

## ***NR Latex Vulcanisation – Pre-vulcanisation and Post-vulcanisation of Dipped NR Latex Films***

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*The tensile properties results of NR dipped latex films prepared by prevulcanisation process and post-vulcanisation imply that the standing period during the latex stage dominates the extent of crosslinking in the latex films. Further crosslinking does not seem to occur in dry latex films as reflected by its modulus values. The results support the idea that most crosslinking in NR latex occurs during the latex stage. Leaching improves the tensile strength and modulus when the latex films were conditioned under low humidity. The presence of water in the dry latex films during leaching may indicate water as a medium for further crosslinking to occur, but the reduction of modulus of the films when conditioned under wet humidity contradicts this idea. Mooney-Rivlin plots of prevulcanised and post-vulcanised latex films prepared from a similar latex mix further confirm that most of the crosslinking occurs during the latex stage. It is concluded from the chemical probe study that the crosslinks in sulphur-vulcanised dipped NR latex films are predominantly polysulphidics.*

The current view of sulphur vulcanisation in NR latex is that the mechanism of sulphur vulcanisation within the rubber particles in the latex is similar to that of bulk rubber<sup>1</sup>. The widely accepted theory on sulphur vulcanisation in latex has been summarised by Loh<sup>2</sup> and Porter<sup>3</sup>. The vulcanisation mechanism basically involves three main steps. First, the vulcanisation ingredients together with the natural activators form sulphurating complexes in the latex. The second step is the assimilation of these complexes into the rubber particles and thirdly, the rubber hydrocarbon reacts with the sulphurating complexes forming a network of crosslinks in the rubber particles. The time needed for these reactions to occur depends on the types and levels of chemical additives that

are added to the latex and the temperature in which these reactions are taking place.

In latex vulcanisation, the network structure within each rubber particle would depend on the extent of vulcanisation that occurs in the particle. A highly vulcanised rubber particle will consist of a denser network of crosslinks compared to a slightly vulcanised rubber particle. The adhering of these vulcanised rubber particles when the latex serum phase is removed, forms the coherent NR latex film. The structural characteristics of the vulcanised latex film is governed by the concentration of the network of crosslinks in the vulcanised latex films and will be reflected in the film's physical properties.

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It is believed that during the vulcanisation of NR latex, the latex serum together with its natural non-rubber content provides a medium in which some of the vulcanisation reactions may occur. The natural non-rubbers in the latex serum are often regarded as secondary activators for vulcanisation reactions. Vulcanisation of NR latex is carried out either during the latex stage [pre-vulcanisation (PV)] or after the films have been formed [post-vulcanisation (PO)].

In the case of PO, the latex is mixed with its vulcanising ingredients and is normally left under ambient conditions for some time before the films are prepared. Films prepared from the latex mixture are then heated at elevated temperatures e.g. 100°C for 20 min and the heating period of these films is sometimes referred to as the vulcanisation period. In contrast to PO, PV is a process whereby the latex together with its vulcanising ingredients is heated at a temperature of 70°C or below. The vulcanisation process depends on the type and amount of accelerators and vulcanising agent and the period and temperature in which the latex mixture is heated. Latex films obtained from the PV mixture do not need further heating to produce vulcanised films.

It is often thought that in post-vulcanisation, most of the crosslinking reactions occur in the dry state, that is when the latex films are heated. However, it is possible that most of the crosslinking reactions can occur during the maturation period, that is if the latex serum phase can activate the reactions under ambient temperature. If this were true, then post-vulcanisation would actually be a drying process of the vulcanised latex as opposed to a vulcanisation process. Wong and Loo<sup>4</sup> have investigated the room temperature sulphur vulcanisation of NR latex. They have demonstrated that the tensile strength of the latex films decreased with storage time. The decrease in the strength properties was

explained by the poor film forming properties caused by the increased crosslinking in the latex particles.

In the present study, the physical properties of dipped latex films prepared from room temperature and heated pre-vulcanised latex mixes are discussed. Similar properties due to post-vulcanised latex films in which the pre-vulcanisation period was kept to a minimum are also discussed. It would be interesting to ascertain whether vulcanisation can proceed in dry latex films when the serum phase is absent. Thus, the role of the natural non-rubbers in the latex serum as activators for the sulphur vulcanisation under ambient condition can be accounted for.

## EXPERIMENTAL

In the ensuing experiments, dipped NR latex films were prepared from a latex mix after the mix was left standing under ambient condition for one, seven, 27 and 56 days. The physical properties of these films were determined immediately after the dipped film had been air-dried. The idea was to allow the vulcanising ingredients to interact during the standing period and produce the sulphurating complexes that will subsequently initiate crosslinking of the rubber hydrocarbon in the latex. Another set of dipped films were prepared from the same mix but were kept in the dry state for a specific amount of time as described below. The reason was to let the complexes that arise from the interaction of vulcanising ingredients during the latex stage continue with the crosslinking process in the dry state under ambient conditions. The formulations used for preparing the pre-vulcanised NR latex mixes are shown in Table 1.

Commercial high ammonia NR latex concentrate was used and the chemicals added

TABLE 1. FORMULATION FOR NR LATEX VULCANISATION MIX.

Formulation	Dry weight (p.h.r.)
60% NR latex	100
20% Potassium laurate	0.3
50% Sulphur	0.3
50% ZDBC	0.3
50% Zinc oxide	0.25

were either in the form of solutions or fine dispersions. Distilled water is the diluent used to prepare these chemicals. After adding the chemicals, the mixes were stirred for 30 min and left at room temperature for 24 h. The latex mixes were then transferred into storage bottles. Dipped latex films were prepared from the mixes at different periods throughout the storage period. The storage bottles were shaken occasionally to ensure proper mixing of the vulcanising ingredients during the storage period. At various periods, the latex mixes were stirred and samples were drawn for dipped film preparation. The period in which the latex films were kept is summarised in Table 2.

TABLE 2. PERIOD IN WHICH THE LATEX FILMS WERE KEPT PRIOR TO PHYSICAL TESTING

Films	Period of latex storage (days)	Period dipped films kept before testing (days)
Film I	1	62
Film II	7	57
Film III	27	34
Film IV	56	8

TABLE 3. FORMULATION FOR PREPARING DIPPED LATEX FILMS FOR PROBE TREATMENT

Formulation	Dry weight (p.h.r.)	
	Mix 1	Mix 2
60% NR latex	100	100
20% Potassium laurate	0.3	0.3
50% Sulphur	1.0	0.3
50% ZDBC	1.0	0.3
50% Zinc oxide	0.25	0.25

In another set of experiments, NR latex mixes according to the formulation in Table 3 were prepared. Each mix was divided into 2 portions. One portion was used for prevulcanisation and the other was used to prepare post-vulcanised latex films.

The prevulcanised portion of *Mix 1* was vulcanised for 3 h at 60°C, whereas portions of *Mix 2*, owing to its low content of sulphur and ZDBC, needed a longer vulcanisation time of 6 h under the same vulcanisation temperature. The post-vulcanised latex films were prepared from portions of *Mix 1* and 2, but the latex mixes were not heated. Vulcanisation during the latex stage was kept to a minimum in preparing the post-vulcanised latex films by avoiding any heat during mixing of the chemical dispersions into the latex. The mixes for the post-vulcanised latex films were stirred for 30 min before being sieved into another container. The sieved latex mixes were placed in a vacuum chamber for another 30 min, to allow bubbles to rise to the surface. These bubbles were scooped off and dipped films were prepared immediately. The films were then dried for 1 h at 70°C in an air oven and then vulcanised at 100°C. In order to vary the level of crosslinking in the latex films, vulcanisation periods of 10, 30 and 50 min were chosen for each set of films.

### Preparation of Dipped Film

The NR latex films were prepared by dipping calcium nitrate coated glass plates into the latex mix for 135 s to obtain films approximately 0.5 mm thickness. In order to achieve similar thickness among the latex films, the glass plates were coated by approximately similar amount of calcium nitrate. This was done by controlling the immersion, dwelling and withdrawing of the glass plate into a solution of 20% calcium nitrate in methylated spirit. The dipped films were dried at room temperature for 24 h. After drying, the films were dusted with talc and stripped from the glass plates. In the experiment, the calcium nitrate and talc taken into the latex films were considered as the non-rubber materials together with the natural non-rubbers from the NR latex mixes. These films were kept away from any light source under ambient condition, prior to determining their physical properties.

### Leaching of Latex Films

Leached films were prepared by immersing each film in about 1 litre of distilled water for 24 h at 25°C. After leaching; the latex films were air-dried. Dumb-bell tensile test pieces were cut from the leached and unleached latex films for testing the physical properties.

### Conditioning and Testing of Latex Films

The test pieces were conditioned in three different humidity conditions before being tested. For low humidity conditions, the test pieces were stored over anhydrous phosphorus pentoxide. The relative humidities were recorded by the 'Thermo-hygro' hygrometer. Low humidity conditions were recorded as less than 5% RH. A beaker of distilled water was placed in another desiccator for the high humidity condition (> 95% RH). The test

samples were conditioned either under one of these conditions or under ambient conditions for 48 h at 25°C. After these conditioning periods, each test sample was tested immediately after removal from the desiccator.

The tensile strength, modulus and elongation at break were determined according to *BS 903: A2* and *ISO 37*. Relaxed modulus (MR 100) value is the stress needed to maintain a latex strip at 100% extension after one min. The elastic constant of the rubber was determined by equilibrium stress-strain measurement on a 14 mm length by 2 mm width test piece using a Greensmith's apparatus<sup>5</sup>. The elastic constants were calculated using the Mooney-Rivlin equation<sup>6</sup> and corrected to 25°C.

#### MOONEY-RIVLIN RELATIONSHIP

According to the Mooney-Rivlin relation (*Equation 1*), for homogeneous NR gum vulcanisates at low and moderate deformations, a plot of  $F/[2A_0(\lambda - \lambda^{-2})]^{-1}$  against the  $\lambda^{-1}$ , where  $F$  is the extension force required to extend a piece of rubber of cross-section area  $A_0$  to an extension ratio  $\lambda$ , gives a straight line:

$$F = 2A_0(\lambda - \lambda^{-2})(C_1 + \Gamma^{-1} C_2) \quad \dots 1$$

The y-intercept  $C_1$  is related to the physically effective crosslink density ( $\eta_{\text{phys}}$ ) by  $C_1 = \rho RT \eta_{\text{phys}}$ .

#### CHEMICAL PROBE TREATMENT

The chemical probe technique<sup>7</sup> was carried out to selectively destroy the polysulphidic crosslinks with a solution of propane-thiol with piperidine in n-heptane. The remaining crosslinks in the film after the polysulphidic crosslinks are destroyed will be primarily mono- and disulphidic crosslinks. The difference between the crosslink densities determined before and after

the probe treatment will be an estimate of the quantity of polysulphidic crosslinks destroyed by the probe treatment. Poly- and di-sulphidic crosslinks can be cleaved with hexane-thiol in piperidine, leaving the mono-sulphidic bonds intact.

The latex films were extracted in a Soxhlet apparatus for 48 h under nitrogen with 150 ml of an azeotropic mixture (704 ml acetone, 582 ml chloroform and 548 ml methanol). All reagents used in the sulphur probe treatments were analytical grades. After extraction, the films were kept under vacuum, away from any light source. Test pieces (3 mm × 120 mm) were cut and four test pieces (duplicates of each film) were placed in a 300 ml horizontal tube fitted with an inlet and outlet for nitrogen flows and also an inlet and outlet for adding and draining of reagents. The tube was purged with nitrogen and 100 ml of degassed n-heptane was added and the tube was left under a slow flow of nitrogen overnight (16 h). A solution of piperidine [1.2 Molarity (M)] and propane-2-thiol (1.2 M) in n-heptane was prepared and 50 ml of the solution was poured into the tube. The flow of nitrogen was stopped and nitrogen's inlet and outlet were shut. The test pieces were placed in the tube and left for 2 h at room temperature. After this period, the nitrogen was allowed to flow into the tube and the reagent was drained off and replaced by 100 ml of petrol (60°C to 80°C boiling point range). After 1 h, with occasional agitation, the petrol was drained off and replaced with fresh petrol. The washing procedure was repeated at least 3 times and after which the test pieces were taken out and dried to constant weight under vacuum. The elastic constant of the propane-2-thiol in piperidine treated test pieces were determined in a similar manner as the untreated samples. The chemical crosslink density due to the polysulphidic links can be determined from the difference in crosslink

density before and after the probe treatment. The crosslink density after the treatment will estimate the mono- and di-sulphidic bonds in the latex films.

In another experiment four test pieces (duplicates for each film) were placed in the longer arm of a h-shaped tube. 60 ml of hexane-1-thiol in piperidine (1 molar solution) was carefully run down the other tube, ensuring none came in contact with the latex test pieces. The solution was degassed three times and the apparatus was sealed under vacuum. The solution was poured into the arm that contained the test pieces. The apparatus was turned to a horizontal position so that the arm containing the test samples and solution was lower than the other arm. The h-shaped tube was left in that position in a 25°C water bath for 48 h. After this period, the tube was cut open and the content was poured into an evaporating dish. Due to the highly swollen and delicate nature of the test pieces, handling of the test pieces was extremely difficult. Attempts to recover the test pieces with subsequent washing with petroleum and acetone mixture were not successful. It was found that the test pieces disintegrated during these attempts and the dried test pieces obtained were not suitable for the stress-strain measurement.

## RESULT AND DISCUSSION

### Tensile Properties of NR Latex Films

Tensile strength and modulus values of the dipped NR latex films prepared from latex mix (*Table 1*) after various storage periods are given in *Table 4*.

The results show that the tensile strength and the modulus at 300% increased after the latex mixes were prevulcanised under ambient conditions. The modulus values can be used as

TABLE 4. TENSILE PROPERTIES OF NR LATEX FILMS VULCANISED UNDER AMBIENT TEMPERATURE

Properties	Film I	Film II	Film III	Film IV
Ambient condition	UL L	UL L	UL L	UL L
Tensile strength	14.6	25.3	26.0	23.4
Elongation at break (%)	900 900	900 950	875 850	850 850
Modulus at 300%	0.54	0.95	1.05	1.06
MR 100 (MPa)	0.39	0.49	0.51	0.51
High humidity	UL L	UL L	UL L	UL L
Tensile strength	10.4	18.4	18.3	16.7
Elongation at break (%)	900 950	950 950	900 900	900 900
Modulus at 300%	0.51	0.54	0.61	0.74
MR 100 (MPa)	0.33	0.37	0.41	0.38
Low humidity	UL L	UL L	UL L	UL L
Tensile strength	17.4	30.7	30.2	30.4
Elongation at break (%)	950 925	900 900	850 850	850 850
Modulus at 300%	0.52	1.12	1.40	1.40
MR 100 (MPa)	0.46	0.61	0.66	0.65

Value is the median value of 5 test pieces: L = leached; UL = unleached

an estimate for the extent of the apparent crosslinking in the latex films, as these values are known to increase with the increase in apparent crosslink density. It appears from the modulus values that the apparent physical crosslink densities of dipped NR latex films, prepared from the NR latex mix that was left standing longer under ambient temperature (*Films II, III and IV*) are higher values than *Film I*, which had been prepared from the same latex mix, but after one day of standing. Although *Film I* was left longer in the dry state compared to the other latex films, there was still no improvement in its tensile strength and modulus. The modulus results are further confirmed by the relaxed modulus results. The MR 100 values increased significantly after 7 days PV and these values appear to be similar afterwards. The tensile strength of

dipped films prepared after 7 days of prevulcanisation improved significantly and then tended to level off. The tensile strength should increase to a maximum as the crosslink density increases and then decrease as the network chains in the crosslinked network become denser and shorter. The results imply that the presence of water together with non-rubber material during the prevulcanisation stage is an important factor contributing to better tensile strength.

Data compiled by Porter<sup>3</sup> showed that the maximum strength occurred at around a MR 100 value of 0.5 MPa for post-vulcanised NR cast latex films and dry NR vulcanisates. The data indicate that leaching appears to improve the tensile properties, however, the mechanism by which this happens is not fully understood.

To investigate the role of water in providing a medium for vulcanisation to take place, the latex films were leached for 24 h. It is hoped that during the leaching period, in the presence of water, any vulcanising complexes left in the dried films can initiate further crosslinking, thus increasing its modulus. The tensile results of test samples conditioned under low and ambient humidity agree with the observation that leaching increases the tensile strength and modulus. This could imply that the leaching water is a medium for further vulcanisation to take place, but the reduction in modulus of sample conditioned under high humidity contradict this idea.

The softening of the NR latex films as indicated by the decrease in modulus is clear in latex films that were conditioned under high humidity. In such conditions, the 300% modulus of *Films I* and *II* were reduced significantly compared to the results after conditioning under low humidity. The significance of the effects due to leaching and humidity on the physical properties of NR latex films have been discussed elsewhere<sup>8</sup>. This behaviour is thought to be due to the presence of water-soluble non-rubbers in the films. When NR latex film is formed, domains of non-rubbers are formed in-between the rubber particle. These domains can act as reinforcing fillers in the NR latex films, however it has been suggested that the domains may hinder rubber particle integration. The effect of leaching on the modulus of prevulcanised latex films is critically dependent on the humidity under which the films are stored prior to testing. At low humidity leached films tend to give a lower modulus than unleached counterparts but at moderate and high humidities this effect is reversed. These effects can be adequately explained in terms of a stiffening effect of natural non-rubbers under dry condition and a softening effect of the same non-rubbers which absorb moisture under humid conditions.

The vulcanising chemicals that were used to compound the latex have low solubility in the latex serum. When added to the latex, the accelerators and the activators will take some time to form active vulcanising complexes that could assimilate into the rubber particles in NR latex. The presence of natural activators and the serum phase during the latex stage assists in forming these rubber soluble complexes. These complexes will then react together with the sulphur donors to form crosslinks with rubber hydrocarbon. Results from this experiment indicate that most of the sulphurating complexes are formed and crosslinking mainly occur during the prevulcanisation period. Although elevated temperature would be expected to accelerate these reactions, results from the experiment agrees with the observation of Wong and Loo<sup>4</sup> that latex vulcanisation is possible under ambient temperature.

Further crosslinking does not seem to occur in dry NR latex films as reflected by the modulus values seen in the latex films. The crosslinking reactions are very slow, if any, and appear to be absent in the latex films that were left to post-vulcanise at room temperature for up to 62 days (*Film I*). The results support the hypothesis that under ambient conditions, most of the vulcanisation occurs in NR latex. Dried films that were left under ambient conditions did not show any improvement in physical properties.

The results from the previous experiment indicated that the standing period in the latex stage dominates the extent of crosslinking in latex vulcanisation. To further clarify the aspects of vulcanisation in NR latex, the tensile properties of prevulcanised and post-vulcanised latex films were compared. The tensile properties of prevulcanised and post-vulcanised latex films from formulation in *Table 3* are shown in *Table 5*.

TABLE 5. TENSILE PROPERTIES OF PREVULCANISED AND POST-VULCANISED NR LATEX FILMS

Tensile properties	Mix I			Mix 2		
Prevulcanised	3 h at 60°C			6 h at 60°C		
Tensile strength (MPa)	25.6			26.6		
Elongation at break (%)	880			860		
Modulus at 300% (MPa)	1.68			1.42		
MR 100 (MPa)	0.78			0.60		
Post-vulcanised						
Vulcanisation time	10 min	30 min	50 min	10 min	30 min	50 min
Tensile strength (MPa)	24.5	24.8	28.0	16.7	16.3	16.9
Elongation at break (%)	840	920	1000	990	980	970
Modulus at 300% (MPa)	1.05	1.10	1.11	0.74	0.75	0.77
MR 100 (MPa)	0.48	0.51	0.48	0.34	0.36	0.39

As expected, the lowest modulus is from *Mix 2* with the least amount of sulphur. The heating of the dried latex films at 100°C does not appear to improve the tensile properties of the films. The increase of the MR 100 values upon heating seen in films from *Mix 2* is too small to be considered significant. There is a possibility that some of the chemical additives settled down after the chemical mixing procedure, thus preventing more chemicals to be deposited in the dried latex films. Therefore, the experiment was repeated and in order to prevent the chemical dispersions from settling to the bottom, the mixes were agitated slowly during the dipping of the films. Similar modulus results were observed in these repeated experiments, thus confirming the validity of the earlier observations.

Although, the post-vulcanised and prevulcanised films are of similar chemical ingredient content, the moduli of latex films prepared from prevulcanised latex are always higher than post-vulcanised latex films. The heating while the rubber particles are in the

serum phase seems to enhance the vulcanisation reaction. The sulphurating complexes from the latex mixes do not appear to continue their crosslinking reactions during heating in the dry state. The period for which the vulcanising ingredients were left to react in the latex stage during the preparation of the post-vulcanised films may have been too short for sulphurating complexes to be formed. Moreover, less, if any, were transferred to the films to initiate further crosslinks in the dry state.

In dry rubber vulcanisation, it is well known that a low ratio of sulphur to accelerator gives vulcanisates consisting of mainly mono-sulphidic crosslinks and the opposite composition would yield predominantly poly-sulphidic crosslinks in the vulcanisate<sup>9</sup>. A chemical probe method to detect the type of crosslinks was attempted on the prevulcanised and post-vulcanised latex films. The aim was to investigate crosslinking activities that take place in terms of crosslinking complexes formed by the interaction of the vulcanising ingredients in the prevulcanised and post-vulcanised latex films.



TABLE 6. PHYSICAL CROSSLINK DENSITY OF VULCANISED LATEX FILMS BEFORE AND AFTER TREATMENT WITH PROPANE-2-THIOL WITH PIPERIDINE IN N-HEPTANE

Mix	Before treatment $C_1$ (MPa)	After treatment $C_1$ (Mpa)
Prevulcanised		
Mix 1	0.16	0.04
Mix 2	0.10	0.05
Post-vulcanised		
Mix 1 (10 mins)	0.08	0.04
Mix 1 (50 mins)	0.03	0.03
Mix 2 (10 mins)	0.08	0.05
Mix 2 (50 mins)	0.05	0.04

The physical crosslink densities of the latex films before and after being treated with propane-thiol mixture were estimated from the y-intercept ( $C_1$ ) of the Mooney-Rivlin plots and these values are given in *Table 6*.

After the probe treatments, the physical crosslink densities estimated from the  $C_1$  values reduced appreciably. The effect of reducing the crosslinks density is clearly seen in the Mooney-Rivlin plots. The plots for prevulcanised films are shown in *Figure 1* and the plots for the post-vulcanised films are shown in *Figure 2* and *3*.

Films from *Mix 1* which have the highest content of sulphur and accelerator are expected to have the highest physical crosslink density and this behaviour is reflected in the Mooney-Rivlin plots of the untreated films. When treated, the y-intercept value of film of *Mix 1* reduced to a relatively similar value to that of the treated films from the other mixes. The decrease in the y-intercept values is due to the destruction of the network structure in the latex films by the propane-thiol mixture and this mixture is known selectively to eliminate polysulphidic crosslinks. Therefore, it can be

concluded that the network structure in these films is mainly polysulphidic in nature.

Similar trends are also observed for Mooney-Rivlin plots of post-vulcanised films (*Figures 2* and *3*). The post-vulcanised film from *Mix 1* shows lower y-intercept value than that of the prevulcanised film of the same mix. This is consistent with the tensile results and implies that a higher crosslink density was achieved during vulcanisation in the latex stage. The Mooney-Rivlin plots of the propane-thiol treated post-vulcanised latex films appear to behave in a similar manner as that of the treated prevulcanised films.

#### CONCLUSION

Results from this study support the hypothesis that the presence of the latex serum together with its non-rubbers can be an activating medium for the vulcanising ingredients to interact and form sulphurating agents at ambient temperature. The crosslinking in the rubber is believed to be due to the reactions of these sulphurating agents with the rubber hydrocarbon. It was demonstrated in the study that interaction of NR with its vulcanising

ingredients during prevulcanisation is a major factor in the course of NR latex vulcanisation. The superiority in physical properties of prevulcanised NR latex films to post-vulcanised NR latex films agrees well with the above hypothesis. The polysulphidic crosslinks that dominate the network structure of NR latex are the type of crosslinks normally favoured by mild temperature vulcanisation. The vulcanisation in NR latex occurs quite readily during the latex stage and it seems impossible to prepare NR latex films in which the vulcanisation can be carried out entirely during the dry state.

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