulation is under greater control, because it is possible, by the judicious use of stabilisers, to facilitate compounding and also to adjust the latex to that condition of stability most suited to the particular coagulant used.

Formaldehyde when added to preserved latex in amounts more than sufficient to neutralise the ammonia present, will cause gelling. With uncompounded latex it is only effective if the ammonia content is not greater than 0.05 per cent (13). By adding a sensitising agent such as zinc sulphate, or ammonium sulphate, it is possible to bring about gelling with latex of higher ammonia content. The higher the alkalinity, however, the greater is the amount of formaldehyde required in excess of that needed to neutralize the ammonia present.

O. de Vries (14) states that papain contains a coagulating enzyme for latex which is active in an alkaline medium. A solution of papain was recently used by E. Rhodes and J. L. Wiltshire* for the coagulation of centrifuged skim. It is, however, difficult to say how far the modified properties of the resulting rubber are due to changes in the latex as a result of centrifuging, or to the proteolytic action of the papain. In this connection it may be noted that it is very difficult to dry and prepare normal sheet rubber from fresh latex by papain coagulation, while J. Groenewege (15) reports that rubber so prepared quickly becomes tacky.

A process has been described by H. Colloseus (16) for coagulation by means of salts, oxides, hydroxides of the alkaline earths or heavy metal salts, latex which has been previously treated with alkali or other bodies to form water-soluble compounds with the albuminoids. W. Spoon (17) discussing the methods considers it is not suitable for the coagulation of Hevea latex.

S. C. Davidson (18) has patented a method of coagulating latex preserved with alkalized phenols, (alkaline mixtures of cresols and isomers, cresylic acids and higher tar acids) by means of a mixture of magnesium sulphate, or magnesium chloride and commercial sulphuric acid. The coagulum is then immersed in boiling water to make it sufficiently pliable for machining. He mentions that the sulphate gives better coagulation than the chloride. This observation is of interest in connection with some of the experiments to be described.

Experimental

The difference between the coagulum obtained from ammoniated and fresh latex by coagulation with a solution of aluminium sulphate is one of degree rather than of kind; in both cases a clear serum can be obtained. It seemed probable that if the non-coherent flocculent particles which result in the case of the ammoniated latex could be made to coalesce uniformly, either immediately or within a reasonable time, it should be possible to deal with the final coagulum in the same manner as with that from fresh latex. Structural differences in the rubbers would no doubt exist, since the flocculent particles produced by the aluminium sulphate in ammoniated latex are considerably larger than those occurring in the coagulation of fresh latex, where the change from latex to solid coagulum involves the coalescence of extremely small floccules.

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*Unpublished work.

CENTRIFUGAL SKIM LATEX

Attempts to effect the coalescence of the flocculent particles were made with a centrifugal skim latex by the addition of a solution of sodium silicofluoride together with aluminium sulphate. The skim latex used was 2½ months old, its D.R.C. 14.7 per cent and the ammonia content had been raised to 0.61 per cent for preservation. Varying amounts of a 0.5 per cent solution of sodium silicofluoride were added to the skim latex together with a 5 per cent solution of aluminium sulphate; no coherent coagulum resulted. Other salts, notably aluminium chloride, zinc chloride, barium chloride, sodium dihydrogen phosphate, and calcium chloride were tried with sodium silicofluoride but no better results were obtained. With magnesium chloride, however, a good coherent coagulum resulted and magnesium sulphate also showed promise. Results were sufficiently encouraging to continue with magnesium chloride and only a few experiments were made with other salts. It is quite possible that some of those which at first gave no indication of being successful will be found satisfactory on further investigation. This observation applies particularly to calcium chloride.

Table I gives the results of the addition of a mixture of different amounts of a 5 per cent. solution of magnesium chloride and a 0.5 per cent. solution of sodium silicofluoride to 100 cc. of skim latex.

TABLE I

Ref. No.	Volume of Coagulant per 100 cc. Skim Latex		Immediate Effect of the Coagulant on the Latex	Effect after 20 hours
	5 per cent $MgCl_2$ cc.	0.5 per cent S.S.F. cc.		
L 57/1	10	50	None	Latex thickened
L 57/2	15	45	,,	Coagulation, with coherent coagulum
L 57/3	20	40	Latex thickened and flocculated	,, ,,
L 57/4	25	35	,,	Coagulation with very "short" coagulum,
L 57/5	30	30	,,	,, ,,

The best coagulum appeared to be either L 57/2 or L 57/3. The coagulum was soft but coherent, while the serum was dark brown. From the appearance of a coagulum made under the trial coagulation conditions of the laboratory it is difficult to decide whether the same coagulum will be suitable for sheet manufacture when prepared on a larger scale. On several occasions larger-scale trials have given a coagulum which proved less easy to handle than was expected from the laboratory experiments. The magnesium chloride solution was mixed with the sodium silicofluoride solution before addition to the latex. When magnesium chloride was added first to the skim latex, local clotting occurred. The effect of the separate addition of the solutions was therefore examined.

A 0.5 per cent. solution of sodium silicofluoride added in amounts from 5 cc. to 100 cc. per 100 cc. of skim latex had no immediate effect and no coagulation had occurred by next day. The clotting which had been observed previously was therefore due apparently to the magnesium chloride. With a 5 per cent. solution of magnesium chloride clotting occurred immediately. The clotting was similar to that produced with acid. By using a weaker solution of magnesium chloride this local clotting was prevented. Dilution of the magnesium chloride is, in fact, achieved by mixing the sodium silicofluoride solution with the magnesium chloride solution before addition to the latex.

TABLE II

Ref. No.	Coagulant Solution containing per 100 cc. Skim Latex		Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
	5 per cent $MgCl_2$ cc.	Water cc.		
L 66/1	15	0	Severe local coagulation	No further change
L 66/2	15	10	,, ,, ,,	,, ,,
L 66/3	15	20	Local coagulation	,, ,,
L 66/4	15	25	Slight local coagulation	,, ,,
L 66/5	15	30	No local coagulation	No coagulation
L 66/6	15	45	,, ,, ,,	,, ,,

Table II gives the results of diluting a fixed volume of 5 per cent. magnesium chloride solution before addition to 100 cc. of skim latex.

It is seen from sample L 66/5 in the above table that the addition of a solution of magnesium chloride stronger than 1.6 per cent., caused local clotting when added to this sample of skim latex. As samples Nos. L 57/2 and L 57/3 (Table I) gave good coagulum, the volumes of magnesium chloride used in experiments with this sample of skim latex were kept constant while variations were made in the amounts of sodium silicofluoride added to these volumes before mixing with the skim latex. The results are shown in Table III.

TABLE III

Ref. No.	Volume of Coagulant per 100 cc. Skim Latex		Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
	5 per cent $MgCl_2$ cc.	5 per cent S.S.F. cc.		
L 64/1 A	15	20	Local coagulation	No further coagulation
L 64/1 B	15	25	" "	" " "
L 64/1 C	15	30	No local coagulation	" " "
L 64/1 D	15	35	" "	Coagulation, coherent coagulum
L 64/1 E	15	40	" "	" " "
L 64/1 F	15	45	" "	" " "
L 64/2 A	20	20	Local coagulation	No further coagulation
L 64/2 B	20	25	" "	Coagulation
L 64/2 C	20	30	Thickening, slight local coagulation	"
L 64/2 D	20	35	Flocculation, slight local coagulation	Coagulation, coagulum "short"
L 64/2 E	20	40	Flocculation, no local coagulation	" " "

Although a coherent coagulum with a clear serum was obtained in L 64/1 D it was extremely soft and such a coagulum could not be handled for the manufacture of sheet. L 64/1 E and L 64/1 F were also soft but firmer than L 64/1 D. With L 64/2 D and L 64/2 E the coagulum was of a different type. The flocculent particles had united to form a coagulum which, although coherent, had an open texture comparable to a sponge and was easily torn. This type of coagulum appeared to result when flocculation happened immediately after addition of the coagulant mixture.

In general the greater the degree of flocculation the "shorter" the coagulum. In cases where this happened, coagulation was very rapid, and separation into serum and coagulum was complete in a few hours.

Factory coagulation in coagulating pans was tried using the coagulant proportions found most promising in the trial coagulations. Pans containing half a gallon of skim latex together with mixtures of the following proportions were prepared:—

L 67/1 12 fluid ounces of 5 per cent. magnesium chloride solution and 32 fluid ounces of 0.5 per cent. sodium silicofluoride solution

L 67/2 12 fluid ounces of 5 per cent. magnesium chloride solution and 36 fluid ounces of 0.5 per cent. sodium silicofluoride solution

L 67/3 16 fluid ounces of 5 per cent. magnesium chloride solution and 24 fluid ounces of 0.5 per cent. sodium silicofluoride solution.

These amounts correspond to the proportions in L 64/1 E, L 64/1 F, and L 64/2 C (Table III). With L 67/1 and L 67/2 no local clotting took place. A slight thickening was noticeable however while the coagulant was being stirred into the latex. With L 67/3, rapid thickening occurred; flocculation had started before the surface bubbles had been removed from the pans, and a soft coagulum was formed in half an hour.

The latex in all the pans coagulated overnight but there were differences in the coagulum. Within each of the three series of experiments the hardness of the coagulum varied as well as the condition of the serum. In some pans the serum was clear and dark brown in colour, while in others the colour was dirty brownish white. The coagulum of L 67/1 and L 67/2 was slippery and somewhat difficult to handle, especially during the first few machinings. Considerable care in machining was necessary but, once the machine settings for the various stages were determined, it was possible to prepare good sheet rubber.

The unexpected variation in hardness of the coagulum from pan to pan in the same series was disturbing. A number of possible reasons for this variation suggested themselves and these were tested experimentally but without success; the precise cause of the variation remains for the present, therefore, obscure.

In the latter experiments, it was found that the magnesium salt and the sodium silicofluoride could be applied as a single solution and that the mixture used in sample L 67/1 was effective. This solution contained 1.36 per cent of magnesium chloride

TABLE IV

$MgCl_2$—S.S.F. Solutions with diluted Skim Latex D.R.C. 7.3 per cent. NH_3 content 0.3 per cent.

Ref. No.	Coagulant Solution containing		Volume of Coagulant per 100 c.c. of Latex c.c.	Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
	$MgCl_2$ per cent	S.S.F per cent			
L 76/1	1.36	0.36	30	None	No coagulation
/2	"	"	35	"	"
/3	"	"	40	"	Coagulation: soft coagulum, serum clear
/4	"	"	45	Flocculation soon after addition	Coagulation: "short" coagulum, serum clear
/5	"	"	50	Flocculation immediately after addition	"
L 79/1	1.50	0.25	20	None	No coagulation
/2	"	"	25	"	"
/3	"	"	30	"	"
/4	"	"	35	"	"
/5	"	"	40	"	"
L 80/1	1.25	0.50	20	None	No coagulation
/2	"	"	25	"	"
/3	"	"	30	"	"
/4	"	"	35	"	"
/5	"	"	40	"	Coagulation: very soft coagulum

TABLE IV—(contd.)

Ref. No.	Coagulant Solution containing MgCl$_2$ per cent	Coagulant Solution containing S.S.F. per cent	Volume of Coagulant per 100 cc. of Latex cc.	Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
L 77/1	1.25	0.75	20	None	No coagulation
/2	"	"	25	"	"
/3	"	"	30	Flocculation soon after addition	Coagulation: "short" coagulum, serum clear
/4	"	"	35	Flocculation immediately after addition	Coagulation: "short" coagulum, serum clear
/5	"	"	40	"	Coagulation: very "short" coagulum, serum clear
L 81/1	1.75	0.25	20	None	No coagulation
/2	"	"	25	"	"
/3	"	"	30	"	"
/4	"	"	35	"	"
./5	"	"	40	"	"
L 82/1	1.75	0.50	20	None	No coagulation
/2	"	"	25	"	"
/3	"	"	30	Flocculation soon after addition	Coagulation: very soft coagulum, serum clear
/4	"	"	35	Flocculation immediately after addition	Coagulation: soft coagulum, serum clear
/5	"	"	40	do.	Coagulation: very "short" coagulum, serum clear

TABLE IV—(contd.)

Ref. No.	Coagulant Solution containing		Volume of Coagulant per 100 cc. of Latex cc.	Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
	$MgCl_2$ per cent	S.S.F. per cent			
L 83/1	1.75	0.75	10	None	No coagulation
/2	"	"	15	"	"
/3	"	"	20	"	"
/4	"	"	25	"	Coagulation: very soft coagulum, serum clear
/5	"	"	30	Flocculation immediately after addition	Coagulation: soft coagulum, serum clear
L 84/1	2.00	0.75	5	None	No coagulation
/2	"	"	10	"	"
/3	"	"	15	"	"
/4	"	"	20	"	"
/5	"	"	25	Flocculation soon after addition	Coagulation: "short" coagulum, serum clear

together with 0.36 per cent of sodium silicofluoride. A coagulum which could be converted to sheet was obtained by using 88 ounces of the solution per gallon of latex. The sheet rubber, after drying in hot air at a temperature of 120°—130°F, was of excellent appearance and free from bubbles and stickiness. It is worthy of note that in the case of sheet rubber prepared in the Institute by papain coagulation of skim latex from the same source, the rubber was much softer, inclined to be tacky and much more difficult to dry.

Experiments with Diluted Centrifugal Skim Latex

The experiments described above were made with a sample of skim latex having the comparatively high D.R.C. of 14.7 per cent and the coagulation of a diluted skim made from it, was next considered. The skim latex was diluted with an equal volume of water and treated with varying amounts of solutions containing magnesium chloride and sodium silicofluoride in different proportions. The results are given in Table IV.

The indications were that satisfactory coagulation should result from the application of 40—45 cc. of a coagulant containing 1.36 per cent magnesium chloride and 0.36 per cent sodium silicofluoride to 100 cc. of latex, which corresponds to 64—72 fluid ounces per gallon of latex. In larger-scale pan coagulation an amount of coagulant equivalent to 68 fluid ounces per gallon of diluted skim latex was found to give a coagulum which, although too soft for immediate machining, was easily handled after soaking for 24 hours in water.

The diluted skim latex required, therefore, as might have been expected, a smaller amount of the coagulant than the parent skim, but the coagulum obtained was less compact. The trials with this sample of diluted skim latex indicated that, after dilution, it was much less sensitive to magnesium chloride than the parent skim latex. The addition to the parent skim latex of solutions containing more than 1.6 per cent magnesium chloride resulted in immediate clotting, but the once-diluted skim was tolerant to magnesium chloride solutions up to a concentration of 2.75 per cent.

Utermark Skim Latex of low initial Dry Rubber Content

Attention was next directed to the coagulation of skim latex from a centrifuge of somewhat higher efficiency. Such a skim latex would have a greater concentration of non-rubber solids than a diluted latex made from a skim of high original dry rubber content, and the coagulation phenomena were not expected to be identical. By courtesy of Dunlop Plantations Ltd., it was possible to obtain for trial a skim latex of suitably low dry rubber content. In order to ensure safe transport, however, the ammonia content was of necessity increased considerably above that of normal skim latex as it emerges from the centrifuges. In small-scale trials it became apparent that the Utermark skim latex was much less sensitive to magnesium chloride and sodium silicofluoride than the richer skim latex previously examined. It was therefore found necessary to employ coagulants of which the two components were present in considerably higher concentrations. The results of a series of pan coagulations with coagulants of various concentrations are set out in Table V.

TABLE V

Coagulation of Utermark Skim Latex
D.R.C. 10.7 per cent. NH_3 content 0.70 per cent.

Ref. No.	Coagulant Solution containing		Volume of Coagulant per gallon of Latex oz.	Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
	$MgCl_2$ per cent	S.S.F. per cent			
L 86/12/1	2.0	0.6	50	None	Coherent coagulum in clear brown serum: Coagulum had a number of bad cracks which made sheeting impossible
L 86/12/2	,,	,,	55	Slight flocculation	Similar but coagulum was "shorter"
L 86/13/1	2.25	0.6	45	None	Coherent coagulum in clear serum but badly cracked
L 86/13/2	,,	,,	50	Slight flocculation	,, ,, ,,
L 86/13/3	,,	,,	54	Flocculation	Coagulum very "short", easily torn
L 86/14/1	2.50	0.6	42	None	No coagulation: latex thickened only
L 86/14/2	,,	,,	45	Slight flocculation	Coherent coagulum made into sheet, serum not so clear
L 86/14/3	,,	,,	50	Flocculation	Coagulum too "short" to machine

Good air-dried sheet rubber, free from bubbles and of excellent general appearance was prepared from the coagulum L 86/14/2 of which the coagulant dose was 45 fluid ounces of a solution containing 2.5 per cent magnesium chloride and 0.6 per cent sodium silicofluoride per gallon of skim latex.

It was observed by trials that, on storage, the reaction of the skim latex to the coagulant mixtures is modified and that a dose of coagulant which is effective on a certain day, when applied a few days later gives an unsuitable coagulum.

Trials in small experimental tanks also indicated that in general the quality of the product from tank coagulation is slightly better than that from coagulation in pans. This is probably due to the fact that the cohesion of the initial flocculates is improved by a self-compacting of the coagulum while standing overnight in comparatively deep vessels.

That variations in one or more of the factors, dry rubber content, ammonia content, and non-caoutchouc constituents of different samples of skim latex, may produce variation in their response to the coagulant mixture, is illustrated by experiments made on another sample of Utermark skim latex, of which the dry rubber content and ammonia content were different from those of the first sample. It was at once found that proportions of coagulant which gave a satisfactory sheet coagulum with the first sample were unsuitable for the coagulation of the second, and a series of laboratory trials were accordingly made with a range of sodium silicofluoride—magnesium chloride coagulants and also with a range in which magnesium sulphate was substituted for the chloride. The more important results are set out in Table VI and indicate that by the use of magnesium sulphate in suitable proportion, slightly better results might be expected than by the use of magnesium chloride. Subsequent large-scale trials confirmed this finding and good sheet was eventually produced by its use in tank and pan coagulation.

In tank coagulation there is a tendency for bubbles of air or ammonia to be entrained in the spongy coagulum and to be locked therein during the early stages of machining. With a reduction of thickness in the later stages of machining, quite large blisters may be observed which may or may not burst on the last smooth roll or on the marking roll. The loss of entrained gas or air is also largely responsible for the extreme shrinkage which invariably takes place in the skim latex sheet on drying.

Creamed Latex

Preliminary trials were next made with creamed latex and for the initial experiments a gum tragacanth cream having a D.R.C. of 51 per cent and an ammonia content of 0.67 per cent was used. The cream had been prepared a little over a month and was diluted to 15 per cent D.R.C. before use. At this dilution it was very sensitive to magnesium chloride and small-scale trials indicated that for satisfactory coagulation the most suitable solution would be that containing 1.36 per cent magnesium chloride and 0.36 per cent sodium silicofluoride.

TABLE VI

Coagulation of Utermark Skim Latex
D.R.C. 12 per cent. NH_3 content 0.57 per cent.

Ref. No.	Coagulant Solution containing			Volume of Coagulant per 100 cc. of Latex cc.	Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
	$MgCl_2$ per cent	$MgSO_4$ per cent	S.S.F. per cent			
L 111/A1	2.5	—	0.6	20	None	No coagulation
/A2	"	—	"	25	"	"
/A3	"	—	"	30	"	Coagulation: soft coagulum, serum dirty white
/A4	"	—	"	35	"	Coagulation: soft coagulum, serum dirty white
/A5	"	—	"	40	"	Coagulation: cracked coagulum, serum clear
L 111/B1	2.7	—	0.7	20	None	No coagulation
/B2	"	—	"	22½	"	"
/B3	"	—	"	25	"	"
/B4	"	—	"	27½	"	Apparent coagulation: flocculent, redispersible coagulum
/B5	"	—	"	30	"	Apparent coagulation: flocculent, redispersible coagulum
L 111/C1	3.0	—	0.6	20	None	No coagulation
/C2	"	—	"	25	"	"
/C3	"	—	"	30	"	Coagulation: soft coagulum, serum dirty white
/C4	"	—	"	35	Flocculation shortly after addition	Coagulation: soft coagulum, serum clear

TABLE VI—(contd.)

Ref. No.	Coagulant Solution containing			Volume of Coagulant per 100 cc. of Latex cc.	Immediate Effect of the Coagulant on the Latex	Effect after 20 Hours
	$MgCl_2$ per cent	$MgSO_4$ per cent	S.S.F. per cent			
L 111/C5	3.0	—	0.6	40	Flocculation shortly after addition	Coagulation : "short" coagulum, serum clear
L 111/D1	—	3.0	0.5	25	None	No coagulation
/D2	—	,,	,,	30	,,	,,
/D3	—	,,	,,	35	,,	Coagulation : soft coagulum, serum dirty white
/D4	—	,,	,,	40	,,	Coagulation : firm coagulum, serum clear
/D5	—	,,	,,	45	,,	Coagulation : "short" coagulum, serum clear
L 111/E1	3.5	—	0.3	25	None	No coagulation
/E2	,,	—	,,	30	,,	,,
/E3	,,	—	,,	35	,,	,,
/E4	,,	—	,,	40	Flocculation shortly after addition	Coagulation : soft coagulum, serum clear
/E5	,,	—	,,	45	Flocculation shortly after addition	Coagulation : "short" coagulum, serum clear
L 111/F1	—	3.5	0.3	25	None	No coagulation
/F2	—	,,	,,	30	,,	,,
/F3	—	,,	,,	35	,,	,,
/F4	—	,,	,,	40	,,	,,
/F5	—	,,	,,	45	,,	,,

The following series of pan experiments was made with varying amounts of this coagulant solution:—

Volume of Coagulant Solution in fluid ounces added to one gallon of creamed Latex diluted to a D.R.C. of 15 per cent.

Sample	L88/1A	L88/1B	L88/1C	L88/1D	L88/1E	L88/1F	L88/1G	L88/1H
Volume added	48	50	52	54	56	58	60	62

No coagulation took place with L 88/1A, while L 88/1B and L 88/1C gave coagulum too soft for successful handling on sheeting rolls and the serum was very milky. From L88/1D onwards the coagulum was firmer and the serum progressively clearer, until with 62 fluid ounces per gallon it was quite clear. The coagulum was easily machined and the resulting sheets were of excellent appearance, although when dried at 120°—130°F they were sticky and soft. When the cream was diluted with one and a half volumes of water to one volume of cream, giving a little higher D.R.C., 62 fluid ounces of coagulant per gallon still gave good coagulation. Coagulation in a tank gave coherent coagulum of which the upper edges were firmer than the lower edges; on this account it was difficult to avoid tearing the sheets in the later stages of machining. With another sample of cream, having a D.R.C. of 52 per cent, and an ammonia content 0.60 per cent, coagulation under similar conditions gave a satisfactory coagulum and good sheet, although blisters due to occluded gas gave trouble in machining.

Ammoniated Latex

(i) *Preserved dilute Latex*

Laboratory trials were first made with latex which had been diluted to a D.R.C. of approximately 15 per cent and preserved for 22 weeks with 0.5 per cent ammonia. Small-scale trials indicated that the most suitable coagulant solutions would be

(a) a solution containing 2.5 per cent magnesium chloride and 0.6 per cent sodium silicofluoride

and (b) a solution containing 3.5 per cent magnesium sulphate and 0.3 per cent sodium silicofluoride.

The following pan coagulations were made with the second coagulant solution:—

Volume of Coagulant Solution in fluid ounces added to one gallon of preserved dilute Latex of Dry Rubber Content 14 per cent.

Sample	L104/6A	L104/6B	L104/6C	L104/6D
Volume added	38	36	34	32

Good coagulation resulted, but the serum from L 104/6C and L 104/6D was not so clear as the others. Coagulation was very rapid, and in all cases good coagulum had formed within an hour. On standing overnight considerable contraction occurred in the coagulum. The dried sheets were extremely sticky and soft. The serum had a strong odour of ammonia and samples taken from a number of pans of L 104/6A and L 104/6B were found to contain 0.28 to 0.30 per cent of ammonia. Similar results were obtained with a similar latex which had been preserved for eleven weeks. In this case, however, the dry sheets were less soft and sticky. The same coagulant solution was found to be effective for similar dilute latex preserved for shorter periods but, where the preservation period was of the order of 1—7 days, the coagulum was undesirably soft and the serum cloudy.

(ii) *Preserved field Latex*

Field latex preserved with 0.5 per cent ammonia and stored for 24 hours was effectively coagulated with the coagulant solution containing 3.5 per cent magnesium sulphate and 0.3 per cent sodium silicofluoride, when applied in the proportion of 60 fluid ounces of coagulant to one gallon of undiluted preserved field latex. The effect of longer storage on the reaction of field latex to this coagulant mixture was next considered, and in the course of the examination, good sheet rubber was produced from undiluted preserved field latex which had been stored for periods of five days, one month, two months and three months respectively. The amount of coagulant required was in general slightly larger, and of the order of 70 fluid ounces per gallon of latex. The coagulum, although obtained from latex containing well over three pounds of dry rubber per gallon, was remarkably plastic and therefore easily machined into a thin sheet.

Until many more samples of stored latex have been examined, it cannot be said with certainty that preserved latex from any source and of any age will be readily amenable to smooth coagulation, but the indications at the present stage are promising.

FRESH LATEX

Magnesium chloride—sodium silicofluoride mixtures have been applied to the coagulation of fresh latex in the undiluted condition and at many dilutions down to a dry rubber content of 10 per cent or approximately 1 lb. per gallon. Excellent coagulation has always resulted. Coagulation is more rapid than when sodium silicofluoride is used alone, and the rate of coagulation increases with increase in the concentration of magnesium chloride in the mixture. Coagulum machined on the following day is slightly softer than formic acid controls, and with similar machining procedure yields sheets of which the area is 7 to 10 per cent larger.

Preliminary vulcanisation tests indicate that sheet rubber prepared by the use of magnesium chloride—sodium silicofluoride mixtures is slightly slower-curing than rubber prepared by the use of formic acid.

Discussion

It has been shown that preserved latex, whether in the form of normal field latex, latex concentrate or centrifugal skim latex, is an extremely difficult medium for the attainment of that smooth and accurate coagulation which is essential in the manufacture of sheet as distinct from crepe rubber. The preservative present produces progressive changes in the non-rubber constituents which result in progressive changes in the reaction of the latex to coagulants. The rate at which these changes take place is dependent to some extent on the amount of alkali present as well as upon time, so that, from the point of view of coagulation, the term "preserved latex", even when applied to preserved field latex coagulated shortly after collection, covers a range of fluids of very great variability. The present preliminary investigation has revealed coagulant mixtures by the use of which the desired smooth coagulation can be achieved and good sheet rubber prepared from numerous forms of preserved latex. It has unfortunately also demonstrated that, although it is comparatively easy to induce a smooth, quick and clean coagulation giving a coagulum which is quite suitable for the manufacture of crepe rubber, the limits between which this coagulum is of a texture suitable for sheet manufacture are very narrow. Inherent variability, coupled with the changes which take place on storage, make it impossible to predict the precise concentration and amount of the coagulant which will yield a coagulum of just the right texture for sheet manufacture. It has been demonstrated however that, if preliminary trials are made in order accurately to determine quantities for each individual sample, sheet rubber can be produced from almost any form of preserved latex. In general the minimum quantity of coagulant produces a coherent coagulum which is so soft that it cannot be handled without distortion. Such a coagulum is unsuitable for sheet manufacture. As the amount of coagulant is increased, the coagulum becomes firmer. Further increase in the amount produces a coagulum which is "short" and, if too great an excess is used, the coagulum may become so "short" that coherence is entirely lost. In the manufacture of sheet rubber from skim latex the best results are usually obtained from coagulum which is reasonably firm but which shows some signs of "shortness". When dealing with creamed or normal ammoniated latex, coagulum which is firm without being "short" can be handled without much difficulty. Where it is desired to manufacture crepe rubber the problem of a suitable texture of coagulum largely disappears, and any of the coagulum, except that which is completely non-coherent, can be satisfactorily treated.

It has not so far been possible to attempt the coagulation of fresh centrifugal skim latex of low ammonia content and it is therefore not possible to state with certainty whether day-to-day variations in dry rubber content and physico-chemical condition of the skim would render this method of coagulation unsuccessful for the routine manufacture of skim latex sheet rubber. It seems fairly certain, however, that it could be applied to the manufacture of skim latex crepe rubber without great difficulty.

It seems probable also that where, as happens occasionally, estates wish to produce a marketable rubber from old stocks of unsaleable ammoniated or creamed latex, the use of coagulant mixtures of this type might prove useful. It should again be emphasised, however, that preliminary small-scale trials would be necessary.

Although the coagulant mixtures described give good results with fresh latex it is improbable that they will find an application in estate practice because of their corrosive action on aluminium.

The results recorded are only a preliminary survey of the coagulation of alkaline latex. They indicate that mixtures of sodium silicofluoride with magnesium chloride or sulphate, and possibly with other salts can be used for the purpose. A more detailed examination of the problem is necessary to determine whether some of the findings can find useful application in estate practice.

Summary

1. Mixtures of sodium silicofluoride and magnesium chloride or sulphate can be used for coagulation of alkaline latex.
2. The production of a coagulum suitable for sheet manufacture is described.
3. Preliminary experiments on the coagulation of centrifugal skim latex, creamed latex and ammoniated latex are described.

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