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A TEST TRIAL IRRADIATION OF NATURAL RUBBER LATEX ON LARGE SCALE FOR THE PRODUCTION OF EXAMINATION GLOVES IN A PRODUCTION SCALE

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ABSTRACT

Radiation Vulcanization of natural rubber latex has been developed extensively through various research and development programme. During these investigations many data was collected and from these data it was proved that radiation vulcanized natural rubber latex (RVNRL) can be used as a new material for industry.(RVNRL symposium 1989; Makuuchi IAEA report) This material has been extensively tested in making of dipped goods and extruded products. However these investigations were confined only to laboratory experiments and these experiments mainly reflected material properties of RVNRL and only a little was observed about its behavior in actual production scale operation.

The present exercise was carried out mainly to study the behavior of the material in production scale by irradiating latex on a large scale and producing gloves in a production scale plant.

It was found that RVNRL can be used in conventional glove plants without making major alteration to the plant. Quality of the gloves that were produced using RVNRL is acceptable. It was also found that the small deviation of vulcanization dose will affect the crosslinking density of films. This will drastically reduce the tensile strength of the film. Crosslinking density or pre-vulcanized relax modulus (PRM) at 100% is a reliable property to control the pre vulcanization of latex by radiation.

INTRODUCTION

Radiation Vulcanization of Natural Rubber Latex (RVNRL) has several advantages over the conventional vulcanization with sulfur such as less or absence of toxicity free from nitrosoamines and accelarator induced allergies, low in cytotoxicity, and cleaner process. Research and development work on RVNRL has been carrying out since 1980's. Most of the work was confined to the laboratory tests while a little was examined in production scale operation.

In this exercise the behavior of natural rubber(NR) latex in production scale was studies. It is known that with increasing radiation dose the tensile strength of irradiate NR latex film passes through a maximum. Therefore the optimum radiation dose should be determined very accurately to obtain the maximum tensile strength of the latex films. Hence the vulcanizing dose was found by carrying out small scale irradiation before the pilot scale production. Two tons of specially prepared centrifuged natural rubber latex (Patent no. 10134) was prepared at Messrs. Latexam (pvt) Ltd. The irradiation of latex was carried out in two batches using the irradiator in Jakarta, Indonesia. Preparation of gloves with irradiated latex was done in a production scale plant at Hanwella Rubber Products (Pvt.) Ltd. in Sri Lanka. Then the two batches were tested in production in two separate stages. The properties of centrifuged latex used in this exercise is given in Table 1.

Table 1: Properties of NR Latex.

Property	Observed Value
Dry rubber content %	60.00
Total solid content %	61.5
MST sec.	1800
VFA	0.017
pH	10.00
Total alkalinity	0.35

EXPERIMENTAL

Materials

Centrifuged latex was supplied by latexam (Pvt.) Ltd. in Sri Lanka. Industrial grade normal butyl accrylate (n-BA), Ammonia were used. Antioxidants namely, Tris (nonylated phenyl) phosphite (TNP),2,5-Di-tert-amylhydroquinone (DAH) were supplied from Japan. All other raw materials required for the exercise were collected from established suppliers in the country.

The Co-60 irradiator at the Centre for the Application of Isotope and Radiation(CAIR) - BATAN, Jakarta was used in this study for irradiation. The activity of Co-60 was 104 kci and dose rate was 1.0 kgy/hr.

Determination of optimum dose

Preparation of latex for irradiation:

Latex was prepared for irradiation according to the following formulation.

	g
60% natural rubber latex	167
0.6% ammonia water	19.1
n-BA	5.00

Firstly natural rubber latex was diluted with ammonia water followed with the addition of n-BA. Normal butyl accrylate was added in small proportions directly to latex while latex was being stirred efficiently. The prepared latex was kept for 24 hr before irradiation.

Irradiation of latex:

Latex was irradiated to appropriate dose in 2.5 lt screw cap glass bottle. The Co-60 panoramic irradiator was used for irradiation of latex.

Preparation of cast films:

Films were cast using 10cm x 15cm glass molds. Cast films were leached in deionized water for 24 hours and dried in air. Dry films were heated at 70^{0}C for one hour and left in a desicator.

Preparation of films by dipping:

Coagulant dipping films were made by using a 7% solution of $Ca(No_3)_2$ in water. Leaching of films were carried out in wet gel stage at 80^{0}C for 5 minutes. Thereafter it was dried at 90^{0}C in an air circulating oven.

Testing:

Prepared RVNR latex and film were tested for tensile strength, croslinking density, pre vulcanized relaxed modulus (PRM) , and viscosity of the latex.

Pilot Scale Irradiation (1)

Latex was irradiated in pilot scale. Batch 1 was prepared to the following formulation.

	kg
Latex	925.0
n-BA	106.3
0.6% Ammonia water	27.7

First latex (185 x 5 kg) was charged in to the mixing vessel followed by the addition of ammonia water. Then n-BA was added slowly into the mixing vessel while stirring. Initially the stirrer speed was set at 35 r.p.m. It was found that this speed was not sufficient for adequate dispersion of n-BA. Therefore stirrer speed was increased to 70 r.p.m. and addition was high mixing was sufficient. The mix was left for 16-24 hours and irradiated to 12 kgy using the latex irradiator. Irradiation time was 12 hours.

Pilot scale irradiation (2)

The batch(2) was formulated similar to the first batch as follows:

	kg
Latex	925
n-BA	27.7
0.6% ammonia water	106.3
Ammonium laurate	0.270

Normal butyl accrylate was emulsified with 30 kg of ammonia water and 270g of ammonium laurate using the emulsifier. The emulsion was stable to carry out further addition. The stirrer speed of the mixing tank was increased to 70 r.p.m. followed with the addition of the rest of the ammonia water. Then n-BA emulsion was added directly in to the mixing tank during a period of 15 - 20 minutes. The stirring was continued for a period of one hour.

The batch was left for 24 hours and transferred to the irradiation vessel. The stirrer speed of the irradiation vessel was set to 25 r.p.m. The latex was irradiated with radiation from Co-60. PRM was measured at different intervals during irradiation. The irradiation was stopped after PRM reached the required value. Nitrogen gas was bubble through the irradiation vessel during irradiation.

Preparation of examination gloves

Latex was diluted to 45% TSC using 0.6% ammonia water. 1.6 phr of antioxidant was added to irradiated latex as a dispersion Antioxidant was a combination of TNP and DAH at a ratio of 3:1 (Makuuchi et al. 1993; Yoshi et al 1993; Chandralal internal report to IAEA). Coagulant was 7% $Ca(No_3)_2$ & 3% $CaCo_3$ in water with 0.3% non Ionic surfactant. The production rate was set for 56 formers per minute. Cycle time was about 30 minutes.

Testing of gloves

Gloves were tested for physical properties and aging. Aging was carried out at 70^0C for 7 days.

RESULTS AND DISCUSSION

In preparation of latex for irradiation, n-BA was added directly into the latex. There should be sufficient mixing for the monomer to get dispersed in the latex for neat addition of sensitiser.

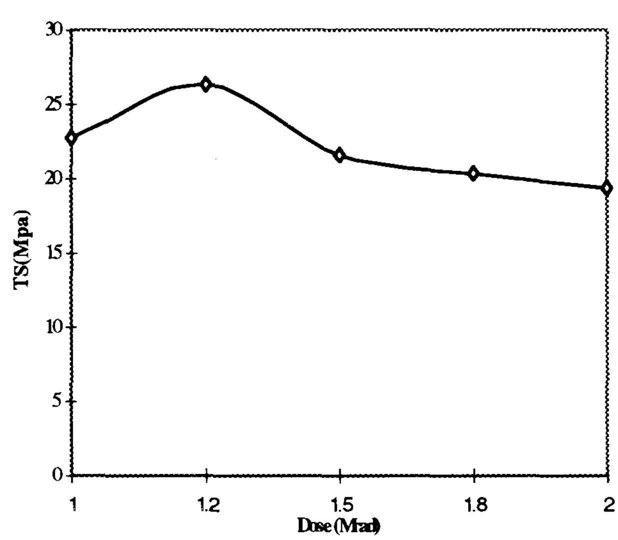

Fig. 3 Tensile Strength of films at different dose prepared with 5 phr n-BA

Figure 1: Tensile Strength films at different dose prepared with 5 phr n-BA

Figure 1 shows the tensile strength of films prepared by casting and by dipping at different radiation doses. It was observed that in both cases the maximum tensile strength of 26 Mpa was reached around 12 kgy radiation dose. Deviation from this value drastically reduces the tensile strength.

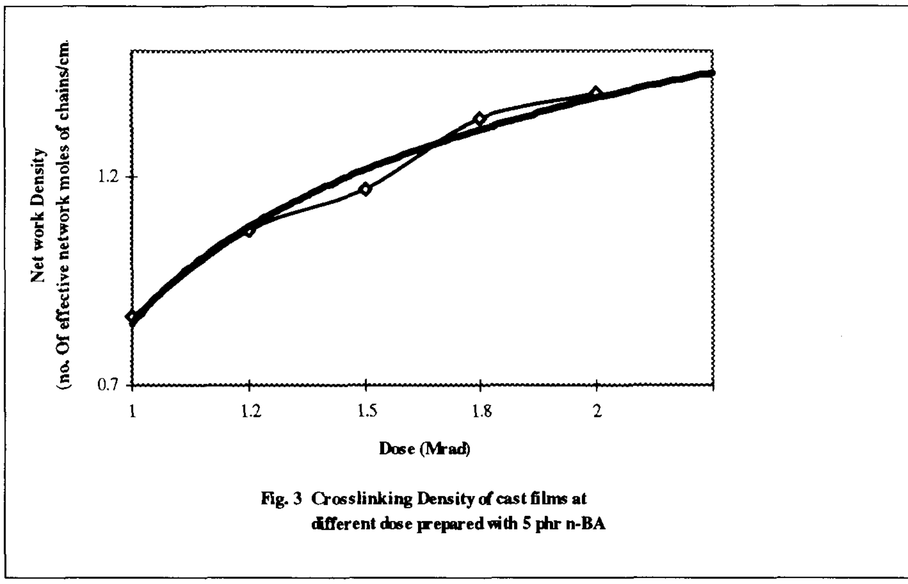

Figure 2: Crosslinking Density of cast films at different dose prepared with 5 phr n-BA

Figure 2 shows the crosslinking density of cast films prepared from irradiated latex at different doses. The crosslinking density was calculated using Flowry Rheiner Equation. It is found that maximum tensile strength is reached when crosslinking density is around 1.07 x 10 $^{-4}$ moles of crosslink/cm3. Crosslinking density was measured from dipped films showed similar values.

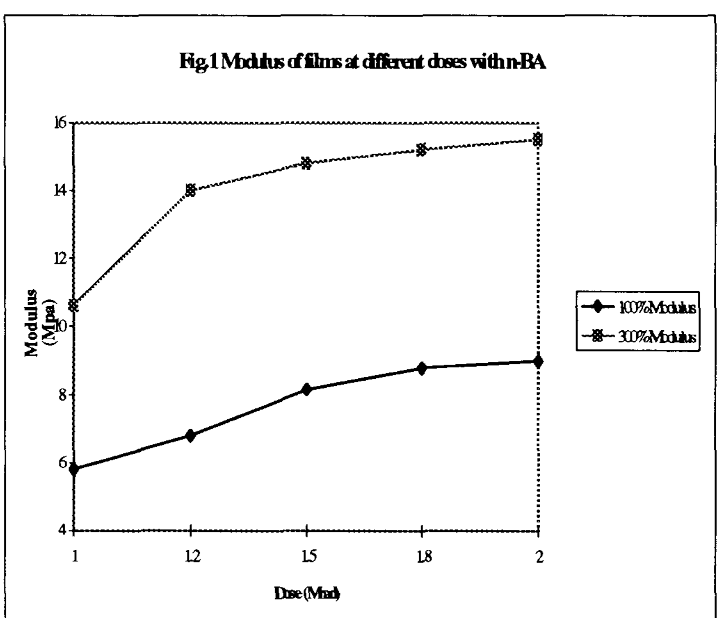

Figure 3: Modulus of films at different doses with n-BA

Figure 3 shows the 100% and 300% pre vulcanized relaxed modulus carried out for latex irradiated to different doses. It can be seen that both properties have similar trends. However from our investigations it was seen that the reproducibility of 100% is better than that of 300%. In our work modulus was recorded as an index.

Table 2 shows the linear swelling ratio of latex films made from irradiated latex. It can be seen that the difference in linear swelling of films over the proposed dose range is not significant.

Table 2: Linear swelling ratio of latex films made from irradiated latex

Dose (Mrad)	Swelling ratio %
1	100
1.2	93
1.5	91
1.8	90
2.0	88

Table 3 shows the viscosity of latex samples measured after two weeks. It can be seen that the viscosity of samples are below unirradiated sample which contains n-BA. The unirradiated latex shows slight increase in viscosity after 2 weeks which indicates that the special latex is stable against increase in viscosity due to n-BA addition.

Table 3: Viscosity of Irradiated latex.

Dose (kgy)	Viscosity (mpas).
10 after 2 weeks	23.7
12 after 2 weeks	21.2
15 after 2 weeks	21.9
18 after 2 weeks	22.5
20 after 2 weeks	22.5
0 after 1 day	30.3
0 after 1 week	35.6

Pilot scale irradiation (1)

The latex mixing tank wall consisted of coagulated latex and irradiated latex contained a small percentage of coagulum. The reason for this was that the neat addition of monomer to latex was not correct. This was mainly due to insufficient stirring which would have prevented the monomer from getting mixed with the latex. If the monomer was found floating without getting mixed with latex then there could be a possibility of lump formation.

Table 4: Physical properties of latex films made from pilot scale batch (1).

Property	Value
Coagulant dipped film	
Tensile strength Mpa	20
Elongation at break %	950
Cast film	
Tensile Strength Mpa	19.4
Elongation at break %	918
Crosslinking density mols/cm3	1.3×10^{-4}
100% PRM (index)	8.8

Examination gloves were made by coagulant dipping method. It was found that gloves could be very easily removed from the glove former without becoming tacky. Table 4 shows the tensile strength and elongation at break of films made by casting and dipping. Results reveal that tensile strength of these films are lower than what was expected. However the crosslinking density shows that the latex is over irradiated.

Pilot scale irradiation (2)

Table 5 shows the PRM value at different irradiation time. It can be seen that the sample after 8 hours of irradiation, reached the PRM value close to the value of the sample that received a radiation dose of 12 kgy from our laboratory experiment.

Table 5: PRM (index) at different time of pilot scale batch (2).

Time (hours)	PRM (index)
3	5.9
6	6.6
8	7.8

The irradiated latex sample did not have any coagulum as in the previous batch. This indicates that the addition of monomer after emulsifying to latex is a better method compared to the earlier method.

Table 6: Physical properties of latex films made from pilot scale batch (2).

Property	Value
Coagulant dipped film	
Tensile strength Mpa	25
Elongation at break %	1050
Cast film	
Tensile strength Mpa	25.1
Elongation at break %	1047
Crosslinking density mols/Cm^3	1.09×10^{-4}
100% PRM (index)	7.8

Table 6 shows the tensile strength value of films prepared with coagulant dipped and cast films. It also shows the crosslinking density of cast films. From these data it can be said that the crosslinking density of the present irradiated latex is slightly higher than what was required. However the batch was within specification for the production of examination gloves.

Glove Production in the Line

The gloves that were made were free of most of the common defects that are observed in conventional systems. The stripping of the glove from the former had no drastic difference compared to conventional system. However the gloves that were produce in this test trial had a thickness between 0.1 - 0.15 mm which is less than the thickness of a normal conventional examination glove. Due to low thickness sometime glove got torn in removing from the former. This could be over come by increasing the thickness.

Table 7: Temperature at different stages.

Stage	Temperature °C
Washing of formers	93
Drying oven 1	50
Coagulant	54
Drying oven 2	00
Dipping tank	30
Drying oven 3	45
Leaching tank	90
Oven entrance	45
Oven max	110
Oven exit	45

Table 8: Aging properties of coagulant dipped latex films from Batch (1) and Batch(2)

Sample	Tensile Strength (Mpa)	Elongation at Break %
Batch (1)		
Before aging	25.7	875
After aging	27.2	956
Batch (2)		
Before aging	27.2	945
After aging	28.0	878

* Aging at 70^{0}C 7 days.

Physical properties of the glove before and after aging is shown in Table 8. Results reveal that they are similar to value that was obtained in Indonesia. Aging properties are acceptable. The increase in tensile strength after aging may be due to better fusion of latex particles in the film.

CONCLUSION

RVNRL as a new material to produce gloves can be used in conventional plants without making major alteration. The gloves that were produced showed similarities in performance or better compared to gloves that were produced using conventional sulfur pre vulcanized systems. However to carry out detail analysis to optimum accepted quality level (AQL) a full scale trial has to be carried out in a production plant with more material.

Pre vulcanization relax modulus is a sensitive test for controlling the vulcanization during irradiation and it could give a grater control to the batch to complete at optimum vulcanization.

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