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DEVELOPMENTS IN SHEET RUBBER PROCESSING AND LATEX TESTING

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The Rubber Research Institute of India (RRII) has been undertaking research and development work on processing of sheet rubber and latex, with a view to reducing the cost of processing and improving the quality. The results of some of the experiments undertaken by us in these lines are presented in this paper. Even though formic and acetic acids have been used as coagulants for the preparation of sheet rubber, a higher cost of these acids prompted the farmers to use stronger and cheaper acids such as the sulphuric acid. Foreseeing the harmful effects of unscientific usage of sulphuric acid on the quality of sheet rubber, the RRII has conducted a detailed study on this aspect and brought out the standard conditions under which sulphuric acid can be safely used to coagulate latex, without adversely affecting the quality of sheets and minimizing the corrosion of sheeting rolls and other utensils used in sheet rubber processing. In another experiment it was found that use of a mixture, consisting of formic acid, calcium chloride and sodium chloride, is very effective in coagulating latex for sheet rubber production. This new coagulant mixture is cheaper than formic acid and the coagulum obtained becomes ready for sheeting within 3 to 4 hours. The quality of sheets is excellent, with no adverse effect on storage behaviour and technological properties.

Expense for drying of sheeted coagulum contributes a major share on the processing cost in sheet rubber production. To reduce this, the RRII has developed a cost effective drying system consisting of a smoke house and a specially designed trolley. This new system allows maximum use of sunlight for drying the sheets during daytime and then to continue the drying in the smoke house during the night. It takes only four to five days to get the sheets dried and the quality of sheets is excellent. There could be 40 to 60% saving in firewood during summer season and 15 to 20% saving during rainy season. Fungus affects rubber sheets on storage under humid condition. Presence of other contaminants such as dirt, dust and foreign particles also adversely affects the grade of rubber sheets. For upgrading the quality of such surface contaminated sheets, the RRII has developed a semiautomatic machine for cleaning the sheets. This machine has an output of 750-800 kg sheets per hour and requires only 2.4 kWh electrical energy for cleaning one tonne of sheets. The machine is easy to operate, requires less labour and has high efficiency in upgrading the quality of sheets by effective removal of the surface contamination.

For a quick estimation of the dry rubber content (DRC) of fresh field latex, the most common practice is to use a metrolac. Even though this is a very quick and convenient method, the level of accuracy is less and there is often considerable variation in DRC values obtained by using two different metrolacs. The RRII has developed a method for quick determination of DRC of fresh field latex by titrating soap treated latex with standard dilute sulphuric acid. This new method gives more accurate DRC values than the metrolac method. The procedure is simple to follow, reasonably quick and requires only common laboratory equipment to perform the estimation. Some of the quality parameters of concentrated natural rubber latex such as mechanical stability time, viscosity, KOH number and volatile fatty

acid number continue to change with time during storage/transportation. Hence the latex processors often find it difficult to supply latex meeting the specifications prescribed by the consuming industry. The RRII has developed an accelerated test to predict the maximum attainable values for MST, viscosity and KOH number of the centrifuged NR latex having VFA under control, with reasonable accuracy within four days of its production.

INTRODUCTION

The Rubber Research Institute of India (RRII) has been undertaking research on primary processing of natural rubber (NR), testing and quality control of latex, prevulcanisation of latex, blends of NR with other elastomers, natural rubber composites and applications of NR. In the primary processing area, the work carried out was aimed at reducing the cost of processing and improving the quality. In testing and quality control of latex, the focus was on development of methods for quick estimation of dry rubber content (DRC) of fresh field latex and to predict the quality of ammonia preserved concentrated latex after storage. Some of the important findings of the studies conducted in these areas are presented in this paper.

PRIMARY PROCESSING OF NR

Use of sulphuric acid as coagulant

Even though many chemicals can be used to separate rubber from latex, weak acids such as formic and acetic acids are commonly used by farmers to prepare sheet rubber. This is because these acids are volatile in nature, easy to handle and the residue has minimum adverse effect on the quality of the processed rubber. However, attempts to use strong non-volatile acids like sulphuric acid were made by earlier researchers (Wiltshire, 1932; Martin and Davey, 1934; Baker and Philpott, 1950; Best and Morell, 1955). Properties of rubber coagulated with sulphuric acid were studied in detail by Neef (1950) and Othman and Lye (1980). All these studies indicated that use of excess sulphuric acid adversely affected the properties of rubber. During 1990's, farmers in India started using sulphuric acid because of the scarcity and relatively high price of formic acid. The RRII conducted detailed studies on various aspects of the use of sulphuric acid for latex coagulation and evolved conditions under which sulphuric acid can be safely used for sheet rubber production (George *et al.*, 1992 and Varghese *et al.*, 1996). Based on the results of the above studies, the Rubber Board of India issued recommendations on the use of sulphuric acid for sheet rubber production. The quantity of acid required (Table 1) and procedure to be followed while using sulphuric acid were also identified based on these studies.

Table 1. **Concentration and quantity of sulphuric acid required for the preparation of 500 gram sheet rubber**

Sheeting schedule	Concentration	Quantity
Same day sheeting	0.5% solution	300 ml
Next day sheeting	0.5% solution	250 ml

The following precautions should be taken while using sulphuric acid

i. For diluting sulphuric acid, it should be added in small quantities to the required quantity of water under stirring. Water should not be added into the concentrated acid.
ii. Fresh latex is to be diluted to 12.5% DRC before adding the acid.
iii. The sheeted coagulum should be

washed thoroughly in running water to remove residual acid.

iv. The sheeting roller and coagulating pans should be washed thoroughly immediately after use to prevent corrosion.

The raw rubber quality parameters and technological properties of the compounds prepared from the sheet rubber made using sulphuric acid as coagulant, were found to be comparable with those of the sheets made using formic acid, when standard procedure was followed.

Use of formic acid-salt mixture as coagulant

While sulphuric acid is a very cheap and effective coagulant for latex, the danger associated with its careless handling and unscientific use for latex coagulation should not be overlooked. There was also a need to make the coagulation process faster, so that the coagulum becomes ready for sheeting within a shorter time. Farmers /tappers use excess formic acid to achieve this objective, incurring additional cost. Hence attempts were made to evolve a coagulant, which is more effective than formic acid, cheaper and devoid of the harmful effects of sulphuric acid. Earlier reports indicated that use of calcium chloride and an anionic surfactant could bring about quick coagulation of latex (John 1971; 1976). Calcium salts are used to cause controlled coagulation of latex compound in the production of dipped latex products (Blackley, 1997). Factors such as reduction in pH, increase in ionic strength of the aqueous phase and removal of the adsorbed soap as insoluble materials, are associated with the destabilization of the colloidal system and coagulation of latex. Hence experiments were conducted with mixtures of salts and formic acid for coagulating latex in the production of sheet rubber. After a series of trials, it was found that a mixture, consisting of sodium chloride, formic acid and calcium chloride in the ratio 1:1.3:2.4 (by weight), dissolved in water, was very effective. A stock solution of the coagulant mixture can be prepared by dissolving the chemicals as given in Table 2.

Table 2. **Stock solution of coagulant mixture**

Chemicals	Quantity (gm)
Sodium chloride	100
Formic acid	132
Calcium chloride	240
Water	As required to make up to 1 lit

From the stock solution 12.5 ml, diluted to 250 ml, will be sufficient to prepare one sheet weighing 500 gm. Quality of the sheets prepared using the coagulant mixture is excellent. Parameters such as drying time, raw rubber properties, storage quality and technological properties of the sheets were comparable to those of sheets prepared using formic acid (Alex *et al.*, 2002). In addition to a slight advantage in cost over formic acid, use of the coagulant mixture is helpful to reduce the maturation time of the coagulum before sheeting and to increase the pH of the serum discharged to the soil / effluent plant. A cost comparison of the different coagulants is given in Table 3.

Table 3. **Cost of coagulants for producing one tonne of sheet (same day sheeting)**

Parameter	Formic acid	Sulphuric acid	Coagulant mixture
Price per kg. (Rs.)	54.00	14.44	17.70
Quantity required (kg.)	5.30	5.53	11.80
Cost for one tonne (Rs.)	286.20	79.85	208.86

Drying of sheet rubber

Cost of drying of sheeted coagulum forms a major share in the processing cost in sheet rubber production. Hence small farmers practise sun drying or partial sun drying followed by smoke drying of the rubber sheets. In this process, a lot of dust and dirt accumulate on the surface of the sheets. Over exposure of the sheets to sunlight leads to development of tackiness on the sheets. All these factors lead to downgrading of the sheets. Studies conducted by different research groups indicated that prolonged exposure of the sheets to sunlight is harmful and it should be avoided (O' Connell,1966; Thomas, 1971). With a view to avoiding exposure to direct sunlight, use of driers based on solar energy for sheet drying was also attempted (Walpita *et al.*, 1984; Nair *et al.*, 1988). But the initial investment of such driers is very high. Later studies on sun-dried sheets indicated that sun drying up to two days followed by smoke drying for two days yielded sheets having equivalent physical properties as compared to sheets dried in a smoke house (Tillekeratne *et al.*, 1995; Jayasuriya *et al.*, 2000). Recent studies conducted at the RRII also indicated that unless exposed to sunlight for prolonged periods, the quality of the sheets are not adversely affected by sun drying (George *et al.*, 2002). Based on these observations, the RRII has developed a system consisting of a smoke house and a trolley, which enabled continuous drying of the sheets in sunlight as well as in the smoke house. In this system the sheets are put on a specially designed trolley. There are five racks in the trolley, four of which can be drawn out in four different directions and the top rack is a fixed one. Each rack can carry 30 sheets and the total capacity is 150 sheets. Sheets on the trolley are dried in sunlight during day time (Figure 1). The racks are drawn in and the trolley pushed in to the smoke house (Figure 2) during night. Drying of sheets is continued inside the smoke house using minimum quantity of firewood. It was observed that 40 to 55 % saving in firewood during the summer season and 15 to 20 % saving during rainy season could be achieved using this drying system. Table 4 provides the data on firewood consumption in one year under full loading of the drier.

Table 4. **Firewood consumption in the new drying system**

Month	Firewood used per kg of dried sheet (kg)
June 1999	0.92
July	0.79
August	0.86*
September	0.99*
October	0.69
November	0.61
December	0.61
January 2000	0.47
February	0.46
March	0.49
April	0.44
May	0.48
June	0.94
July	0.82
Average	0.64

*Drying with firewood only and hence excluded while taking the average.

It takes only three to four days during summer and four to five days during rainy season to get the sheets dried. In addition to the savings in firewood, the new drying system has the following advantages.

i. Minimum handling of the sheets and hence less chance for surface contamination.
ii. Maximum utilization of sunlight since shade from the top layer is avoided.
iii. Faster drying due to better aeration.
iv. Uniform colour due to even drying and smoking.
v. Higher grades for the sheets.
vi. Easy operation, especially in case of occasional unexpected rain.

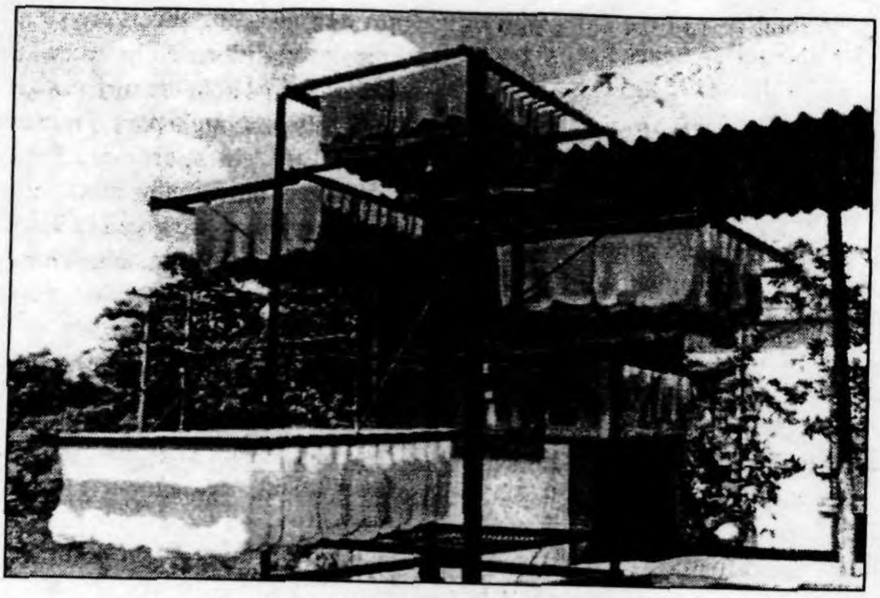

Figure 1. Trolley with sheet in sunlight drying

Figure 2. Trolley inside the smoke house with the racks pushed in

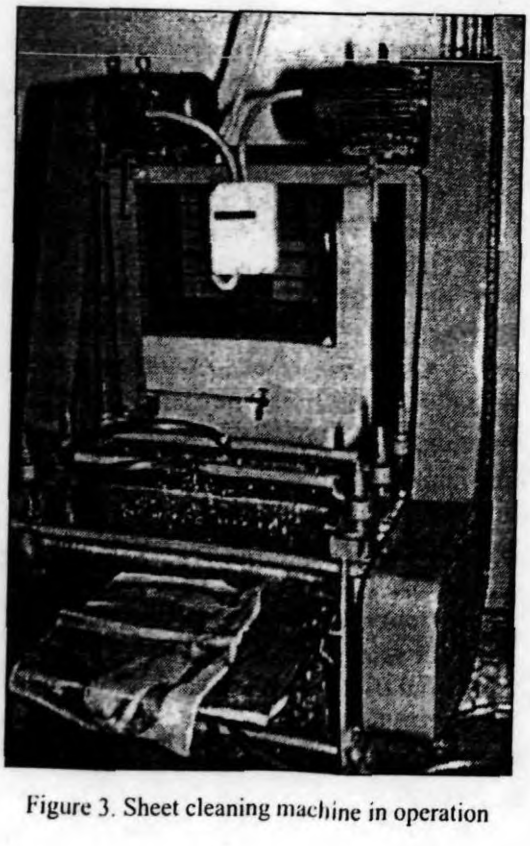

Figure 3. Sheet cleaning machine in operation

vii. The total cost of the smoke house and the trolley, which has a capacity of 150 sheets (75 kg) is Rs.23000/- only.

Reprocessing of sheet rubber

Rubber sheets get affected by fungus, when stored under humid conditions. Careless handling during drying leads to surface contamination of the sheets by dust, dirt and other foreign particles. Presence of such impurities on the surface of the sheets lowers the grade of sheets. By cleaning the contaminants from the surface and re-drying, the quality and grade of such sheets can be improved. Rubber dealers / marketing societies / farmers adopt manual cleaning of such sheets, engaging labourers. The RRII has developed a semiautomatic machine for cleaning surface contaminated sheets. The machine consists of a set of mild steel (MS) feed rolls (having square cut design on the surface) which runs at 90 rpm, a set of brush rolls rotating at 300 rpm and a second set of brush rolls at 360 rpm. The length of each roll is 70cm. The bristles of the brushes are of nylon having 2.5cm length and fixed on a PVC roll, which has a steel shaft through its centre. The rolls are arranged on a MS frame work, which is provided with aluminum feeding and receiving trays. The top rolls have upper and lower movement facility to accommodate sheets of variable thickness and are driven by a 2 H.P. motor. The bottom rolls are in fixed position and are driven by another 2 H.P. motor. Water jets are provided from the top and bottom, in between the three sets of rolls. The sheets are passed through the feed rolls and as they come out, are pulled by the first set of brush rolls and cleaned in a spray of water. The semi-cleaned sheets pass through the second set of brush rolls and are cleaned thoroughly in another spray of water. The sheets are stretched due to the speed difference between successive sets of rolls and both the surfaces get cleaned thoroughly in a single pass. Figures 3 shows the machine in operation. Performance evaluation of the machine was conducted and the data obtained are given in Table 5 & 6.

The design of the machine has been further modified to improve the performance and reduce cost. The modified machine has 54cm length for the rolls; spring loading for the feed rolls, only one 3H.P. 3 phase motor and less space between successive rolls. The price of the new machine is Rs.53000/- only.

Table 5. **Requirements for reprocessing one tonne of sheets**

Parameter	Requirement
Time, min	84.0
Power, kWh	4.16
Water, kl	2.4
Manpower, Nos.	7
Firewood, kg	150
Weight loss, %	1.7

Table 6. **Grades of sheets before and after cleaning**

Grade	Before cleaning	After cleaning
RSS 4, %	0.0	60.5
RSS 5, %	20.2	30.6
Off-grade, %	79.8	8.9

DEVELOPMENTS IN LATEX TESTING

The RRII has been conducting research on various aspects of latex technology, which included development of prevulcanized latex (Claramma and Mathew, 1997), deproteinized latex (George *et al.*, 2001) and radiation grafted latex (Claramma *et al.*, 2000 and George *et al.*, 2002).

Quick estimation of DRC

Knowledge of the DRC of fresh field latex is required for calculating the quantity of various chemicals and water for processing and for effecting payment for the latex. Methods based on hydrometers (Nair and Sivaramakrishnan, 1970), specific heat (Harris *et al.*, 1985), microwave (Khalid *et al.*, 1989) and NMR (Gambhir *et al.*, 1993) have been reported the quick estimation of the DRC of fresh latex. Out of these methods, only the metrolac method is widely practised due to its easiness to perform and simplicity. However, the accuracy of this method is poor and the estimated DRC values vary when different instruments are used. The RRII has developed a quick method to estimate the DRC of fresh field latex. The basic principles involved in this method are given below.

When a water-soluble fatty acid soap is added to latex, the fatty acid anion displaces the protein molecules and gets strongly adsorbed on the rubber particles. When acid is added to the soap treated latex, the adsorbed soap anions react with the acid, depriving the rubber particles of stabilizers. As a consequence of this, latex coagulates immediately. As the DRC of latex increases, the total adsorbed soap increases and the acid required for coagulation also increases. Hence it is possible to estimate the DRC of the latex from a calibration curve drawn with the known values of DRC of latex and the volumes of standard acid required to coagulate it. For estimating the DRC, about 1.0 gram of latex is accurately weighed into a 250ml beaker and 30ml of distilled water and 0.133ml of 15% potassium oleate soap solution are added. Two drops of phenolphthalein are also added and the contents titrated against 0.05N sulphuric acid at the rate of one drop of acid per second. The volume of acid required for complete disappearance of pink colour is noted as V_1. Titration is continued at the same rate till the end point, which is the curdy appearance, just followed by separation of latex into rubber and clear serum. The volume of acid is noted as V_2. The volume of acid required for coagulation, V is calculated as V_2-V_1. Following the same procedure, the volumes of acid required to coagulate different latex samples of known DRC values are found out. A graph with volume of acid V as the X-axis and the actual DRC values on the Y-axis is drown. From the straight line plot obtained, the DRC of the unknown latex sample can be read out, knowing the volume of acid required to coagulate the sample under the conditions specified above. A quadratic equation may also be derived from the data using regression analysis and the value of V substituted in the equation to get the DRC of the unknown sample. The variation from the actual DRC values obtained using the metrolac and the titration methods in a typical trial using fresh field latex are shown in Table 7.

Early prediction of latex quality parameters

Quality parameters such as mechanical

Table 7. **DRC variation between metrolac and titration methods**

Date		Actual DRC (%)	Metrolac 1, DRC (%)		Metrolac 2, DRC (%)		Titration, DRC (%)	
09/10/2002	a.	36.69	33.0	(-3.69)	39.0	(+2.31)	35.75	(-0.94)
	b.	29.15	30.0	(-0.85)	27.0	(-2.15)	29.65	(+0.50)
18/10/2002	a.	38.39	45.0	(+6.61)	39.0	(+0.61)	37.40	(-0.99)
	b.	30.18	36.0	(+5.82)	30.0	(-0.18)	30.15	(-0.03)
21/10/2002	a.	37.84	45.0	(+7.16)	34.5	(-3.34)	38.19	(+0.35)
	b.	34.11	42.0	(+7.89)	36.0	(+1.89)	34.42	(0.31)

Figures in brackets show the difference from the actual DRC values stability time (MST), viscosity, potassium hydroxide number (KOH No.), volatile fatty acid (VFA) number etc. of concentrated natural rubber latex change during storage. Such changes happen at a fast rate during the early days of storage, but continue to occur during transportation / storage of latex. As the customer specifies the quality parameters which the latex should have on its arrival at his site, it is often difficult to meet these quality parameters. The RRII has developed a method, which would predict the quality parameters such as MST, viscosity and KOH number of concentrated NR latex. The method consists of subjecting the freshly centrifuged ammonia preserved latex to accelerated ageing under the conditions specified in Table 8 and then testing the aged latex for these parameters.

Table 8. **Conditions for accelerated ageing of centrifuged latex**

Parameter	Value
Ammonia content, %	1.0
Temperature, °C	50
Time, h	96

The results indicated very good agreement between the accelerated test values and the actual values of the quality parameters after storing the latex for three to six months, provided the VFA is under control. Some of the typical values obtained in the trials conducted are given in Tables 9,10 and 11.

The changes in quality parameters taking place during accelerated ageing and normal storage could be correlated with the quantity and type of higher fatty acid soaps formed in latex under such conditions.

Table 9. **Accelerated and natural MST build up of latex (seconds)**

Latex source	Second day of production	Accelerated condition	Storage period (month) 1	2	3	6
A	90	500	400	605	700	584
B	120	555	650	748	772	600
C	450	824	730	756	978	1025
D	570	885	785	876	1046	600
E	510	640	540	518	479	420
F	750	675	675	670	670	424
G	450	570	540	540	547	554
H	62	525	620	627	600	760

Table 10. **Accelerated and natural viscosity values, (cps)**

Latex source	Second day of production	Accelerated condition	Storage period (month). 1	2	3
A1	144	120	122	104	106
B1	88	84	86	86	84
C1	108	92	108	106	102
D1	224	180	180	178	172
E1	156	124	134	130	116
F1	144	128	130	128	126
G1	192	140	132	128	126
H1	120	110	107	105	100

Table 11. **Accelerated and natural KOH values**

Latex source	Second day of production	Accelerated condition	Storage period (month) 1	2	3
A2	0.39	0.46	0.47	0.50	0.55
B2	0.40	0.51	0.52	0.62	0.63
C2	0.29	0.44	0.44	0.54	0.55
D2	0.62	0.67	0.70	0.75	0.79
E2	0.42	0.45	0.48	0.51	0.54
F2	0.57	0.59	0.62	0.68	0.69
G2	0.62	0.74	0.76	0.83	0.84
H2	0.55	0.57	0.60	0.62	0.63

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