

SULPHUR-FREE PREVULCANIZATION OF MALAYSIAN NATURAL RUBBER LATEX USING A HYBRID ULTRAVIOLET-PEROXIDE VULCANIZATION METHOD

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ABSTRACT

A preliminary study on vulcanized natural rubber latex via hybrid ultraviolet C (UVC) and peroxide vulcanizations was conducted using 2-hydroxy-2-methyl-1-phenylpropanone (Irgacure 1173) as the photoinitiator, 1,6-hexanediol diacrylate (HDDA) and tert-butyl hydroperoxide (t-BHPO) as the sensitizers/coagents. The effects of various amounts of t-BHPO on crosslink density, mechanical and physical properties of UV-peroxide prevulcanized natural rubber latex were investigated and recorded. The tensile strength, modulus at 500% and modulus at 700% elongation of rubber film obtained from one-hour UVC irradiation (temperature within 70-75 °C) were 22.0, 3.0 and 9.0 mPa respectively, which is more than 83 % of increment compared to the control sample. The crosslink density of the rubber films showed an increment of 28.5 %.

Keywords: UVC, vulcanization, irradiation, latex

INTRODUCTION

Natural rubber latex or its scientific name *cis*- 1, 4- polyisoprene naturally exists in the form of a white, sticky and non-elastic liquid. In the middle of the 19th century, Charles Goodyear managed to find a method to overcome all the weaknesses found in natural rubber. This method is known as sulphur vulcanization. The sulphur vulcanization requires four different materials that need to be mixed in the natural rubber latex; i.e. sulphur as the cross-linking agent, carbamate compounds as accelerator, zinc oxide (ZnO) as activator and potassium hydroxide (KOH) as stabilizer. The mixture is then heated to a temperature of 40-65 °C for a maximum duration of 10 hours. This process can impart some major properties such as chemical resistance, aging resistance, improved film properties and mechanical strength which are not present in the unvulcanized natural rubber latex.

However, the chemicals used in this process tend to cause health problems such as skin allergies to users of the latex products. In 2016, it was reported that average prevalence of latex allergy worldwide remains at 9.7%, 7.2%, and 4.3% among healthcare workers, susceptible patients, and the general population, respectively [Wu *et al.*, 2016]. Researchers have found out that the carbamate compounds that are used as accelerators to speed up the vulcanization process and reduce the reaction temperature have high risk on tissue irritations and cytotoxicity. This issue has led researchers to look for vulcanization technique that is free from sulphur and its related compounds.

Currently, there are two alternative crosslinking processes that are free from sulphur, i.e. peroxide (chemical vulcanization) and ionizing radiation (electron and gamma rays). Both of this processes do not use chemicals related to sulphur vulcanization such as accelerator and activator which may cause skin allergic, emission of sulphur dioxide, high residual chemical in the end product and pollution to environment. However, further consideration has to be made since both crosslinking techniques had

their own issues such as high capital investment in infrastructure, production and maintenance cost, low physical properties of final products and longer vulcanization time than sulphur vulcanization [Lazim *et al.*, 2021].

Since the last decade, the study on vulcanization of natural rubber latex through the use of non-ionizing UV radiation has begun to receive attention from researchers. Schlögl [2010] has successfully produced prevulcanized natural rubber latex with the help of a photoinitiator and a poly-functional thiol. Through this vulcanization technique, sulphur-free prevulcanized natural rubber latex with good biocompatibility and low amount of leachable chemical compound has been successfully produced. Wiroonpochit [2017] thru her research has combined the use of photoinitiator with coagents such as 1,4-butanediol diacrylate (BTDA), 1,6-hexanediol diacrylate (HDDA) and 1,9-nonanediol diacrylate (NDDA). The results of the study showed that prevulcanized natural rubber latex with relatively good mechanical strength has the potential to be produced.

The present work attempts to combine UV and peroxide vulcanizations with the presence of acrylate coagent. Effects of the following factors on the mechanical properties (tensile strength) of the resulting prevulcanized natural rubber latex were assessed in order to determine optimal conditions for prevulcanization, i.e. amount of peroxide and the duration of the UV irradiation. This research output may trigger the diversification encourage the use of sulphur free prevulcanized natural rubber latex in local latex product industries and abroad.

MATERIALS AND METHODS

Materials

The natural rubber latex (NRL) utilised in this work was a high ammonia type (HA latex) supplied by Getahindus (M) Sdn. Bhd., Malaysia. The sensitizer used was hexanediol diacrylate (HDDA) supplied by Allnex, China and tert-butyl hydroperoxide (*t*-BHPO) supplied by Fluka. The photoinitiator, 2 hydroxy-2-methyl-1-phenylpropanone (Irgacure 1173) was purchased from Sigma Aldrich (M) Sdn. Bhd., Malaysia. The stabilizer used was Potassium laurate supplied by Tiarco Chemical (M) Sdn. Bhd., Malaysia. The activator used was Hydroxyacetone (HAC) supplied by Sigma Aldrich (M) Sdn. Bhd., Malaysia. All the stated materials were used as received.

Preparation of the latex formulation

Latex formulations prepared in this study were planned and decided based on present study objectives and findings of previous studies [Lazim, M. N. M., *et al.*, 2021; Cahya, W. & Herry, P. 2018; Hansupalak *et al.*, 2016 & Wiroonpochit *et al.*, 2017]. The steps for preparing the latex formulations are described below.

Preparation of UV prevulcanized natural rubber latex in the presence of diacrylates (control sample without peroxide)

A latex formulation of 52% total solid content was prepared from materials listed in Table 1. The NRL was transferred to a beaker and followed by addition of stabilizer, sensitizer, photoinitiator and water while stirring within 5 minutes interval. The beaker was covered with aluminum foil to avoid light exposure [Lazim, M. N. M., *et al.*, 2021 & Sofian *et al.*, 2018]. The latex mixing was done at room temperature in the dark. Once the addition of the materials was completed, the latex mixture

was left stirring for one hour. The formulated latex was then transferred into 250 x 350 mm polyethylene (PE) bags and sealed, of which the thickness of the plastic bag containing latex sample is less than 10 mm. The latex sample in PE bags were subjected to UVC irradiation for one hour as described the UVC irradiation section. After irradiation, the latex was made into film using a coagulant dipping method and the mechanical properties of latex films were performed [Pairu *et al.*, 2016].

Table 1. Compounding formulation of UV pre vulcanized natural rubber latex (Control)

Materials	Part per hundred rubber (pphr)
NRL (62% *TSC)	100.0
Stabilizer	0.1
HDDA	2.0
Irgacure 1173	2.0
Water	Add to 52% TSC

*TSC- Total solid content

Preparation of hybrid UV-peroxide pre vulcanized natural rubber latex at different amount of *t*-BHPO

Latex formulations as shown in Table 2 were prepared by adding the stabilizer, sensitizer, peroxide (various amount), activator, photoinitiator and water into NRL within 5 minutes interval while stirring in the dark and at room temperature. The latex mixture was stirred for one hour, followed by transferring to 250 x 350 mm PE bag and sealed. The thickness of the plastic bag containing latex sample is less than 10 mm. The latex samples were subjected to UVC irradiation for one hour as described in the UVC irradiation section. After irradiation, coagulant dipped rubber films were prepared from the latex samples and the mechanical properties of latex films were performed.

Table 2. Compounding formulation of Hybrid UV-peroxide pre vulcanized natural rubber latex at different amounts of *t*-BHPO

Materials (pphr)	Part per hundred rubber
NR Latex (62% TSC)	100.0
Stabilizer	0.1
HDDA	2.0
* <i>t</i> -BHPO	
0.5/1.0/1.5	
Irgacure 1173	2.0
HAC	0.5
Water	Add to 52%
TSC	

UVC irradiation

The latex formulations packed in plastic bags were irradiated with four UVC lamps (Philips TUV PL-L 55W/4P HF 1CT); of which two UVC lamps were placed at the top and another two UVC lamps placed at the bottom as shown in Figure 1. The intensity of the incident radiation at the surface of the plastic bag was 3707.9 $\mu\text{W}/\text{cm}^2$ (measured by Non Ionized Radiation Group, Malaysian Nuclear

Agency). The distance between the lamps and the surface of the latex sample were 100 mm and all experiments were conducted in a dark room.

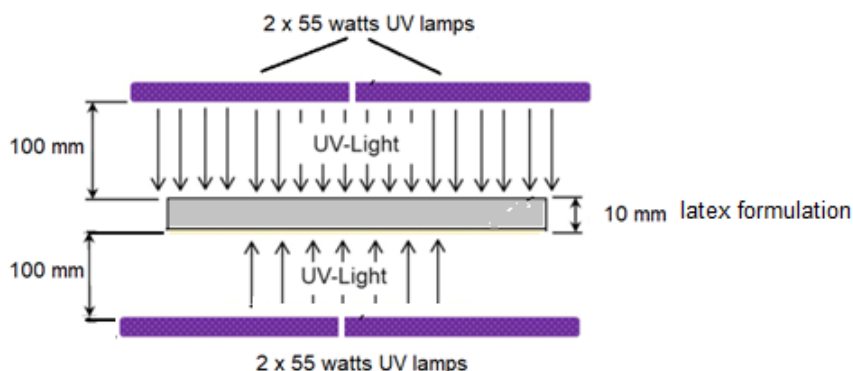


Figure 1. The experimental setup for UVC irradiation of latex formulation samples

Measurement of tensile properties

The latex films made by the coagulant dipping method were cut into dumbbell shape test pieces according to MS 1523:2001 (Figure 2). For each latex film sample, five dumbbell test pieces were used for the tensile test (using Universal Testing Machine SHIMADZU, M703911, 50N) and a median value was taken as the final result in accordance with ASTM D412.

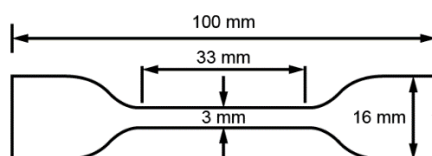


Figure 2. Dimension of dumbbell cutest piece

Determination of gel content

The gel content or percentage of latex film crosslinked density was determined by the extraction of latex film in toluene for 8 hours using Soxhlet apparatus as required by [ASTM D3616-95, 2014; Jayasuriya *et al.* 2001]. Then the extracted samples were dried in an oven at 70°C till constant weight was achieved. The gel fraction was calculated as Equation (1):

$$\text{Gel content, \%} = \frac{w_1}{w_0} \times 100 \quad (1)$$

where W_0 and W_1 are the weights of the dried samples before and after extraction, respectively.

Determination of total solid content (TSC)

The total solids content (TSC) is the percentage by weight of latex which is non-volatile at a definite temperature. The standard method is as described in ISO 124:2014. In this method, 2.0 g ± 0.5 g of latex was poured into the petri dish and weighted to the nearest 0.1 mg. Then the dish was placed into

an oven and heated at $70^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 16 h or at $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 2 hours or until the test portion has lost its whiteness. After that, the dish was cooled down in a desiccator and weighted, and the mass of dry latex was recorded. The TSC was calculated as Equation (2):

$$\text{Total solid content, \%} = \frac{m_1}{m_o} \times 100 \quad (2)$$

Where m_o and m_1 is the weight (gram) of the latex and dried latex, respectively.

Determination of alkalinity

ISO 125 standard was referred for determination of alkalinity. 5 g of latex sample was poured into a 500 cm³ beaker and followed by 200 cm³ of water. The solution was stirred thoroughly. While stirring, the pH meter electrode was inserted into the solution. Hydrochloric acid solution was added slowly into the solution via titration until the pH is reduced to a value of 6.00 ± 0.05 .

The alkalinity of the latex was calculated as Equation (3):

$$\text{Alkalinity} = \frac{1.7cV}{m} \quad (3)$$

Where c is the actual concentration of HCl per cubic decimetre of acid used, V is the volume, in cubic centimetre, of acid used; and m is the mass, in grams, of the latex sample.

Determination of mechanical stability (MST)

The latex samples were subjected to MST testing using Klaxon MK3 machine in accordance with ISO 35:2004 requirement, where $80.0 \text{ g} \pm 0.5 \text{ g}$ of the latex was first added into MST machine container. Then the container was positioned in the Klaxon MK3 MST machine and stirred at $14000 \text{ rev/min} \pm 200 \text{ rev/min}$ throughout the test until the end-point is reached. The arrival of the end-point is preceded by a marked decrease in the depth of the vortex around the stirring shaft, loss of turbulence and change in the sound of stirring action. The end-point which is the first appearance of flocculum was determined by removing a drop of the sample with a clean glass rod at intervals of 15 seconds and gently spreading the sample on a suitable surface; for example, the palm of the hand.

Measuring of viscosity

Latex sample with temperature range at $25 - 29^{\circ}\text{C}$ was measured its viscosity in accordance with MS 281 standard requirement. The viscometer used in this study was Brookfield model DV-II+. 200 g of latex was weighed into a 250 ml capacity beaker. Then the spindle was inserted into the latex and the machine was set at 60 rotation speed per minute (RPM) frequency. The equilibrium viscosity reading is attained after 20 to 30 seconds.

RESULTS AND DISCUSSION

Effect of *t*-BHPO on mechanical properties of hybrid UV- peroxide prevulcanized natural rubber latex

In UV vulcanization, the UVC light will be absorbed by photoinitiator present in latex formulation to form free radicals. These free radicals will help to form crosslinks in polyisoprene. For this study, the UV dose delivered to form free radicals were calculated as below;

$$\begin{aligned}\text{UV dose} &= \text{UV light intensity } (\mu\text{Wsec}/\text{cm}^2) \times \text{time (s)} \\ &= 3707.9 \times 3600 = 13348440 \mu\text{Wsec}/\text{cm}^2; \text{ which } 1\mu\text{Wsec}/\text{cm}^2 = 0.001 \text{ mJ}/\text{sec} \cdot \text{cm}^2 \\ &= 13348.44 \text{ mJ}/\text{cm}^2\end{aligned}$$

Besides, the vulcanization method using UV radiation will also generates heat from the UV lamps. Since the hybrid UV-peroxide vulcanization method uses peroxide as the co-sensitizer, the heat produced from the UV lamps (irradiation for one hour has caused the temperature of the samples to increase within 70-75 °C) will be manipulated to decompose the peroxide compounds for the formation of radicals that will help in improving the mechanical properties and cross-link density of hybrid UV-peroxide prevulcanized natural rubber latex. The heat was produced alongside UV irradiation and caused the increment of latex temperature up to 70 – 75 °C. In peroxide vulcanization, heat is required to decompose peroxide compounds to form free radicals and produce crosslinks to improve the mechanical properties of the vulcanized latex. Hence, it is anticipated that the produced heat from UV irradiation can be a compliment to UV and photoinitiator in decomposing peroxide to achieve vulcanization of natural rubber latex.

Tensile strength properties

Tensile strength is considered one of the most important parameters by latex product industries when qualifying a latex product for intended use during the development and quality control stages. A latex product must possess the required minimum tensile strength to confirm its quality and usefulness. For example, a nitrile examination glove requires a minimum tensile strength of 14 mPa, whereas a latex surgical glove may need a minimum tensile strength of 24 mPa [ASTM D3577].

Modulus is another mechanical property of latex film. However, it cannot be used as an indicator of the quality of natural rubber latex films. Although modulus value is frequently used to indicate the degree of crosslinking in a film, it does not reveal the film's physical strength. In industry, modulus value is used to quantify the stiffness of a film that is subjected to stress. Tensile strength values are the greatest stress that a film can bear while being stretched before breaking, and they are a significant factor in defining the elasticity of the film. Therefore, for this study, the modulus and tensile strength of the films were measured.

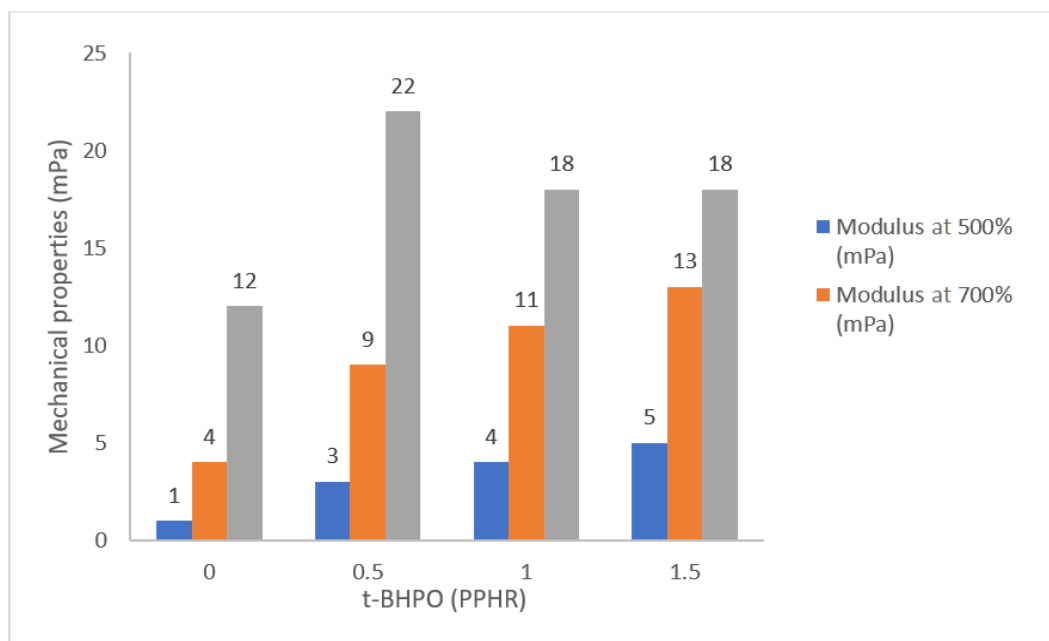


Figure 3. Mechanical properties of hybrid UV-peroxide prevulcanized natural rubber latex (taken as the median values from tensile test results) prepared with different amount of *t*-BHPO

Figure 3 gives the mechanical properties of control and hybrid UV-peroxide prevulcanized natural rubber latex samples that have been prepared by UV irradiation at various amount of *t*-BHPO and tested as required by ASTM D412 standard. In comparison to the control sample, it is obvious that hybrid UVNRL-peroxide samples with *t*-BHPO showed improved mechanical properties upon UV irradiation. The tensile strength and modulus of hybrid UVNRL-peroxide sample with 0.1 pphr *t*-BHPO showed around 100% increment compared to the control sample. The increment in tensile strength, modulus at 500% and modulus at 700% are due to the enhancement of intra-particle crosslink density (chemical crosslinking) induced by UV radiation and monogeneity of the vulcanization from the UV radiation and peroxide [Siri Upathum 1996]. However, it was observed that the addition of 0.2 and 0.3 pphr of *t*-BHPO into the formulation will produce latex films with higher modulus at 500% and modulus at 700% but lower tensile strength values. This indicates that the formation of cross-links occurs at a high rate as well, causing the C-C bond to become too stiff and easy to break, hence, latex films with lower tensile strength.

Effect of irradiation doses on gel content of hybrid UV-peroxide prevulcanized natural rubber latex

Gel content is a measurement of crosslinking that exists in latex polymer film. Gel content is also one of the important parameters in latex product manufacturing as it correlates with the mechanical properties such as tensile strength. Gel content provides stronger evidence to support the tensile strength and modulus value obtained from the previous section.

Based on the modulus value shown in Figure 3, it was observed that the value increases as the concentration of *t*-BHPO increases, as well as the decrement of tensile strength with *t*-BHPO beyond 0.1 pphr. It was assumed that there is a higher amount of crosslinking produced that made the latex film become stiff. From the data obtained that was illustrated in Figure 4, the average gel content of the latex film increases as the concentration of *t*-BHPO increases. It can conclude that the concentration of *t*-BHPO is directly proportional to modulus value, but it was inversely proportional with tensile strength beyond 0.1 pphr of *t*-BHPO, due to an increase of gel content or crosslink.

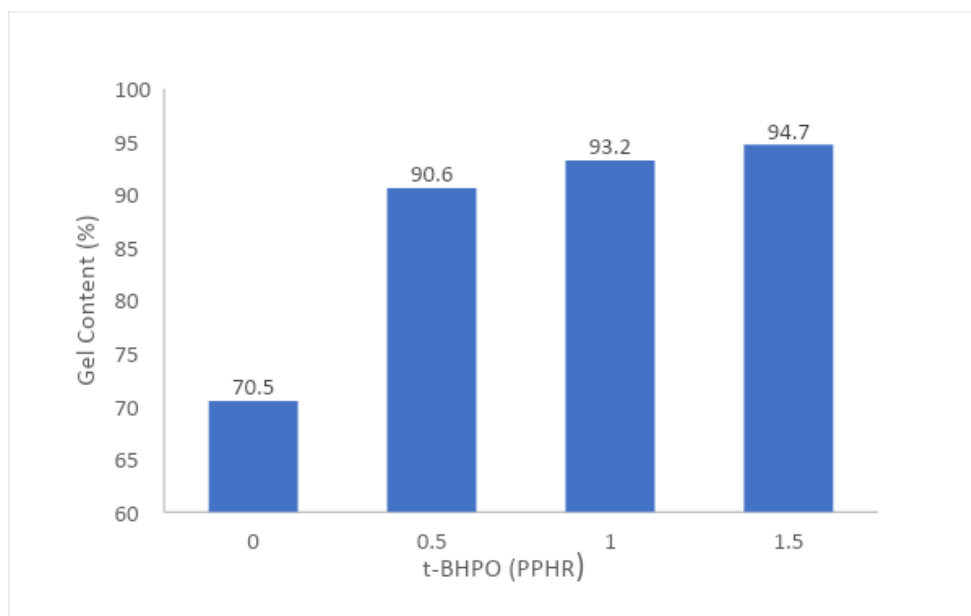


Figure 4. Gel content of hybrid UV-peroxide prevulcanized natural rubber latex prepared at different concentration of *t*-BHPO

Effect of *t*-BHPO on physical properties of hybrid UV-peroxide prevulcanized natural rubber latex

Table 3. Comparisons of physical properties of the raw latex, control (UVNRL) and hybrid UVNRL-peroxide

Sample	Raw latex	Control (UVNRL)	Hybrid UVNRL-peroxide	Specification	Test Method
TSC, %	62.13	52.25	52.11	Min 52	ISO 124
Alkalinity, %	0.65	0.57	0.50	0.3-0.7	ISO 125
MST, sec	950	1000	1150	650	ISO 35
Viscosity, cps	31.71	33.75	35.55	30-40	MS 281

TSC is the measurement of the percentage of rubber (hydrocarbon) and non-rubber contained in latex. TSC is an important indicator to show a latex formulation is correctly prepared in accordance to plan. Additionally, through this method the presence of non-volatile but soluble additives are also measurable [Akademi Hevea Malaysia 2012].

During the preparation of the hybrid UVNRL-peroxide prevulcanized natural rubber latex, the final latex concentration along with a mixture of acrylate, peroxide, activator, stabilizer, photoinitiator and water will be ensured at a minimum of 52% TSC. The selection of 52% of TSC was made for two reasons. The first reason is to prevent an increase in latex viscosity after the UV radiation procedure is complete and the second is to improve the crosslink density in prevulcanized natural rubber latex by the presence of OH radicals produced from water radiolysis [Makuuchi 2003]. However, one needs to be careful as latex with a low percentage of TSC will be difficult to produce thick rubber latex products due to a lack of rubber molecules in the latex.

Apart from TSC, measuring the alkalinity of the prevulcanized latex is also crucial. Alkalinity refers to the latex's free alkaline content, which is typically expressed as the latex's ammonia concentration in percentage. Ideally, prevulcanized latex's alkalinity should fall between 0.3% and 0.7%. Under an alkaline environment, fatty acids and proteins will be ionized to produce negative charges on the rubber particles surface. The colloid stability of the latex will be maintained by the presence of this charge particles since two particles with similar negative charges that are nearby will repel one another due to electrostatic attraction.

When the latex alkalinity is lower than 0.3 %, it will create a suitable environment for the bacteria to break down natural proteins in latex and spoiling it [Abu Bakar & Rosley 1994]. Meanwhile, ammonia over 0.7 % will disrupt the production process of dip-products through difficulty to form thin latex gelatine lining in the industrial production line. According to the results of this study, adding *t*-BHPO, HDDA, HAC and Irgacure 1173 had no significant effect on the alkalinity of the hybrid UVNRL-peroxide samples as compared to control.

The MST test is used to examine the stability and resistance of latex to mechanical agitation. In addition, it can be described as the amount of time, measured in seconds, needed for rubber particles to coalesce when latex is stirred at a high speed under precise circumstances [Akademi Hevea Malaysia 2012]. In the production of latex dip-products, the minimum requirement for the MST of the prevulcanized latex is 650 seconds. In dipping tanks at factory production lines, latex with a low MST value can clump quite quickly. This will cause a defect in the manufactured products. From Table 3, it was found that the addition of *t*-BHPO, HDDA, HAC and Irgacure 1173 into the hybrid UVNRL-peroxide compounding formulation can help to improve the stability of the latex towards mechanical agitation.

According to the research of Blackley (1997) and Roshanie (2010), oxygen (from the air) can create hydroperoxide groups on rubber molecules, and those groups may be ionised to unstable ions. Subsequently, this ion encourages the breakdown of proteins and phospholipids to produce polypeptides and amino acids, the latter of which can be converted into a variety of compounds, including glycerol, fatty acid anions, and phosphate anions. These materials are then absorbed at the particle interface, increasing the latex's stability. Coincidentally, oxygen and water are the by-products of peroxide decomposition in peroxide vulcanization. Thus, it was proposed that oxygen produced by the breakdown of *t*-BHPO might aid in increasing the latex's stability.

The final physical properties measured are latex viscosity. The viscosity of the prevulcanized latex must be kept under control at a specific range so that the finished goods fall within the desired thickness range, which makes this parameter crucial to the manufacturer of latex dip-products. Typically, the manufacturer specifies a viscosity range of 30 to 40 centipoise (cps). According to Table 3, despite the fact that the viscosity of the hybrid UVNRL-peroxide sample increased marginally, it still falls within the acceptable range. It is suggested that the addition of *t*-BHPO, HDDA, HAC and Irgacure 1173 or other chemicals will disturb the stability of the rubber particles in the hybrid UVNRL-peroxide and cause a minor increment of latex viscosity. This can be tackled by increasing the amount of stabilizers used in the compounding formulation of hybrid UVNRL-peroxide.

CONCLUSION

Prevulcanized NRL with good mechanical properties can be prepared by hybrid UV and peroxide vulcanization. UV irradiation of latex formulations based on 2.0 pphr of Irgacure 1173 as photoinitiator, 2.0 pphr of HDDA and 0.1 pphr of *t*-BHPO as co-sensitizers at one-hour exposure time (temperature within 70-75 °C) can produced rubber film with tensile strength of 22 mPa and crosslink percentage of 90.6%. In addition, this vulcanization method does not cause significant adverse changes to the physical properties of the prevulcanized natural rubber latex. Hence, it can be concluded that hybrid UV-peroxide vulcanization is a potential sulphur-free vulcanization method for natural rubber latex.

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