

CHAPTER THREE



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METHODOLOGY

Centrifuged concentrated low ammoniated latex was brought with given specifications. The specifications were further confirmed by tests done at the University of Moratuwa. The concentrated latex was prevulcanized by using a standard formula. The prevulcanized latex films were prepared by using glass plates and a shim. After drying the films at room temperature for 72 hours the films were leached for 2 minutes in running tap water. Then they were again air dried for 72 hours before chlorination. The films were chlorinated at different levels by standardized bleaching powder of Sodium Hypochlorite and 1% m/m HCl solution. Then films were again leached in running tap water for 2 minutes before air drying for 3 days. Tensile strength was found by using an electronic tensile testing machine for all the samples and their relevant data were recorded. Surface friction test was carried out with all the samples. A glass plate and a match box were used for this test. Friction coefficient was checked for all the films.

After analyzing the physical properties of tensile strength and surface friction, it was observed that 1150 ppm Chlorine concentration with 12 minutes immersion time gave the optimum result. Hence samples were selected which had undergone chlorination at 1150 ppm concentration at different immersion times and different concentrations with 12 minutes immersion time. The protein content was checked for all these selected samples by using BCA enhanced protocol method at the RRI Sri Lanka. The topology of the selected samples were observed by using the optical microscope. The formation of dark domains and surface cracks were identified and their pictures were taken.

3.1 SPECIFICATION TEST

Low ammoniated Tetra Methyl Thiurum Disulphide/Zinc Oxide preserved concentrated latex was obtained from S&G factory at Horana. The specifications are given in annex. 1.

In order to verify the specifications the required tests were done at the University of Moratuwa. The results are given in annex. 11.

3.2 PREPERATION OF PREVULCANIZED LATEX FILMS

3.2.1 COMPOUND FORMULATION

The prevulcanised latex compound formulation shown in table 3.1 was obtained from the literature (Blackley 1997).

Table 3.1 Prevulcanised latex formulation

	Dry formulation Phr	Wet formulation Phr
60 % NR Latex LA - TZ	100	167
20 % Potassium Laurate	0.26	1.30
10 % Potassium Hydroxide	0.25	2.50
50 % Sulphur	1.00	2.00
50 % Z D E C	2.00	4.00
50 % ZnO	0.20	0.40

Potassium Laurate was used to stabilize the latex. It was prepared by allowing Lauric acid to react with KOH at 75 °C and was available in the form of an emulsion. 10% Potassium Hydroxide was prepared and the dispersions of 50% S, 50% ZDEC, 50% ZnO were separately prepared in slurry form. Their formulations are given in annex 3. The prevulcanized latex was prepared at the RRI Sri Lanka.

3.2.2 PREVULCANIZATION OF LATEX

First the Potassium Laurate emulsion was added to NR latex followed by Potassium Hydroxide dispersions of ZnO, ZDEC and finally Sulphur. Then the mix was heated in a water bath at 60⁰C for 3 hours. The quality of prevulcanised latex was inspected by the chloroform coagulation test from time to time till the required level. Small dry crumbs were obtained when it came to the needed prevulcanised stage. Then the latex was allowed to mature for 40 hours.

3.2.3 PREPARATION OF FILMS

The required number of latex films was found to be thirty six. Hence to lay the latex films 36 glass plates were cut according to the following dimensions

Length	- 25 cm
Width	- 25 cm
Thickness	- 0.30 cm

For the laying of latex films an applicator bar (shim) was made by fixing very thin Aluminium strips on both ends of a glass rod. A strip had an average thickness of 0.20 mm.

For the preparation of films a required volume of latex was strained through a mesh sieve and allowed to fall on to a glass plate. Then the shim was pushed along the glass plate leaving an uniform wet latex film. It was the first time that this type of efficient system was adopted to lay the latex films. This process was done for all the thirty six glass plates. Then the plates were kept on a flat uniform surface and allowed to dry in air for 72 hours. Thus latex films with a very uniform thickness were obtained in a very short time period. There were other methods where latex films were obtained without any uniformity in thickness and so much time consuming. Due to those reasons the other methods were not

used. The dried films were leached in running tap water for 2 minutes & then they were again air dried for 72 hours.

3.3 CHLORINATION OF LATEX FILMS

For the chlorination procedure a glass tank was made with following dimensions.

Length - 35 cm

Width - 35 cm

Height - 35 cm

First a study was done about the chlorination of water at the National Water Supply and Drainage Board, Ratmalana. The selected chlorinating agent was standardized bleaching powder consisting of Sodium Hypochlorite. The tests which were done for the bleaching powder by the Chemical department of Water Supply Board revealed that 100 g of this powder dissolved in 100 g of water had 67.7 g of available chlorine content. The Chlorine was found to be available in the form of Cl_2 , HOCl , OCl^- , Cl^- .

The chlorination process was done at the Chemical Engineering laboratory of the Moratuwa University. First a 1% m/m diluted HCl solution was prepared by using a standard HCl acid which contained 0.37% of HCl. Hence the standard HCl 2 g was diluted with 72 g of distilled water to obtain 1% m/m diluted HCl acid of 74 g. After the calculations it was found that 2.056 g of 1% m/m HCl acid had 200 ppm of Chlorine content. This weight of 1% m/m diluted HCl acid was taken as a constant weight in each of the chlorination processes.

For the preparation of 1150 ppm Chlorine content the balance 950 ppm was given by dissolving 2.8064 g of NaOCl bleaching powder in 2 liters of water. Thus 2 liters of NaOCl solution was prepared and it was allowed to react with 1% m/m diluted 74g HCl solution. Then the total solution

was transferred to the glass tank and a glass plate with the latex film was kept in the Chlorinated solution for an immersion time of 24 minutes. During this period the tank was kept tightly closed by a larger glass plate. After the required immersion time the glass plate was removed and the latex film was leached with running tap water for 2 minutes. Finally No.1 was marked with Tipex on the other side of the glass plate for reference and allowed to dry in air for more than 72 hours. Each latex film was subjected to Chlorination in the same way for immersion times of 20, 16, 12, 8 and 4 minutes. Then they were marked No.2, 3, 4, 5 and 6, respectively. For each immersion time a fresh Chlorination system was used at 1150 ppm Chlorine content.

During the preparation of Chlorine contents of 1300 ppm, 1400 ppm, 1500 ppm, 1700 ppm and 1000 ppm solutions a Chlorine content of 200 ppm was given by 2.056 g of 1% m/m HCl solution and the balance Chlorine contents were given by weights of 3.250 g, 3.545 g, 3.840 g, 4.431 g and 2.363 g of NaOCl bleaching powder respectively. These weights were dissolved in 2 liters of water and allowed to react with the prepared diluted HCl acid to obtain the required Chlorine concentrations. For the chlorination six different immersion times were selected. Altogether chlorination was done at various levels for 36 samples. After leaching the chlorinated films they were given reference numbers.

3.4 MEASURING OF TENSILE STRENGTH

For the tensile strength, the latex films had to be cut according to the standard dumb bell shape using a cutter. It was a difficult thing because the film stuck to the cutter blades. Finally, overcame the problem by placing a portion of the latex film between two pieces of smooth paper and cutting it with the dumb bell cutter. Since the results were considered in duplicate, two test pieces were prepared from each latex film. Their thicknesses and widths were checked at three different points by the Wallace Thickness gauge and ruler, respectively. Then the tensile

strength was found using the Hounsfield H10KT electronic tensile testing machine for each test piece.

It was a very tedious process because the test pieces slipped at the grips. Hence several wasted latex films had to be kept between the test piece end and the grips. Maximum force was noted down for each sample. The tensile strength was found for 36 Chlorinated and raw samples in duplicate for better accuracy.

3.5 MEASURING OF SURFACE FRICTION

For the determination of surface friction, a simple procedure was adopted. A glass plate of length and width of 91.52 cm x 15 cm was used with a small match box which had a smooth surface. Latex films were cut into sizes of length and width of 20 cm x 4 cm. Each sample was kept between a folded smooth paper for better surface protection. A sample was taken and its inner surface was pasted along the glass plate. During this process air bubbles entrap between the latex film and the glass plate. Hence it was done very carefully. One end of the glass plate was kept at a fixed point and the small box was kept on its smooth surface along the rough surface of the latex film.

Gradually very slowly and carefully the free end of the glass plate was lifted until the box slips along the latex film. Then the vertical height of the other end of the glass plate was taken. This was a very time consuming process. Each sample took more than 20 minutes for a result and the readings were taken in triplicate for better accuracy. This surface friction test was done for all the chlorinated and raw latex films.

3.6 ANALYSIS OF SURFACE TOPOLOGY

For the observation of surface cracks an OPTIPHOT-POL Microscope was used. This facility was obtained from the laboratory of the Material

engineering Department of University of Moratuwa. Each selected sample was cut very carefully into length and width of 6 cm x 4 cm without stretching to avoid surface damages.

The objective magnification and photo eye piece were adjusted at 5X. Hence the overall magnification on the negative was 25X. The photographs were taken at the sharpest stage by adjustments for each selected latex film. For each of the relevant samples, the frame number of the Optical Microscope was noted. The photos were obtained after washing the negatives from a studio.

3.7 DETERMINATION OF PROTEIN CONTENT

The protein content tests were done at the laboratory of the Department of Polymer Chemistry, Rubber Research Institute, Ratmalana. The BCA enhanced protocol method was selected since it is a standard method at the RRI Sri Lanka. This was a very sensitive test. Hence to avoid protein contamination a pair of new Nitrile gloves and a clean scissor were used during the cutting of latex films. First all the selected samples were cut into length and width of 1 cm x 1 cm. For cleaning of the glass ware, Pynol liquid detergent was used because other cleaning agents interfere with the BCA enhanced protocol method. All the glass tubes and the beakers were twice washed thoroughly with Pynol and water. Then they were filled with boiling water and kept for 10 minutes. Thereafter each of the above glass ware and two 10 ml pipettes were washed with acetone and allowed to dry for 10 minutes in normal air. Again they were washed with acetone and repeated the same. Finally, they were twice washed with fresh distilled water and dried in an oven at 130⁰C for 3 hours to complete the washing procedure. After that they were allowed to cool at room temperature.

About 1g of small pieces which were cut from a latex film was weighed by an electronic weighing balance and put into a beaker. The



weight of latex film was noted and distilled water of ten times of that weight was taken into a pipette and transferred to a beaker with the pieces of latex film. The beaker was kept tightly closed by a polyethylene film and a rubber strip. A price tag roller was used for marking purpose. This process was adopted to other selected latex films starting from the raw to Chlorinated samples. A batch of 4 beakers were prepared and placed tightly fitted on the shaker for 2 hours to remove the water soluble proteins from the latex films. During the shaking time reagents were prepared. The arrangement was to take 2 readings each from the 4 samples and the control sample. Hence for each of the 10 tubes, 20 ml from reagent A and 0.4 ml from reagent B was used. Micropipettes with disposable syringes were used to measure these volumes. The reagents A and B were mixed together in a small beaker and 2 ml of reagent mix was transferred to each of the 10 tubes by using the micropipette. Then 100 ul of distilled water was added onto the 2 control samples. According to the reference numbers of the latex films all the tubes were labeled.



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After completion of 2 hours shaking at room temperature, the beakers were taken out and transferred 100 ul from each to their relevant tubes in duplicate by the micropipette. A fresh syringe was used throughout the entire process to measure the volumes from the micropipette. Each tube was tightly covered with a thin Aluminium foil and they were kept in the water bath for 30 minutes.

During the 30 minutes the Spectrometer was prepared and an equal number of valves were cleaned for each of the tubes in the water bath. The valves were first washed with distilled water and then with Ethyl alcohol. Then they were again washed with distilled water and allowed to dry in air.

After completion of the 30 minutes the tubes were cooled and from each tube a small portion was taken to a valve to take 2 readings

from the spectrometer. Portions were taken from each tube. The entire set of readings has to be taken within 10 minutes time for better accuracy.

The same procedure was adopted for the other selected latex films and their relevant readings were taken from the spectrometer. Their protein content was found from the graphs and reported with the relevant sample.



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