



The Effects of HDDA and n-BA & TMPTMA on the Physical and Thermal Properties of UV Irradiation Vulcanization Natural Rubber Latex

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Abstract

Natural rubber (NR) latex was vulcanised by exposing it to ultraviolet (UV) irradiation for 1.5 hours in the presence of 1,6 hexanediol diacrylate (HDDA), n-Butyl acrylate (n-BA), and trimethylolpropane trimethacrylate (TMPTMA). 2-hydroxy-2-methyl-1-phenylpropanoid was used as a photoinitiator. The effect of HDDA, n-BA, and TMPTMA on the physical, and thermal properties of ultraviolet irradiation vulcanisation natural rubber latex (UVNRL) was investigated. The wetting and absorption of acrylate monomer into NR film were determined by using contact angles, and absorption tests. Tensile strength, modulus, and crosslink density were investigated to study the physical properties. The Fourier Transform Infrared spectroscopy (FTIR) was used to determine the chemical interaction between acrylate monomer, and NR. The thermal properties of UVNRL samples were determined using a thermogravimetric analyser (TGA), and a differential scanning calorimetry (DSC). It was found that NR latex formulated with HDDA had the highest tensile value and stable thermal degradation. The carbonyl group attached in UVNRL is shown in the FTIR spectrum. The diffusion of acrylate monomer into NR latex particles and the number of functional groups in acrylate monomer influenced the properties of UVNRL.

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1 Introduction

NR latex is a natural polymer with tensile strength around 3-4MPa, and it is easy to lose its elasticity upon stretching. The vulcanisation process is introduced to enhance tensile properties by

chemically crosslinking the 1,4 polyisoprenes chain in NR to form 3 dimensional networks. The vulcanised NR latex has improved tensile properties but still maintains its previous homogeneity, and fluidity. There are three types of vulcanisation namely sulfur, peroxide, and radiation vulcanisation. The accelerated sulfur vulcanisation NR latex (SVNRL) produced products with superior tensile strength as compared to radiation, and peroxide vulcanisation (Ramli *et al.*, 2014). Sulfur vulcanisation offers many advantages such as the lack of oxygen, and moisture sensitivity, with cheap, and easy processes to be carried out. Researchers have developed sulfur-free vulcanisation processes such as peroxide vulcanisation NR latex, and radiation vulcanisation NR latex which have several advantages over SVNRL, such as less toxicity, being free from nitrosamines, being low in cytotoxicity, and being environmentally friendly (Makuuchi, and Hagiwara, 1984). Unfortunately, sulfur-free vulcanisation processes often result in other drawbacks, such as poor aging stability, long vulcanisation time, high cost of investment in set-up, production, maintenance of facilities, and operational safety.

The UVNRL has been developed to produce NR latex that is free from accelerated sulfur chemicals that cause latex allergy. The UVNRL has good biocompatibility, is less dangerous in terms of operation safety than radiation vulcanisation, has a low amount of leachable chemical compound, and no need for costly shielding measures against ionising radiation, and low energy consumption. UVNR latex crosslinking is based on free radical mechanisms, such as thiol-ene addition reaction, radiation-induced, and peroxide dissociation. The first step of UV light vulcanisation is the hemolytic cleavage of a photoinitiator. UV light is absorbed by a photoinitiator present in latex formulation, undergoes unimolecular bond cleavage, and forms free radicals. The free radicals generate thiyl radicals by hydrogen transfer. The crosslinking is generated due to the addition reaction between thiyl radicals, and C=C double bonds in poly-isoprene. The thiyl radicals are formed again due to a hydrogen abstraction from another thiol by carbon-centred radicals. Termination occurs due to radical coupling under the formation of disulfides, thioethers, and covalent carbon-carbon bonds (Schlögl *et al.*, 2012).

Several factors contribute to efficient UV irradiation on NR latex, such as the type of UV lamp used for irradiation, photoinitiator, type of monomer, and duration of irradiation. In a recent publication, a low-pressure UV lamp at 8W was used for vulcanising NR latex formulated with acrylate monomer, and propanone photoinitiator. The optimum tensile properties were achieved at 12.05MPa (Wiroonpochit *et al.*, 2017). In this work, n-BA, HDDA, and TMPTMA were used as a monomer in the NR latex mixture and 2-hydroxy-2-methyl-1-phenylpropanone photoinitiator. The UV light source was from a medium pressure mercury UV lamp 400W (250-350nm wavelengths). The effects of acrylate monomer on tensile, and thermal properties of UVNRL were analysed.

2 Method

The NR latex concentrated at 60% total solids content (tsc) preserved with 0.6% ammonia was obtained from Titilatex (M) Sdn Bhd. The photoinitiator, 2-hydroxy-2-methyl-1-phenylpropanone (Irgacure 1173), HDDA, n-BA, and TMPTMA were purchased from Sigma-Aldrich

(M) Sdn Bhd. 20% Potassium Laurate solution and 10% KOH solution were prepared in a laboratory. All chemicals were used as received without further purification.

Table 1: The latex formulation for preparing UVNRL

Materials	Part Per Hundred Rubber (pphr)
HA latex	100
Acrylate monomer (HDDA, n-BA, TMPTMA)	3
Irgacure 1173	2
Potassium laurate	0.2
KOH	0.2
Water	Reduce to 45% TSC

The acrylate monomer, photoinitiator, potassium laurate, and water were homogenised using an emulsifier for 20 minutes and slowly added into the NR latex with gentle stirring. The KOH solution was added to the latex mixture to stabilise the pH of the mixture in the range of 9-11. The latex mixture was stirred continuously for 1 hour. It was then transferred into a reaction flask with an immersion well and then exposed to UV irradiation for 1.5 hours from a medium pressure mercury lamp with a current supply of 400W (250-350 nm wavelength) as shown in Figure1. The latex mixture was continuously stirred, and cold water was continuously circulated in the immersion well at a set temperature of 20°C to reduce the rate of evaporation. The control sample was prepared by diluting NR latex with distilled water to reduce TSC to 45% without exposure to UV irradiation. The control sample was labelled as NR, and UVNRL with acrylate monomer was labelled as NR/HDDA, NR/n-BA, and NR/TMPTMA.

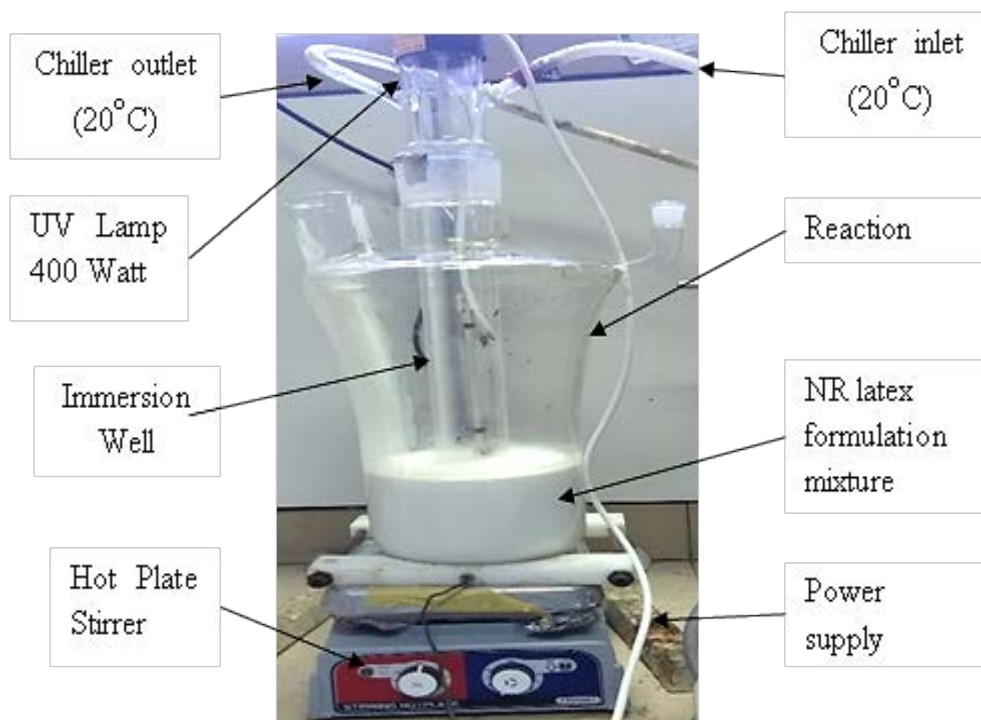


Figure 1: Lab-scale set-up of UV irradiation vulcanisation of NR latex using reaction flask with an immersion well, and UV lamp

Specimens for tensile testing were prepared using the coagulant dipping method. A glass plate was immersed in the 30% calcium nitrate solution, and placed in an oven at 100°C temperature to turn the mixture into a partial gel. It was then immersed in the UVNRL for the 30s. The wet gel of UVNRL was allowed to gel at 100°C for 1 minute, then leached in distilled water at 60°C for 5 minutes, and finally dried at 100°C. The UVNRL film was cut into seven dumbbell-shaped test pieces and subjected to tensile strength, Modulus@500% (M500), and Modulus@700 (M700) tests using the UTM SHIMADZU (50N) in accordance with ASTM D412(10) at a crosshead speed of 500mm/min until breakage. The median value was taken as the final result.

The UVNRL film was immersed in toluene until the swollen film's weight no longer increased, as the equilibrium state of rubber solvent was achieved. The swollen film was taken out, dabbed with a filter paper to remove the adhering solvent on the surface, and weighed. The film was then dried to a constant weight in an oven at 60°C, and cooled to room temperature in a desiccator, and weighed. The volume fraction (ϕ_r) of the rubber in the swollen rubber was calculated as

$$\phi_r = \frac{V_r}{V_r + V_s} \quad (1),$$

where V_r is the volume of dried rubber film, and V_s is the volume of solvent in swollen rubber film. The crosslink density of the [X]phy in mol/g was then calculated by using the Flory-Huggins equation,

$$[X]_{\text{phy}} = \frac{-\ln(1-\phi_r) - \phi_r - \phi \phi_r^{1/3}}{2\rho V_0 V_r^{1/3}} \quad (2),$$

where ρ is the density of NR, ϕ is the Flory-Huggins parameter for rubber-solvent interaction, and V_0 is the molar volume of solvent (Mullins, 1957).

An NR film's wettability with acrylate monomers was evaluated using the contact angle method at ambient open-air conditions. A contact angle was measured using Attention Theta Optical Contact Angle. At least ten separate measurements were made each time by placing a drop of an acrylate monomer on the pure NR film's surface. The data were averaged, and the measurement was reproducible to $\pm 2^\circ$. The adsorption of acrylate monomers by NR films was measured by immersing a piece of NR film with a known weight in the acrylate monomer for 1.5 hours. The film was then removed, and the excess acrylate monomer was blotted with lint-free paper. The weight of the swollen NR film with acrylate monomer was measured. The experiment was repeated at least three times with each acrylate monomer. The percentage adsorption of acrylate monomer into pure NR film was calculated. Specimens for FTIR Spectroscopy are used with NR, and UVNRL film. FTIR spectroscopy analysis was carried out using Bruker's Tensor II Platinum Attenuated total reflection spectrophotometer.

The thermogravimetric analysis (TGA) was carried out with a Perkin Elmer TGA-7 thermogravimetric analyser. Each sample's mass was 3-6 mg in the atmosphere filled with nitrogen gas. The samples were heated from 35°C to 500°C to record the thermogravimetric (TG), and differential thermogravimetric (DTG) curves. The NR and UVNRL films' thermal behaviours were

studied using a Mettler Teledo DSC 822 thermal analyser. The samples underwent a DSC scan from -60°C to 200°C at a heating rate of 10°C min⁻¹ in the nitrogen atmosphere. Each sample's mass was about 5.5mg with heat flow at 8.74 m/W. The glass transition temperature of each sample was taken at the initial change in the slope of the DSC curve's heat capacity.

3 Result and Discussion

Table 2 shows the tensile strength of samples formulated with acrylate monomer progressively increased around 51% to 59% compared to the tensile strength of NR. The NR/HDDA has the highest tensile strength followed by NR/n-BA and NR/TMPTMA. This indicated that UV irradiation to NR latex induced a chemical reaction with acrylate monomer. The crosslink density data showed that crosslink density increases with acrylate monomer addition. NR/HDDA had the highest crosslink density value followed by NR/n-BA, NR/TMPTMA, and NR. The crosslink density value indicated that there was chain entanglement in NR that may be due to physical crosslink, and chemical crosslink enhanced tensile properties. The UVNRL with acrylate monomer show increment in tensile strength due to chemical, and physical crosslink while for NR the crosslink value contributed from molecular chain entanglement.

Table 2: The M500, M700, tensile strength, and crosslink density of samples

Sample	M500 (MPa)	M700 (MPa)	Tensile Strength (MPa)	Crosslink density (mole/g)
NR	0.590	0.896	4.685	1.206 x 10 ⁻⁶
NR/n-BA	1.046	2.109	10.781	4.272 x 10 ⁻⁶
NR/HDDA	1.392	3.753	11.444	6.524 x 10 ⁻⁶
NR/TMPTMA	0.792	1.898	9.718	2.223 x 10 ⁻⁶

The effectiveness of crosslink between NR, and acrylate monomer depends on the acrylate monomer's reactivity and solubility. When a sufficient amount of monomer is unable to enter the inside of the NR particle, the crosslinking of NR cannot be accelerated even though the monomer's reactivity is high (Manaila *et al.*, 2014). The solubility test (monomer uptake by NR film), and wettability (contact angle of monomer on NR surface) of acrylate monomer with NR films are shown in Table 3. The n-BA had the highest absorption in NR film at a 960% weight increase from the initial weight while the percentage absorption of HDDA and TMPTMA increased by 38%, and 16%. Table 3 shows the contact angle of acrylate monomer drop on the NR film surface. Generally, if the value is less than 90°, the surface is hydrophilic, which means the monomer tends to wet the surface. If the value is more than 90°, it represents the surface's non-wetting characteristics. The lower contact angle indicates the high wettability of the monomer on NR particles' surface. It can be seen that n-BA had the highest absorption, and wettability than HDDA, and TMPTMA monomers. Unfortunately, the tensile property of NR/n-BA is slightly lower than NR/HDDA. The reactivity of acrylate monomers depends on the polyfunctional group in the present. Based on its chemical structure, TMPTMA consists of 3 polyfunctional groups attached with the methyl group while HDDA contains two polyfunctional groups, and n-BA has one polyfunctional group.

Interestingly, the tensile test results showed that the NR/TMPTMA has the lowest tensile properties, and crosslink density. This result is expected despite that TMPTMA has three polyfunctional groups with the highest reactivity but at the same time reduce the level of diffusion of TMPTMA into NR particles. Thus the TMPTMA tends to react to each other, and form poly-acrylate TMPTMA. The existence of poly-acrylate TMPTMA is supposed to contribute to better tensile properties due to physical crosslink. Unfortunately, this did not happen as the hard segment of poly-acrylate TMPTMA in the branched network formed from the tertiary radical reaction has a very stable bonding. These bonding reduced the flexibility for chain alignment during extension, causing poor crystallisation, and lower tensile strength (Hansupalak *et al.*, 2016). The NR/HDDA has the highest tensile properties due to two polyfunctional groups in HDDA, and the ability to wet the NR latex particle partially. The ability of the HDDA monomer to migrate into NR latex particle and the position of the polymerised double bond at both sides in the HDDA chemical structure contributed to an effective crosslink formation. The first thing to point out is the tensile strength, and the crosslink density of NR/HDDA, and NR/n-BA, as shown in Table 2.

Table 3: The monomer uptake, and contact angle of monomer on NR film

Monomer	Monomer uptake by NR film (%)	Contact angle
n-BA	960	45±1.9°
HDDA	38	57±2.0°
n-BA	17	63±1.9°

There was only a 5.79% difference in tensile strength value between NR/HDDA and NR/n-BA. Comparing the crosslink density value differences, NR/HDDA was 34.52% higher than NR/n-BA. This indicated that physical crosslink contributed significantly to the tensile strength of NR/n-BA while the chemical crosslink contributed more to the tensile strength of NR/HDDA. This proves that the HDDA monomer was effectively crosslinked to NR. This result is expected as HDDA is a di-functional monomer in which the possibility to crosslink with NR is higher than n-BA. The formation of branched poly-acrylate HDDA increased the molecular chain entanglement, and the bonding from secondary radical reaction allows a reasonable degree of flexibility for chain orientation during extension. While n-BA is a monofunctional monomer, it has a low possibility to crosslink with the NR chain. However, there is a high tendency for the n-BA to form poly-acrylate n-BA grafted to the NR polymer chain. The grafted NR with poly-acrylate n-BA increased the degree of chain entanglement contributing to physical crosslink. The poly-acrylate HDDA, which has a low T_g at -53°C , provides more flexibility in chain orientation and made it easy to align for crystallisation during extension. This factor results in the marginal difference in tensile strength between NR/n-BA, and NR/ HDDA. The UVNRL exhibited an FTIR spectrum similar to an NR spectrum, as shown in Figure 2. The peaks appeared in NR at absorption bands around $2954\text{--}2856\text{cm}^{-1}$ bands are attributed to C-H stretching in $-\text{CH}_3$, and $-\text{CH}_2-$, 1666 cm^{-1} band is due to C=C stretching, the 1445 cm^{-1} bands are related to bending in $-\text{CH}_3$ in $-\text{CH}_2-$, and the band absorption at

1375 cm^{-1} is due to C-H bending in $-\text{CH}_3$. The absorption peak at 1735 cm^{-1} , 1745 cm^{-1} , 1747 cm^{-1} , 1604 cm^{-1} , 1576 cm^{-1} , 1551 cm^{-1} , and 1532 cm^{-1} occurred only in the spectrum of the NR films made from a latex mixture with acrylate monomer. These peaks are associated with the C = O group in the acrylate monomer, possibly with the ester group, and the ketone group in the photoinitiator. This peak did not appear in the spectrum of NR as it has not been added with any acrylate monomer, and a photoinitiator. This indicates that acrylate monomer, and photoinitiator molecules in these film samples were strongly crosslinked to the NR polymer chain (Wiroonpochit *et al.*, 2017).

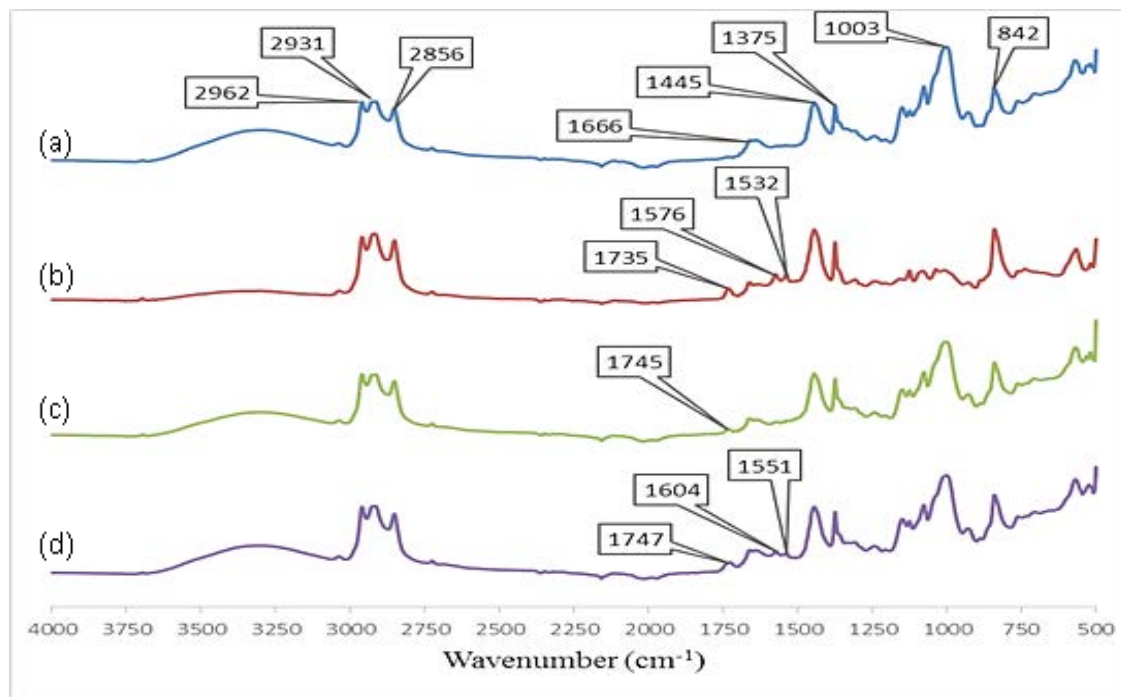


Figure 2: FTIR spectrum of (a) NR, (b) NR/HDDA, (c) NR/n-BA, and (d) NR/TMPTMA

According to Figure 3, the initial thermal degradation of NR starts at 280°C, and below this temperature, solid rubber is in a stable form. This shows that the NR may maintain its molecular weight up to 280°C in the absence of oxygen. About 18% of weight loss occurred from 280-350°C. Rapid changes in degradation occurred in the intervals between 350-470°C. Above 350°C, the volatilisation became rapid, and complete decomposition occurred in about 44 minutes. The DTG curves of NR showed that major degradation occurs at 370°C, and about 2% of weight residue remained above 470°C. The solid is insoluble and may be likened to cyclised rubber. The initial degradation temperatures of sample NR/HDDA, NR/n-BA, and NR/TMPTMA were 256°C, 242°C, and 217°C, respectively. The initial degradation temperature of samples with acrylate monomer was lower compared to NR. However, rapid volatilisation of samples occurred at temperatures close to the NR at 350°C, 344°C, and 342°C for samples NR/HDDA, NR/n-BA, and NR/TMPTMA, respectively. The same major thermal degradation of samples occurred at temperatures 373°C, 369°C, and 370°C for NR/HDDA, NR/n-BA, and NR/TMPTMA. Figure 3 shows the TG, and DTG curves of NR, and samples' thermal degradation with different acrylate monomer. It can be seen that the TG curve is a smooth curve with one turn, and there is only one obvious peak on the DTG

curves, indicating that the thermal degradation of each sample in nitrogen gas was simple, and a one-step reaction (Rao, and Johns, 2008).

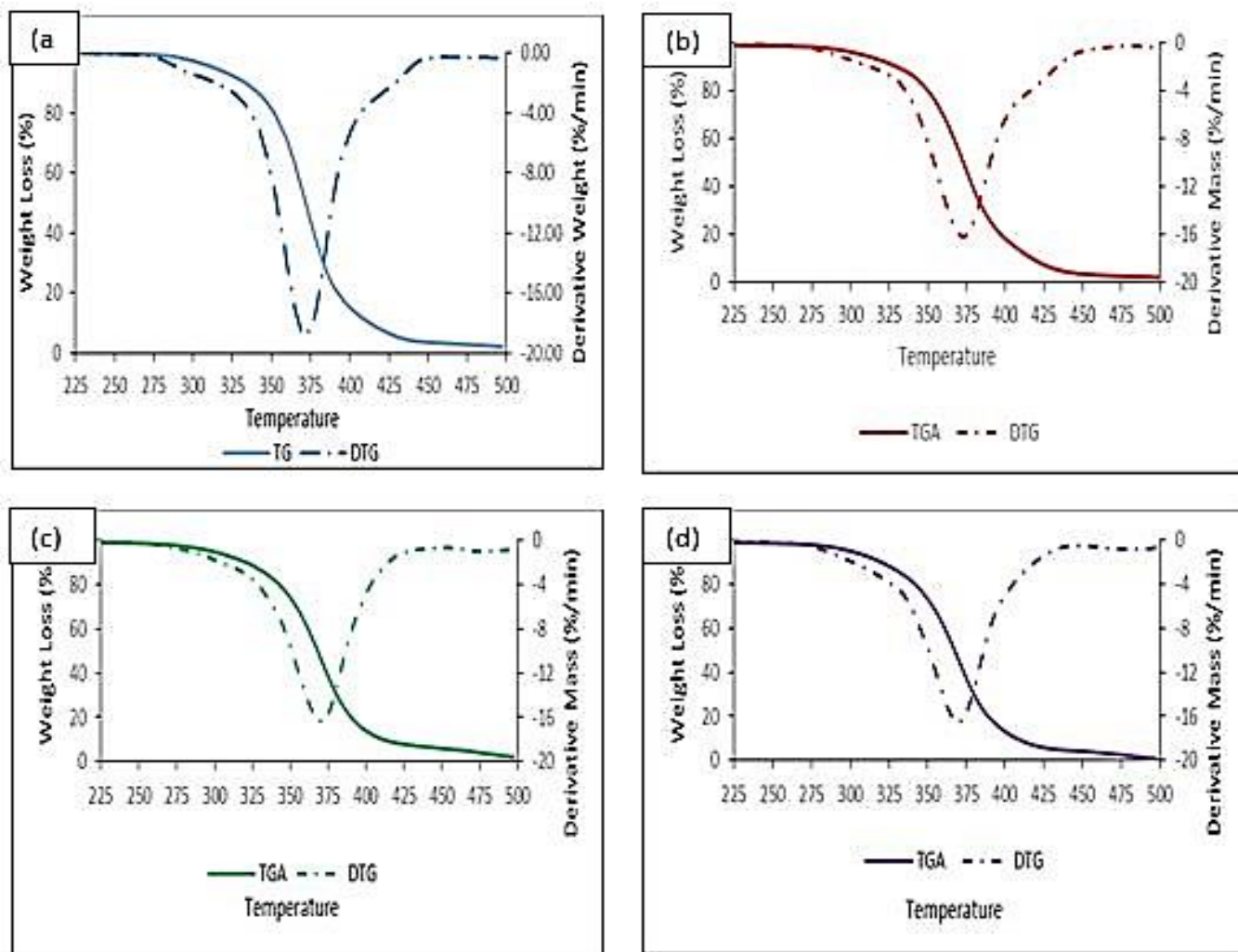


Figure 3: Thermogravimetric curves, and differential thermogravimetric curves of thermal degradation of (a) NR, (b) NR/HDDA, (c) NR/n-BA, and (d) NR/TMPTMA.

The DSC analysed the thermal behaviour of the NR, and UVNRL with different acrylate monomers. Table 4 shows the T_g of NR/HDDA, NR/n-BA, NR/HDDA, and NR film as reference. The T_g of NR was found to be at -66.34°C . The NR had no chemical crosslink, and thus the chains gain a stiffening effect due to the existence of the methyl group in the poly-isoprene chain (Loadman, 1985). UVNRL with acrylate monomer showed an increment in T_g in NR/HDDA < NR/n-BA < NR/TMPTMA, and the increment in tensile strength, and M700 in the reverse sequence. The introduction of chemical crosslinks in NR hinders the NR polymer chain's segmental mobility. The denser the network is, the shorter the molecular segments between crosslinks, thus requiring higher thermal energy to enable the rubber polymer segment's molecular mobility. The addition of HDDA, and n-BA monomer into NR latex causes the formation of chemical crosslink as shown by the increase of T_g at 0.87%, and 1.41%, and shows an increase in tensile strength of 11.444 MPa, and 10.781 MPa, respectively. The addition of TMPTMA monomer in NR latex results in an increase

in T_g , which is bigger than other formulations. However, the tensile strength of NR/TMPTMA was the lowest compared to NR/HDDA, and NR/TMPTMA. This shows that TMPTMA did not effectively crosslink with NR but tends to form crosslinked polyacrylate TMPTMA that could contribute to the hard segment, and chain entanglement in NR molecular chain, thus an increase of the T_g value.

Table 4: The glassy transition temperature (T_g) of NR, and UVNRL samples

Sample	NR	NR/HDDA	NR/n-BA	NR/TMPTMA
T_g	-66.34	-65.76	-65.40	-64.95

4 Conclusion

NR latex can be vulcanised using the UV irradiation method as demonstrated in this work. The highest tensile strength can be achieved by NR/HDDA latex at 11.444 MPa and with a crosslink density of 6.254×10^{-6} mole/g. However, further studies on the irradiation time should be carried out to determine the optimum tensile properties. It can be concluded that chemical and physical crosslinks contribute to tensile strength enhancement. The formation of poly-acrylate n-BA grafting and crosslinked polyacrylate HDDA in NR molecular chains can enhance the tensile properties. The reactive tri-methacrylate group leads to poly-acrylate TMPTMA rather than crosslinking with NR. The addition of HDDA into NR latex can produce effective crosslink, which enhances the tensile, and stable thermal properties.

5 Availability of Data, and Material

Data can be made available by contacting the corresponding authors.

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