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Radiation vulcanization of natural rubber latex using 250 keV electron beam machine

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Abstract

The sensitized radiation vulcanization of natural rubber latex has been carried out with 250 keV electrons. Latex was irradiated over a range of the beam current from 5 to 20 mA in the presence of sensitizers like the *n*-butyl acrylate (*n*-BA). The vulcanization dose decreases with increasing beam current condition. The rate of vulcanization (R_{vul}) depends on the beam current (I) as given by the equation $R_{vul} = kI^{0.6}$. © 2003 Elsevier B.V. All rights reserved.

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1. Introduction

Natural rubber (NR) latex is conventionally cured by a reaction with sulfur, zinc oxide and accelerators. As accelerators dithiocarbamates are mostles used. And these compounds are potentially allergic. This is a problem, especially in under examination, for surgical uses, and in household gloves [1]. The main sensitizers were found to be the rubber accelerator of the carbamate. The bio-toxicity of latex catheters is known and is attributed to the dithiocarbamates accelerators [2]. All these difficulties and problems could be avoided by changing from sulfur-heat crosslinking to radiation vulcanization of NR latex (RVNRL).

As the price of a Co-60 source is likely to increase, the irradiation cost of NR latex will be much affected. Therefore, it is necessary to investigate the application of electron beam (EB) for irradiation of NR latex, since the irradiation cost by electron accelerators is expected to be much lower than that for gamma irradiation [3]. The compact size of an EB irradiator is another advantage. One of the handicaps in using EB irradiation is the low penetration of the beam. For a 250 keV EB machine, the beam penetration is about 0.3 mm in NR latex.

The purpose of this paper is to report on our study of the effect of dose rate or beam current on the vulcanization dose of sensitized NR latex irradiated with low energy EB.

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2. Experimental

Concentrated and commercial high ammonia NR latex was used in our work. The total solids content was reduced from 60% to 30% using ammonia solution (1%). *n*-Butyl acrylate (*n*-BA, 5 phr) without purification was used and slowly added into diluted NR latex.

The latex irradiation vessel under EB was a cylindrical stainless steel vessel (29 cm diameter, 30.5 cm height) containing four plates on the inner wall at 90° intervals and fitted with a propeller type stirrer and outer jackets. The accelerating voltage was fixed at 250 keV and the beam current was varied (2.5, 5.0, 7.5 and 10.0 mA). Sensitized latex (9 l) was poured into the irradiation vessel. The sensitized latex was irradiated under EB at 150 rpm with constant stirring. The irradiation time (5, 10, 15, 20, 25 and 30 min) and EB current (2.5, 5, 7.5 and 10 mA) conditions varied to determine the relationship between vulcanization rate versus beam current. The same sensitized latex was irradiated under gamma rays from a Co-60 source with 10 kGy/hr dose rate and 10, 20, 30, 40 and 50 kGy irradiation doses. Both gamma facilities and EB machine used are in the Japan Atomic Research Institute.

The preparation of cast films (0.2 mm thick) was done by spreading. 13 ml of irradiated latex on each of the several raised rimmed glass plates. They were air dried (48 h) to transparent films, in a circulation oven at 50 °C for 24 h after leaching. The leaching of the films was done with 1% ammonia solution at room temperature. The period of leaching was about 30 min.

3. Measurement of tensile strength at break (Tb)

Dumbbell shaped test pieces were cut using standard dumbbell cutter ASTM D 1822-L of precise size for natural rubber film. The thickness of the samples was measured using a micrometer (KS Automation Co. Ltd.) The average thickness was 0.23 mm with 4.0% deviations coefficient of the five samples used.

Tensile strength at break (Tb) and strain at break (Eb) were measured with a tensile machine,

Strografh-R1, Toyoseiki, Japan. All tests were done in room temperature.

4. Measurement of swelling ratio and crosslink density

A weighed amount of rubber film was immersed into toluene for 48 h. The rubber film was taken out from the solvent and the swollen film was weighed. The swelling ratio was calculated from the difference of the weight of the films before and after swelling. The crosslink density was calculated from Floury and Rehner equation as follows:

Crosslink density (number of cross-link/ml)

$$= K \times Q^{-5/3}$$

where K is a constant $(4.71 \times 10^{20}$ for a system of toluene–natural rubber) and Q is the swelling ratio.

5. Results and discussion

When the NR latex was irradiated by gamma rays from a Cobalt-60 source with 10 kGy/h dose rate, the vulcanization dose of the NR latex sensitized with 5 phr of n-BA is around 12 kGy. The vulcanization dose decreased in the oxygen bubbling before irradiation, as shown in Fig. 1. These results confirm the Makuuchi's suggestion [4] that the NR latex contains natural radical scavengers, which prevent radiation vulcanization, and oxygen



Fig. 1. Tensile strength of films from RVNRL by gamma-rays bubbling nitrogen and oxygen, sensitized with 5 phr *n*-BA.

decomposes the radical scavengers. The maximum tensile strength of the films from irradiated latex by gamma rays was 32.8 MPa.

Fig. 2 shows that the maximum tensile strength of 29.1 MPa for the films from irradiated latex by EB was achieved in 20 m, with a 10 mA beam current. This figure shows that the vulcanization dose decreases by increasing the beam current. The tensile strength and elongation at break (Figs. 2 and 3) were in agreement with the swelling ratio and gel fraction data (Figs. 4 and 5). A higher gel fraction value also indicates a higher crosslink density. The gel fraction was found to reach a maximum value in 10 m at 7.5 and 10 mA beam current conditions respectively, as shown in Fig. 5.



Fig. 2. Tensile strength of films from NR latex vulcanized by 250 keV EB, sensitized with 5 phr *n*-BA.



Fig. 3. Elongation at break of films from NR latex vulcanized by 250 keV EB, sensitized with 5 phr *n*-BA.



Fig. 4. Swelling ratio of films from vulcanized latex by 250 keV EB, sensitized with 5 phr *n*-BA.



Fig. 5. Gel fraction of films from vulcanized latex by 250 keV EB, sensitized with 5 phr *n*-BA.



Fig. 6. Effect of beam current at 250 keV on crosslink density.



Fig. 7. Effect of beam current on rate vulcanization.

Fig. 6 shows the effect of beam current on the crosslink density. From the initial slope of the rate vulcanization (R_{vul}) was calculated and plotted against the beam current (*I*) as shown in Fig. 7. It gives a linear relationship with the slope which equals 0.6. The dependence of the rate of vulcanization on the beam current is given by

$$R_{\rm vul} = k \times I^{0.6},$$

where k is a constant.

6. Conclusion

The vulcanization dose of sensitized NR latex irradiated with low energy EB was affected by the beam current. The vulcanization dose decreases by increasing the dose rate. The relationship between vulcanization rate versus beam current is given by $R_{\rm vul} = k \times I^{0.6}$.

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