

# Influences of Accelerators on the Structures & Properties of Nitrile Butadiene Rubber

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## Abstract

The effect of incorporation of different types of accelerators i.e. TMTD, TMTM, DOTG and NPV/C on the properties of NBR rubber was investigated. Without any organic accelerator it required long time to cure; tensile properties showed the least value but elasticity was the highest one and ageing properties were poor. Thiuram accelerators gave very fast cure; good tensile properties; high crosslinking density reflected on its resistance to organic solvents and gave the best ageing characteristics. Di -o- tolyl guanidine and vulcacit NPV/C thiourea gave slow onset of cure; long scorch time; high tensile and resilience properties, DOTG gave better ageing properties than NPV/C accelerator. The effect of accelerator type on compounds viscosity corresponded well with the melting point of the accelerators. NBR had high resistance to oil, in toluene and benzene TMTD and TMTM gave the best resistance. Elucidation of the structure of vulcanizates was determined by ATR- FTIR technique.

**Keywords:** Acrylo nitrile butadiene rubber, Accelerators, Mechanical properties, Ageing, ATR- FTIR

## 1. Introduction

The discovery of organic accelerators by Oenslager in 1906 greatly increased the rate of vulcanization to make mass production possible; these accelerators also permit wide choice of method and temperature of processing and vulcanization. Thiuram accelerators are suitable for all conventional curing processes (press, steam, hot air). They are also used to cure mixes intended for transfer moulding, injection moulding, or continuous vulcanization. If thiuram are used alone attention should be paid to the tendency of the mix to revert: the higher the curing temperature, the poorer the mechanical properties of the vulcanizates. Guanidine like other basic accelerators, produce polysulphidic crosslinking in the rubber matrix this type of cross- linkage results in a high tensile strength and excellent resilience properties Guanidine or the many different combination in which they are used are suitable for all curing processes. DOTG is a basic slow accelerator with a relatively slow onset of vulcanization and hence is preferred in the vulcanization of thick-wall. Because of its high activity as across linking agent and owing to the fact that it gives a relatively favorable flow time/curing time ratio vulcacit NPV/C is more important than any of the thiourea derivatives.

## 2. Materials and methods

### 2.1 Materials and Mixing & Vulcanization Procedures

This was accomplished on a laboratory calender with two horizontal cylinders (200mm. diameter and working length of 400 mm) the revolving speed of the front slower cylinder was 16 r.p.m. and hind roll speed 20 r.p.m. The hollow cylinders were cooled by means of flushing water in order to regulate temperature not to exceed 60 °C during different stages of mixing. The mix kept at room temperature for 24 h before testing.

## 2.2 Cure Characteristics

Cure characteristics were studied using Moving Die Rheometer (Zwick 4308) according to ASTM D 2240-93. Samples (4 g) of the respective compounds were tested at the vulcanization temperature (150 °C). The use of this cure meter and standardized values read from the curve is specified in ASTM D 2084. Some of these recommended values that are important to know for this study are

$M_L$ : Minimum torque in N.m or lbf.in.

$M_H$ : Maximum torque where curve plateaus are in N.m or lbf.in.

$t_x$ : Minutes to  $x\%$  of torque increase,  $t_x$  = minutes for torque value equivalent to In rubber terminology,  $t_{90}$  is defined as “optimum cure time”  $M_L + x(M_H - M_L)/100$ .

## 2.3 Mooney Viscosity

Mooney viscometer is probably the most widely used method for measuring the quality of natural rubber. This viscometer was invented by Melvin Mooney, US Rubber Company, in the 1930s and is now used to measure the viscosity of both natural and synthetic rubber worldwide. The Mooney viscosity results are reported in arbitrary Mooney Units (MU) which is based on torque as defined by ISO 289 and ASTM D1646. The Mooney viscosity was determined by using a Monsanto automatic Mooney viscometer (MV 2000) at 120 °C. The testing procedure was conducted according to the method described in ASTM D 1646-94.

## 2.4 Vulcanization Process

Rubber sheets (3 mm thick) were compression moulded at 150 °C with force of 10 MPa using a hot press (Atsfaar-Vignate-Mi – Italy) according to respective cure times,  $t_{90}$ , determined with the (Zwick 4308).

## 2.5 Tensile Properties

Dumb-bell-shaped samples were cut from the moulded sheets according to ASTM D 412. Tensile test were performed at a cross-head speed of 500 mm/min. Tensile testing was carried out with (universal testing material Zwick 1425)

## 2.6 Hardness Properties

Samples of at least 12 mm thickness. The measurement was according to ASTM D2240 using Durometer of Model 306L Type A. The unit of hardness is expressed in (A Shore).

## 2.7 Rebound Resilience

Rebound resilience is a very basic form of dynamic test in which the test Piece is subjected to one half-cycle of deformation only. The strain is applied by impacting the test piece with an indenter which is free to rebound after the impact. Rebound resilience is defined as the ratio of the energy of the indenter after impact to its energy before impact expressed as a percentage. The test is performed by (digi test Ruckprall 567 BJ.06)

## 2.8 Swelling Study

Swelling was studied in toluene, benzene and processing oil 37; according to ASTM D 471-79.

Cured test pieces of the compounds of dimension 30×5×2 mm were weighed using an electrical balance and this was taken to be the initial weight,  $M_1$ . Calculation of the change in mass was as follows:

$$\text{Swelling percentage} = [(M_2 - M_1) / M_1] \times 100$$

Where  $M_1$  is the initial mass of specimen (g) and  $M_2$  is the mass of specimen (g) after immersion, the network absorbs a certain amount of liquid which depends strongly on the molecular weight of this liquid and the degree of cross linking of the polymer. A high degree of swelling indicates that the rubber is not suitable for use in that environment.

## 2.9 ATR-FTIR Measurements

Were run with a JASCO instrument (FT/IR-6100typeA, accessory ATR PRO410-S) in the following conditions: wave number range: 400-4000  $\text{cm}^{-1}$ ; aperture setting: 3.5mm; scanner velocity: 2.2 kHz; background scan time: 32 sec; resolution 4  $\text{cm}^{-1}$ , beam splitter: KBr; angle of incident radiation: 45°. After recording, the ATR-FTIR spectra were converted into transmission FTIR spectra. The plate samples (6 x 6 mm) were simply posed on the sampling stage, in intimate contact with the optical element, a hemi cylindrical prism of SeZn (called Internal Reflection Element (IRE)). The incident radiation arrives onto the sample with a certain angle usually, between (30° and 60°) to the normal of the sample plane. Then, the reflected (beam is collected by a mirror, which focuses the reflected radiation onto the detector.

## 2.10 Aging of Rubber

### 2.10. 1 Thermal Ageing

The air ageing was conducted in an air oven (Chirana STE 39/11, Chekoslovakia) at various times at 90 °C the aged samples were allowed to rest at room temperature for 24 h and the physical properties were then measured.

### 2.10. 2 Weathering Ageing (Xenon Test)

Experiments were carried out in a modified Q-Panel QUV<sup>®</sup> Climatic test cabinets introduced by (Heraeus150 S original HANAU, Germany) Equipment can simulate extreme By accurately controlling both humidity and temperature, the HC test cabinets can subject samples to climatic conditions they would otherwise experience over years of exposure. Weathering device equipped with UVA-340 lamps. Components and systems are subjected to extremes of high and low temperature using a variety of thermal chambers, in which temperature and humidity conditions can be programmed and applied to the test parts in cycles. We routinely test over conditions of standard high temperature range of 177°C to 190°C (350°F to 375°F). Single-stage refrigeration systems typically can pull the temperature in the chamber down to - 34 °C. By bubbling air through the water bath (maintained at 45 °C), the relative humidity could be maintained at about 45%. Samples with formulations shown in Table 2 were cut to (1.3 × 3.2 cm) and adhered to the center portion of a 6" × 12" aluminum panel using silicone RTV in a single 3 × 9 array. In this series, the samples were exposed to U.V for 200 hours. During this period the samples were subjected to artificial condition of rain, wind and sun heat in an alternative manner for a half minute for each cycle.

## 3. Results and Discussion

### 3.1 Rheological Properties

Cure characteristics of the mixtures are strongly influenced by the accelerator type. It is widely known that the torque difference is directly proportional to the state of cure or the total crosslink density. The results in figure 1 reveal that the mixture which didn't contain any type of organic accelerators showed a longer scorch and optimum cure time than other mixtures it provide an unsatisfactory state of cure. To achieve sufficiently high crosslink density either TMTD or TMTM seems to be a more appropriate choice. They are ultra accelerators giving faster cure time Compared with DOTG, NPV/C, which is classified as a delayed action accelerator, give longer scorch which is favorable and longer optimum cure time. Generally, in rubber technology and processing, rubber manufacturers always prefer a vulcanization systems that can give low cure time ( $t_{90}$ ), high scorch time and high cure rate as a result of processing advantages in time.

### 3.2 Viscosity

Figure 2 represents the effect of accelerator type on Mooney viscosity. Even though only a small amount of accelerator it noticeably influenced the Mooney viscosity of the compound. Obviously, the results revealed that the compound viscosity depends on the accelerator type i.e., viscosity of the compound tended to increase from TMTM, DOTG, TMTD to NPV/C. It could be observed that the effect of accelerator type on compound viscosity corresponded well with the melting point of the accelerators (the melting points of TMTM, DOTG, TMTD and NPV/C are 105, 167, 175 and 195 oC, respectively). The higher the melting point of the accelerator, the greater the Mooney viscosity of the compound. The mixture which didn't contain any organic accelerator showed the highest viscosity this was due to the lack of formation of crosslinks. In addition to the compound viscosity the torque increased for a few seconds to a maximum value, then decreased very rapidly to reach a minimum value LM) in many cases, and lastly rises again at varying speeds to a plateau (VR or ML(1+4)120) reached after 2–4 min depending on the sample. It is this last value (VR or ML (1+4)120). In general, stress relaxation indicates the rate of decay i.e. faster rate of decay indicates that the polymer has a lower elastic quality. Mixture without any organic accelerator had the highest stress relaxation value and thus was more elastic.

### 3.3 Physico-mechanical Properties

Figure 3 illustrates the effect of accelerator type on physico-mechanical properties of the mixtures. The results clearly reveal that mixture without organic accelerator has a lower tensile property compared to mixtures which contain organic accelerators, but elasticity was the highest one, it also gives the highest elongation at break value. These physico-mechanical properties reflect that mixture B1 has a lower crosslinking density than other mixtures. Incorporation of organic accelerators increased tensile strength but elasticity tended to decrease. Di -o- tolyl guanidine is a basic slow accelerator with a relatively low modulus, it gave high tensile strength, elongation at break and resilience properties, vulcacit NPV/C showed also high tensile strength value but lower than DOTG and gave the best resilience properties among organic accelerators. Ultra accelerators TMTD, TMTM gave better

tensile properties than mixture without accelerator but lower than DOTG and NPV/C which gave higher tensile strength and elasticity.

### 3.4 Spectroscopic Measurements

In mixture B1 Very weak signals for the range  $4000-3200\text{ cm}^{-1}$  for the samples the vibrations responsible for bands in this region are O-H stretching organic acids and phenols it is strong at  $3266.82\text{ cm}^{-1}$ . we assume that the activator (Stearic acid) and the antioxidant irganox1010 are completely incorporated in the polymer blends, the same assumption should be considered in the case of the other polymer additives Thus, the C=O group of saturated aliphatic carboxylic acids absorbed in the peak at  $1743\text{ cm}^{-1}$ , peak at  $2917.77\text{ cm}^{-1}$  corresponded to C-H stretch, peak at  $2231.24\text{ cm}^{-1}$  was attributed to nitrile group in krynak, peak at  $1595\text{ cm}^{-1}$  represents aromatic system due to processing oil and anti oxidant,  $\text{CH}_2$  scissoring bend is represented by peak at  $1430\text{ cm}^{-1}$ , C-O Ether linkages and Hydroxyl appear at  $1081\text{ cm}^{-1}$ , Carbon double bond (1,4 trans bonding,  $957\text{ cm}^{-1}$ ), the (S-S) linkage indicated by absorbance at  $559\text{ cm}^{-1}$

By introducing TMTD accelerator C-N stretch tertiary amine appeared by absorption at  $1200\text{ cm}^{-1}$ , in addition to other distinguishing peaks. Peak at  $2231.24\text{ cm}^{-1}$  was attributed to nitrile group in krynak, peak at  $1742\text{ cm}^{-1}$  indicated the presence carbonyl peak, the absorbance of the carbon – carbon double bond (1,4 trans bonding,  $957\text{ cm}^{-1}$ ) S-S stretching peak appeared at  $550\text{ cm}^{-1}$ .

By introducing TMTM accelerator C-N stretch tertiary amine appeared by absorption at  $1209\text{ cm}^{-1}$ , peak at  $2231.24\text{ cm}^{-1}$  is attributed to nitrile group in krynak, peak at  $1743\text{ cm}^{-1}$  indicated the presence of carbonyl peak the absorbance of the carbon – carbon double bond (1,4 trans bonding,  $958\text{ cm}^{-1}$ ),

S-S stretching peak appeared at  $556\text{ cm}^{-1}$

By introducing DOTG accelerator peak at  $1537\text{ cm}^{-1}$  indicated the presence of double bond aromatic, the presence of C-N stretch in aromatic secondary amine indicated by peak at  $1256\text{ cm}^{-1}$ , the presence of NH stretch indicated by peak at  $3358\text{ cm}^{-1}$ ,  $\text{NH}_2$  wag similar bending mode is present in secondary amine but not on tertiary ones represented by peak at  $795\text{ cm}^{-1}$ , peak at  $1537\text{ cm}^{-1}$  due to C-N (amide), the absorbance of the carbon – carbon double bond (1, 4 trans bonding,  $955\text{ cm}^{-1}$ ), S-S stretching peak appeared at  $556\text{ cm}^{-1}$

By introducing NPV/C accelerator N-H stretch appeared at  $3317\text{ cm}^{-1}$ , C-H stretch appeared at  $2849\text{ cm}^{-1}$ , carbonyl bond appeared at  $1742\text{ cm}^{-1}$ , peak characteristic for the cure agent NPV/C indicated N-C=S group gave IR spectra at  $1576\text{ cm}^{-1}$ , C-N stretch at  $1254\text{ cm}^{-1}$ , C=S thio carbonyl group indicated by absorption at  $1077\text{ cm}^{-1}$ ,  $\text{CH}_2$  scissoring bend at  $1428\text{ cm}^{-1}$ ,  $\text{NH}_2$  wag similar bending mode is present in secondary amine but not on tertiary ones was represented by peak at  $794\text{ cm}^{-1}$ , The absorbance of the carbon – carbon double bond (1, 4 trans bonding,  $957\text{ cm}^{-1}$ ), S-S stretching peak appeared at  $540\text{ cm}^{-1}$ .

### 3.5 Results of Equilibrium Swelling in Toluene, Benzene and Processing Oil.

The relationship between the swelling ratio and accelerator type is illustrated in Fig. 4. NBR generally has high resistance to oil however the addition of accelerators improved this feature. As it is widely accepted that the swelling ratio is inversely proportional to crosslink density in toluene and benzene TMTD and TMTM gave a better crosslinking density than other accelerators which reflected on its smaller swelling value than other accelerators used. Mixture that didn't contain organic accelerator showed a weak resistance to both benzene and toluene.

### 3.6 Change in Physico-mechanical Properties of Aged Samples at $90\text{ }^\circ\text{C}$ (Geer Ageing) for Different Periods According to Different Types of Accelerators

#### 3.6.1 Change in Tensile Strength ( $\text{Kg}/\text{cm}^2$ )

Upon thermal ageing at  $90\text{ }^\circ\text{C}$ , all the systems of vulcanization showed a further increase in tensile strength. And then decreased. This was due to the formation of additional crosslinks during thermal ageing Rubber samples are usually cured in industry only to 90%. The allowance of 10% is generally kept to accommodate the introduction of cross links in the matrix during service. When samples were subjected to thermal ageing at  $90\text{ }^\circ\text{C}$ , the formation of additional crosslinks got accelerated. The incorporation of accelerators fastened this process. However, the tensile strength of all samples then decreased due to the degradation of cross links. The mixture which didn't contain any accelerator gave a weak resistance to thermal ageing due to the slower of formation additional crosslinks than other mixtures. The mixture which contained slow accelerators DOTG gave a better resistance to thermal ageing than NPV/C accelerator. Ultra accelerators TMTD, TMTM showed the best ageing characteristics.

### 3.6.2 Change in Elongation at Break %.

The effects of different crosslinking systems on elongation at break of un-aged and aged samples are represented below in figure 11. It had been found that the elongation at break of the vulcanisates decreased due to thermal ageing. The decrease in elongation at break could be attributed to the weakening the matrix after thermal ageing.

### 3.6.3 Change in Modulus at 200%

The modulus increased after ageing at 90 °C. This was probably due to the formation of additional cross links. The modulus was found to increase with the periods of subjecting the samples to accelerated ageing till 250 hours.

### 3.6.4 Change in Hardness (Shore)

The hardness was found to increase with the periods of subjecting the samples to accelerated ageing till 250 hours.

### 3.6.5 Change in Elasticity %

The elasticity was deteriorated for all the samples and the deterioration increased with increasing the period of subjection to accelerated ageing.

### 3.7 Deterioration in Physico-mechanical Properties of NBR after 200 Hours Using Xenon Apparatus (Weathering Test Results)

It was found that mixture B1 gave the least resistance to U.V radiation, rain and hot air produced by xenon apparatus. Introducing of different accelerators enhanced rubber resistance to weathering ageing TMTM and TMTD gave the best results however TMTM was better than TMTD also DOTG was better than NPV/C accelerator. The deterioration effect of physico-mechanical properties are represented in Figure 15. It had been found that the tensile strength and elongation at break of the vulcanisates decreased due to weathering ageing. The decrease in tensile strength and elongation at break could be attributed to weakening of the matrix. The modulus was found to increase with the periods of subjection to weathering ageing. The hardness was found to increase with the periods of subjecting the samples to accelerated weathering test. The elasticity was deteriorated for all the samples

## 4. Conclusions

Mixture which didn't contain any organic accelerators required long time to cure giving lower tensile properties than other mixtures and the least ageing characteristic due to the slow formation and weakness of cross links. Incorporation of Thiuram accelerators i.e. TMTD and TMTM gave very fast cure which is favorable. Thiuram cures gave an excellent vulcanization plateau with almost no reversion, thus gave a mass production and a high crosslinking density reflected on its resistance to organic solvents and also gave the best resistance to thermal and weathering ageing. Di -o- tolyl guanidine is basic slow accelerator with a relatively slow onset of cure, high tensile strength and resilience & good ageing properties. Vulcavit NPV/C is more important than any of the thiourea derivatives with a relatively slow onset of cure and long scorch time giving high tensile strength, elongation at break and elasticity but low modulus. Viscosity tended to decrease by incorporation of organic accelerators this was due to the start of formation of crosslinks. It could be observed that the effect of accelerator type on compound viscosity corresponded well with the melting point of the accelerators. NBR generally has high resistance to oil however the addition of accelerators improved this feature. In toluene and benzene TMTD and TMTM gave a better crosslinking than other accelerators which gave a higher swelling value, mixture that didn't contain accelerators showed a weak resistance to both benzene and toluene. FTIR investigation elucidated the structure of accelerators incorporated and functional groups of compounding ingredients incorporated in the mixtures.

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Table 1. Details of materials

Material.	Ingredients.	Sources.
Elastomer.	Acrylo nitrile butadiene rubber ( 34% nitrile content)	Malaysia
Filler.	High Abrasion Furnace Carbon black (HAF).	Degussa W. Germany.
Accelerators	Tetra methyl thiuram disulphide (TMTD) Tetra methyl thiuram mono sulphide(TMTM) Di-o-tolyl guanidine (DOTG) Ethylene thiourea of 2- mercapto imidazoline (NPV/C)	Chemapol CSSR. Chemapol CSSR. Chemapol CSSR. Chemapol CSSR.
Activators.	Zinc oxide Stearic acid	Chemapol CSSR. Chemapol CSSR.
Softeners.	Processing oil (37)	Misr Petroleum.
<i>Anti oxidant</i>	Anti oxidant irganox 1010 Tetrakis [methylene (3, 5-di-t- butyl-4-hydroxyhydrocinnamate)] methane	Chemapol CSSR.

Table 2. Structure of NBR, accelerators and antioxidant irganox1010

Name	Symbol	Structure of Compound
acrylonitrile-butadiene rubber	NBR	<p>The structure shows a copolymer chain with two repeating units: a butadiene unit (CH<sub>2</sub>-CH=CH-CH<sub>2</sub>) and an acrylonitrile unit (CH<sub>2</sub>-CH(C≡N)).</p>
Tetra methyl thiuram di sulphide	TMTD	<p>The structure shows a central S-S bond connected to two carbon atoms, each double-bonded to a sulfur atom and single-bonded to a nitrogen atom. Each nitrogen atom is bonded to two methyl groups (CH<sub>3</sub>).</p>
Tetra methyl thiuram monosulfide	TMTM	<p>The structure shows a central sulfur atom bonded to two carbon atoms, each double-bonded to a sulfur atom and single-bonded to a nitrogen atom. Each nitrogen atom is bonded to two methyl groups (CH<sub>3</sub>).</p>
Di-o-tolyl guanidine	DOTG	<p>The structure shows a central guanidine group (C(=NH)NH<sub>2</sub>) bonded to two ortho-tolyl groups (2-methylphenyl rings).</p>
Ethylene Thiourea; 2-Thioxoimidazolidine	NPV/C	<p>The structure shows a five-membered ring containing two nitrogen atoms and one sulfur atom, with a double bond to a sulfur atom (thio group).</p>
<i>Tetrakis [methylene (3,5-di-<i>t</i>-butyl-4-hydroxyhydrocinnamate)] methane</i>	Irganox 1010	<p>The structure shows a central carbon atom bonded to four hydrocinnamate chains. Each chain consists of a 3,5-di-<i>t</i>-butyl-4-hydroxyphenyl group connected to a propyl chain ending in a methyl group.</p>

Table 3. Compounding ingredients incorporates in NBR during Vulcanization according to different types of accelerators

Composition (phr)	B1	B2	B3	B4	B5
NBR	100	100	100	100	100
Zinc oxide	5	5	5	5	5
Stearic acid	1	1	1	1	1
Anti oxidant irganox 1010	1	1	1	1	1
Carbon black (HAF)	45	45	45	45	45
Processing oil (37)	5	5	5	5	5
Sulphur	2.5	2.5	2.5	2.5	2.5
TMTD	-	1	-	-	-
TMTM	-	-	1	-	-
DOTG	-	-	-	1	-
NPV/C	-	-	-	-	1

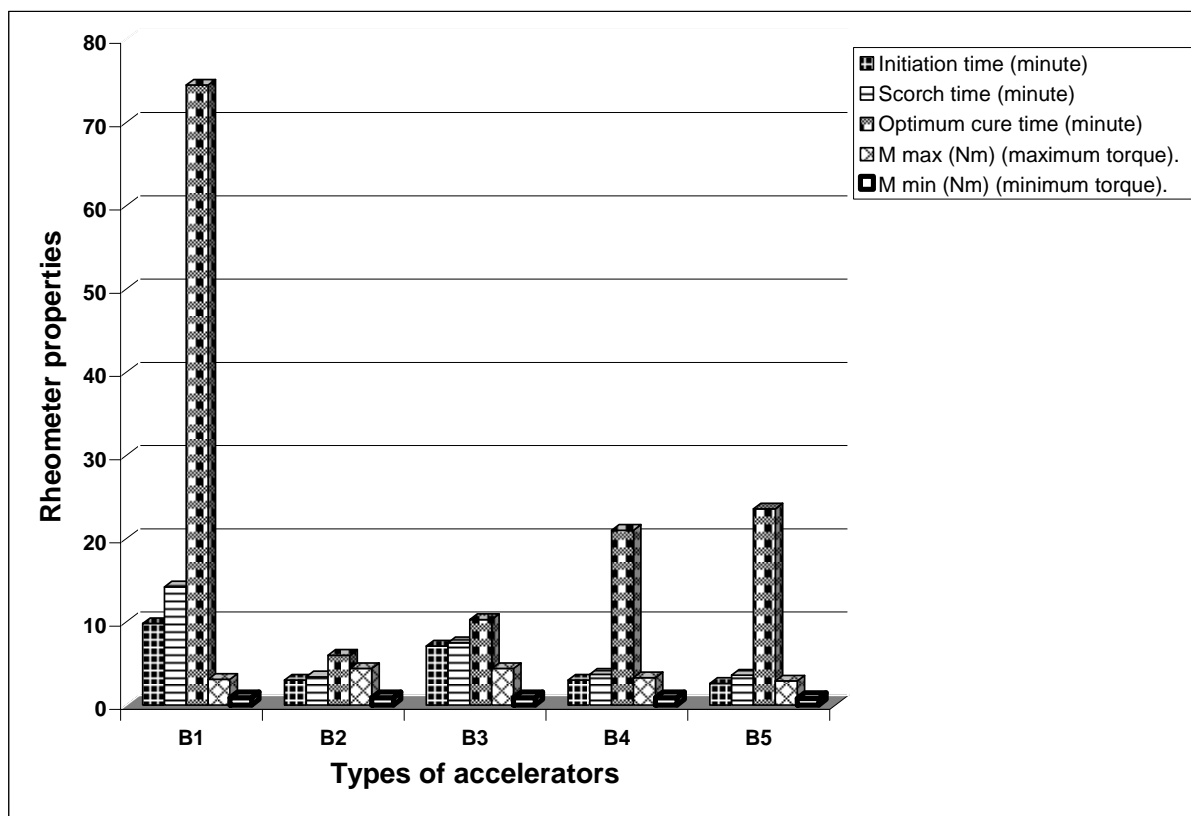


Figure 1. Effect of accelerator type on Rheological properties of NBR



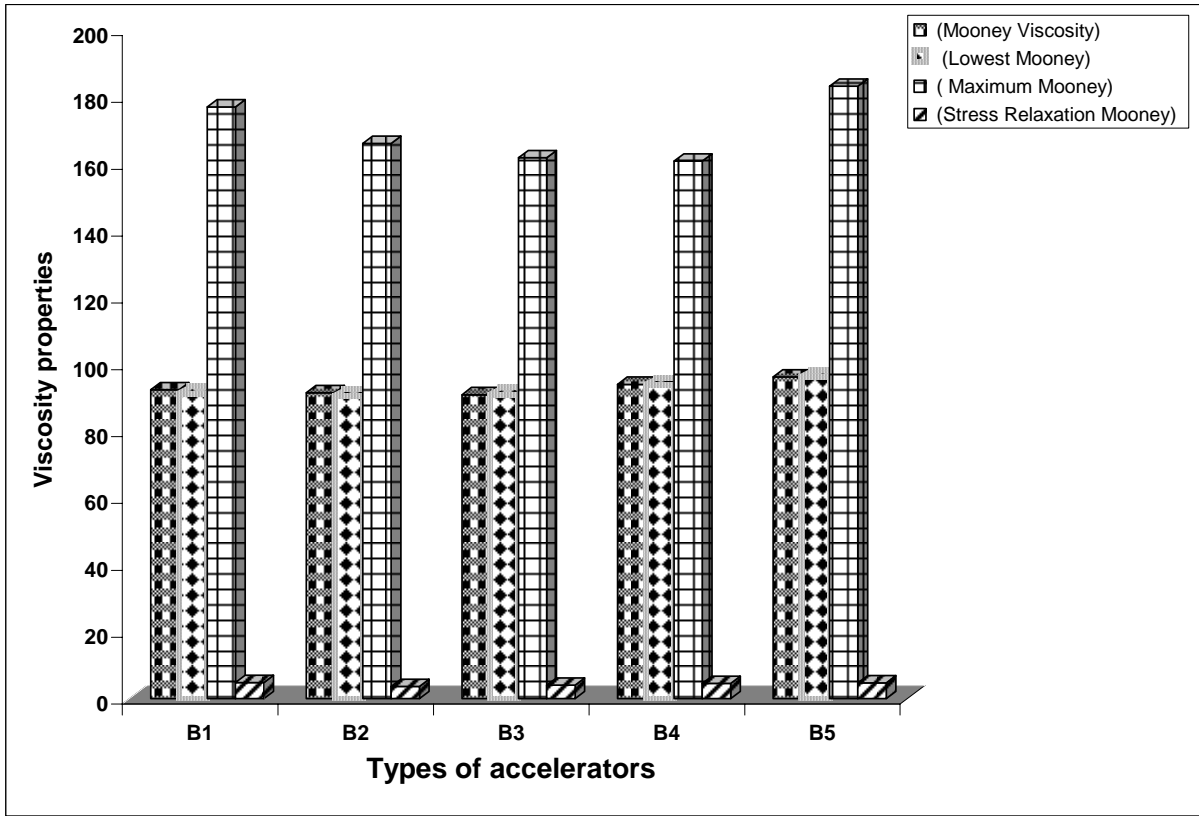


Figure 2. Effect of accelerator type on Mooney viscosity of NBR

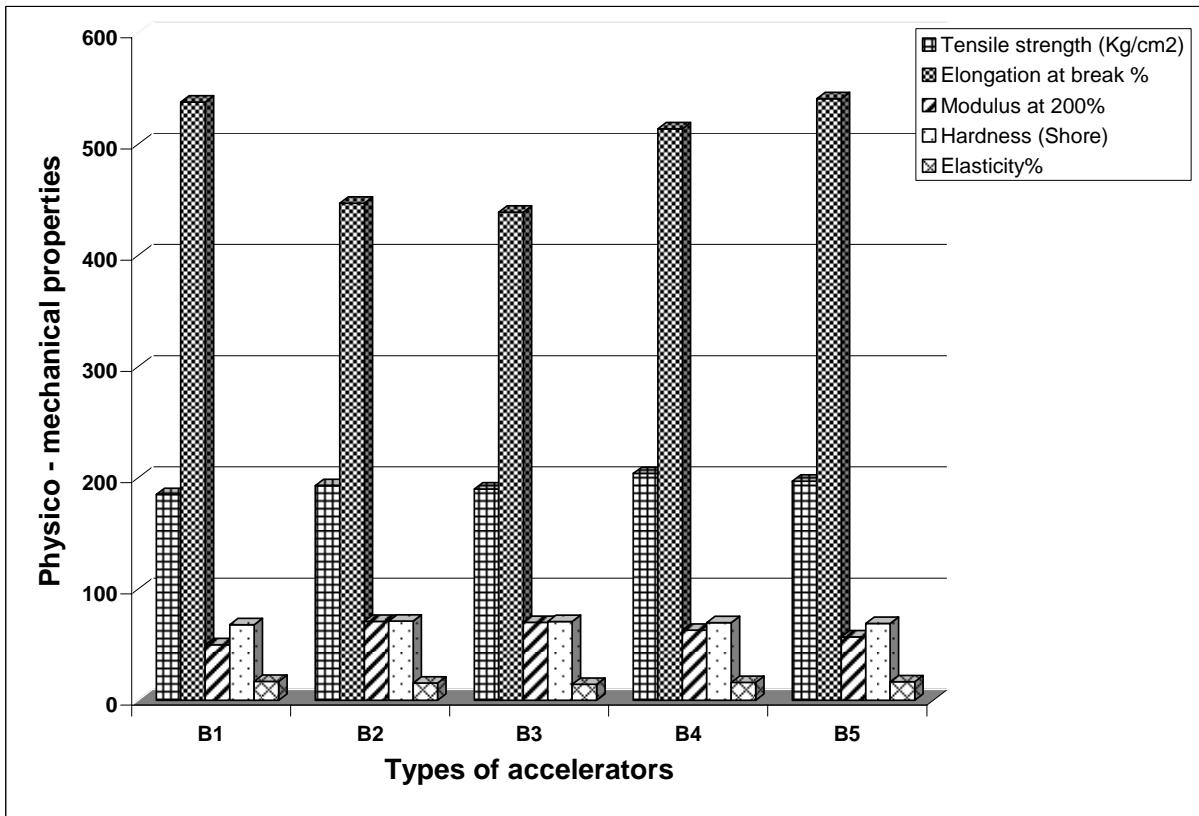


Figure 3. Physico-mechanical of NBR according to different types of accelerators

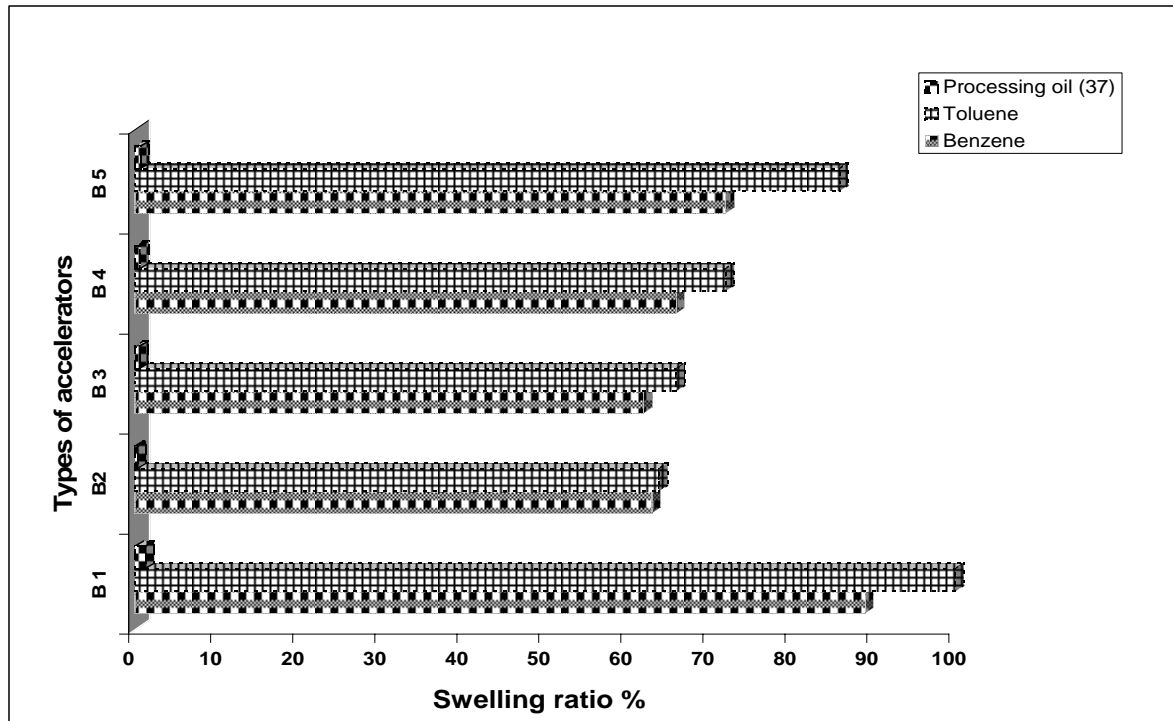


Figure 4. Swelling properties of NBR according to different types of accelerators

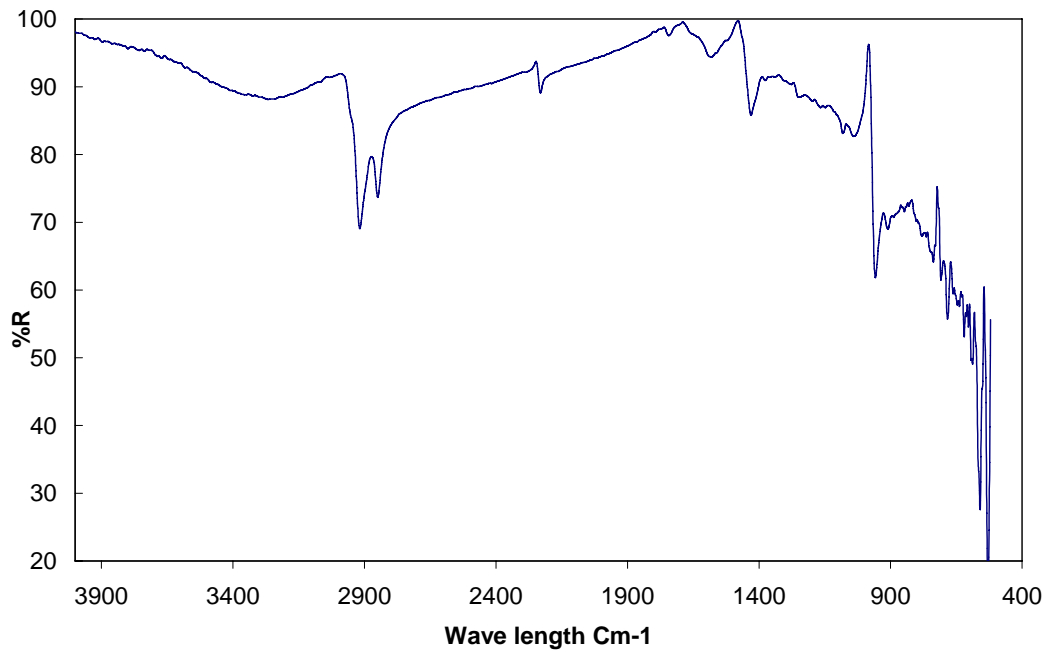


Figure 5. IR spectra of mixture B1

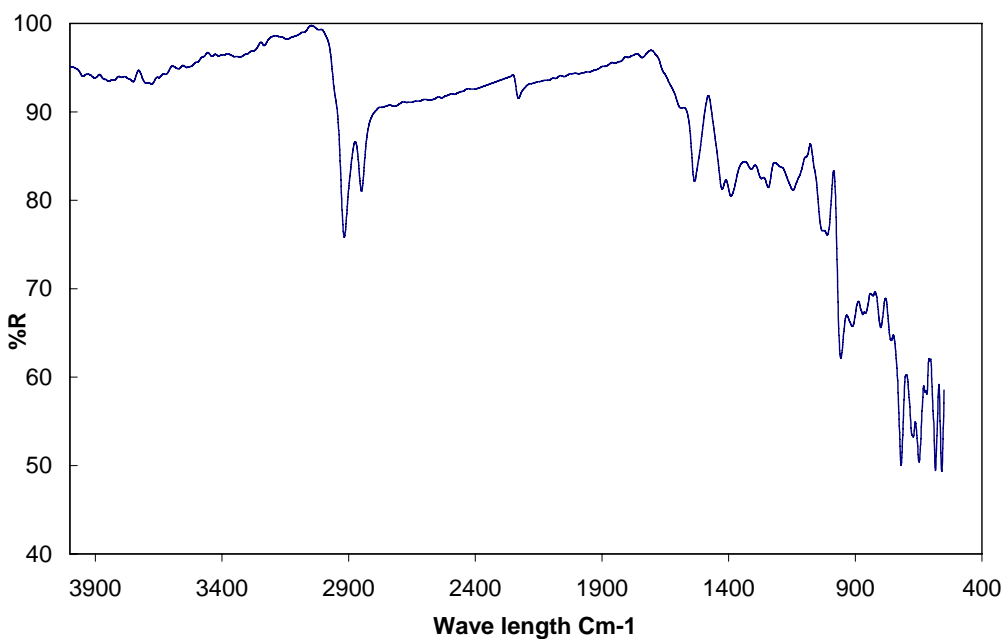


Figure 6. IR spectra of mixture B2

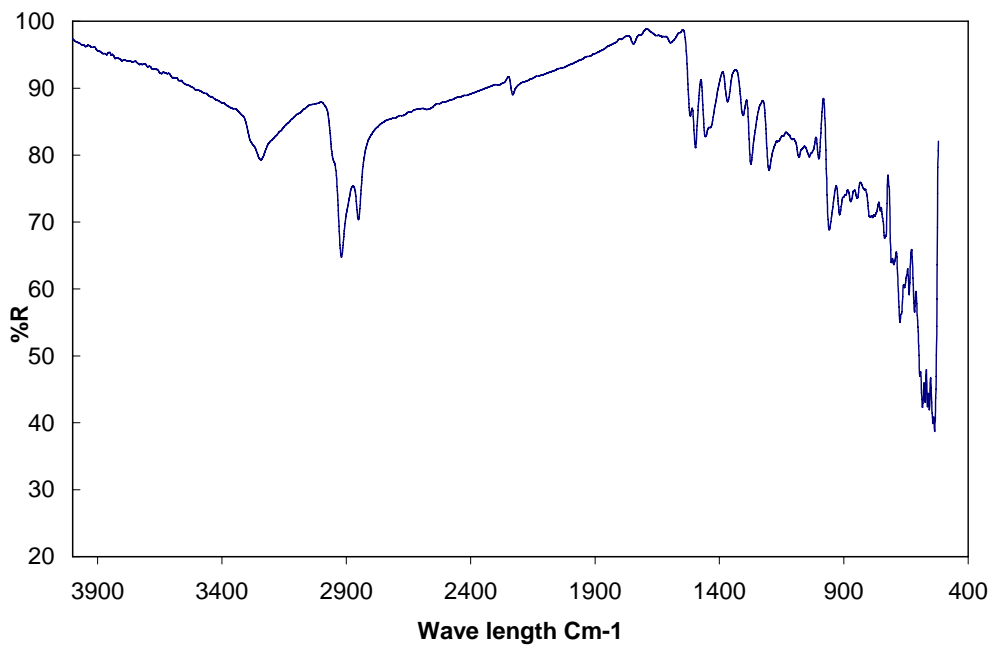


Figure 7. IR spectra of mixture B3

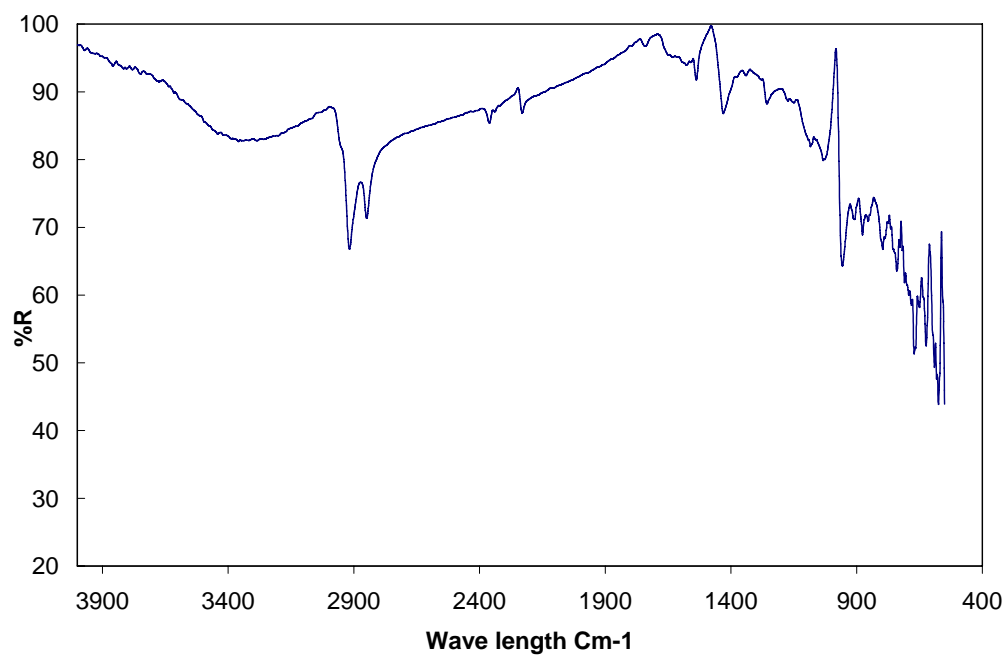


Figure 8. IR spectra of mixture B4

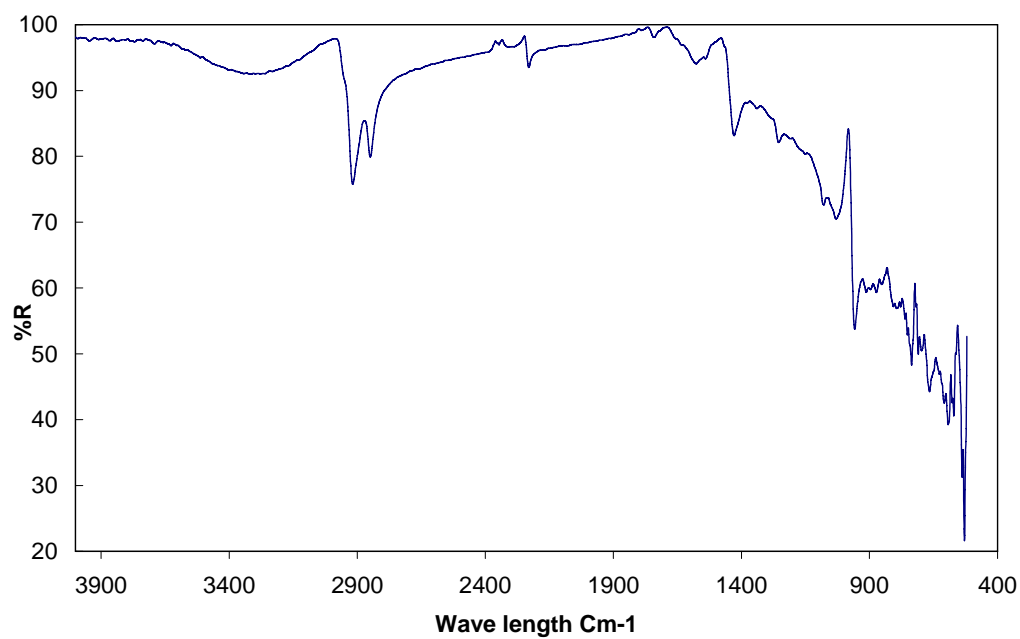


Figure 9. IR spectra of mixture B5

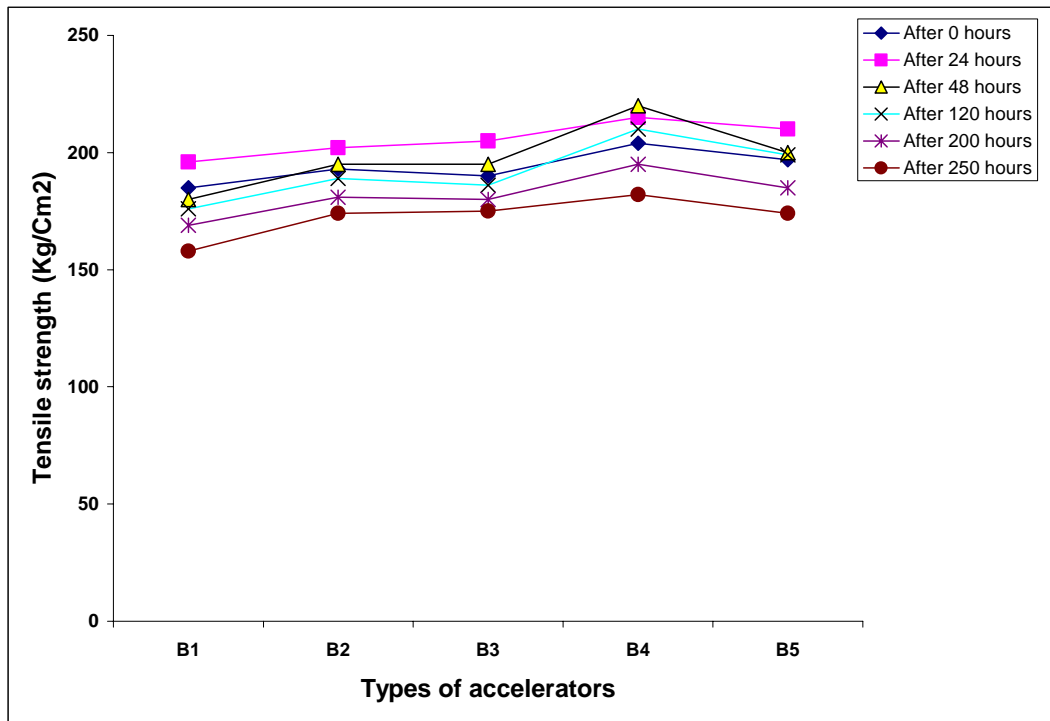


Figure 10. Effect of thermal ageing on tensile strength of NBR containing different types of accelerators

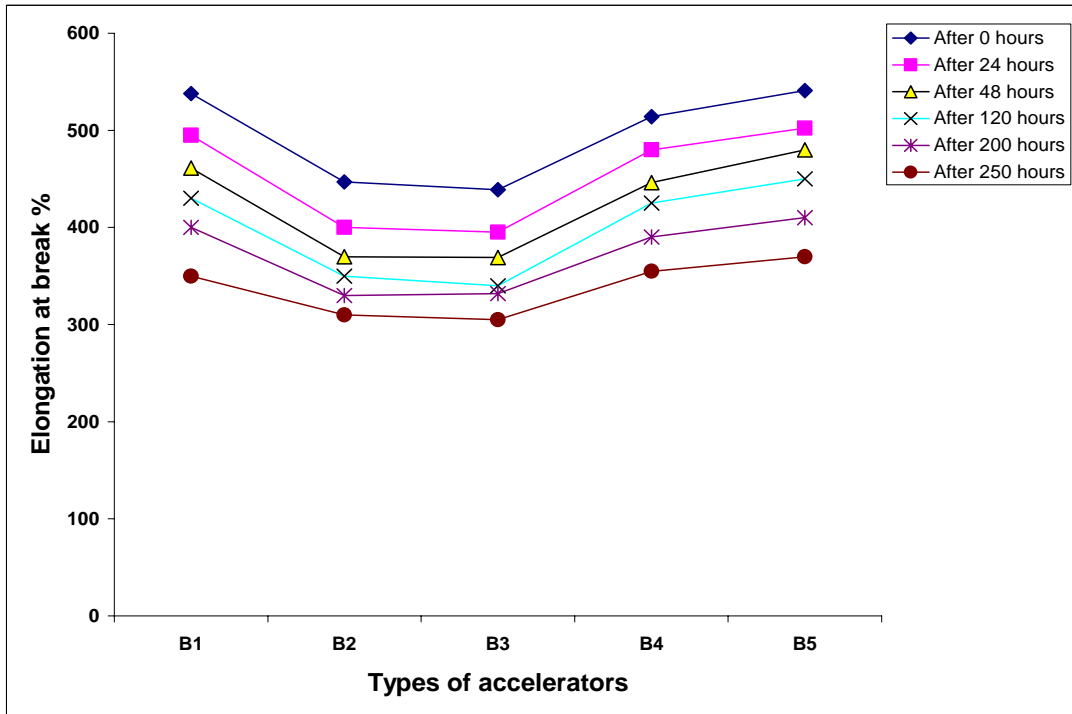


Figure 11. Effect of thermal ageing on elongation at break % of NBR containing different types of accelerators

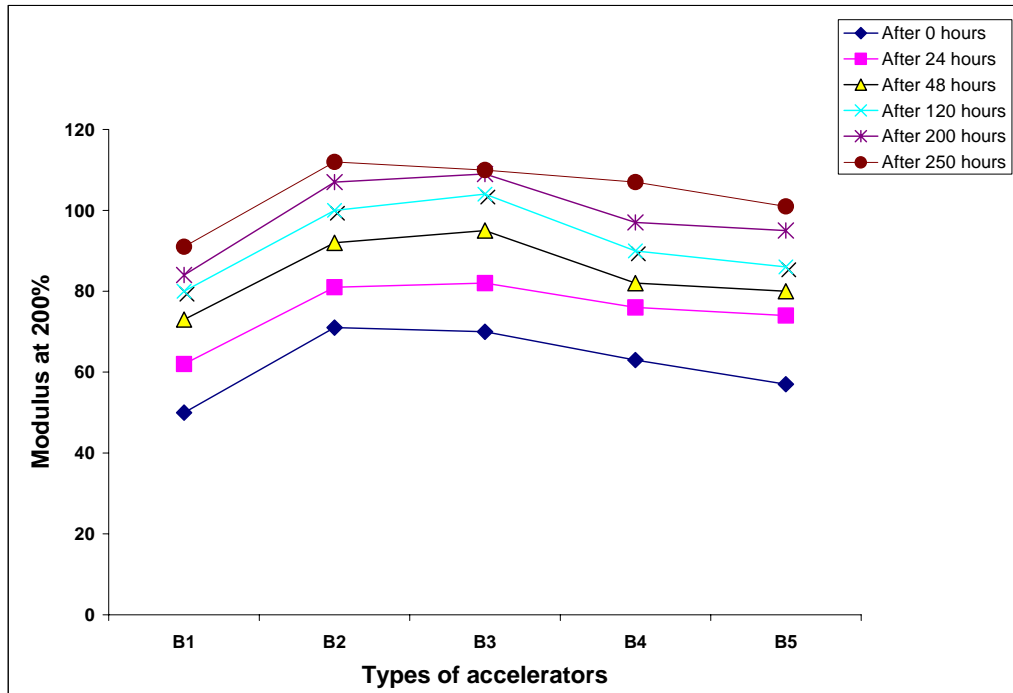


Figure 12. Effect of thermal ageing on modulus at 200 % of NBR containing different types of accelerators

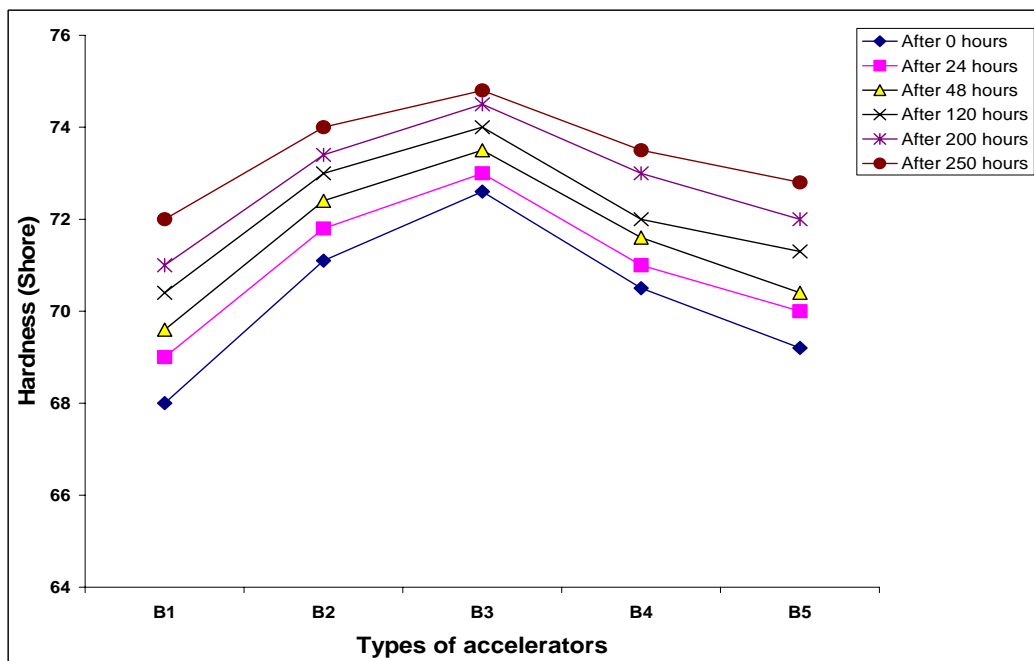


Figure 13. Effect of thermal ageing on hardness of NBR containing different types of accelerators

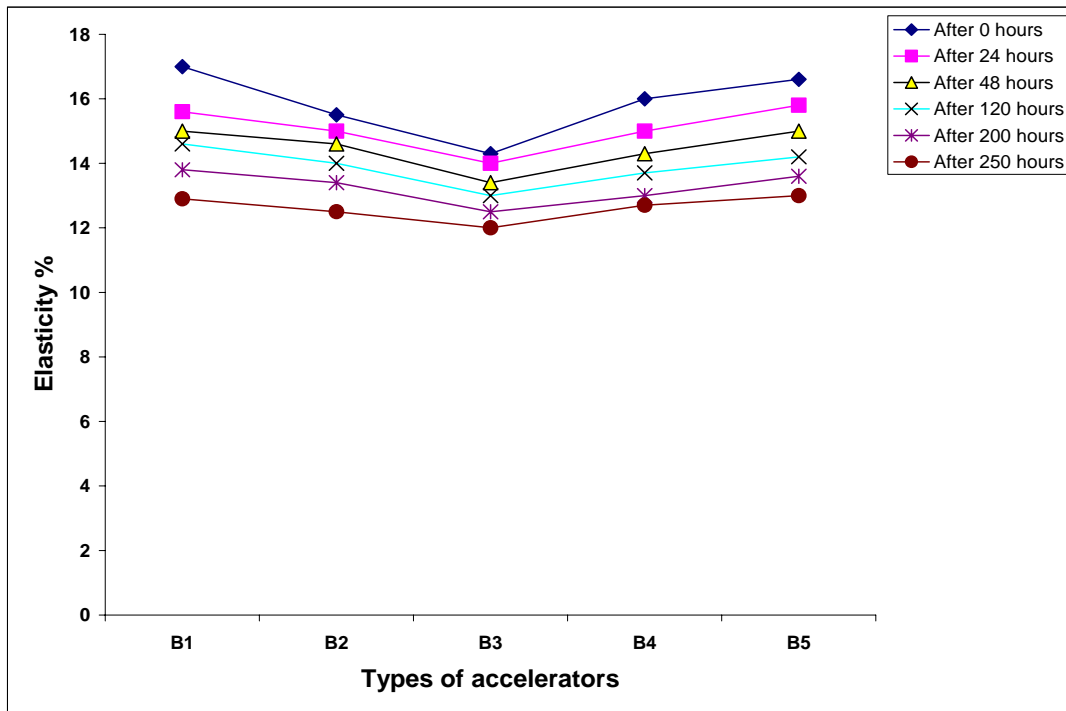
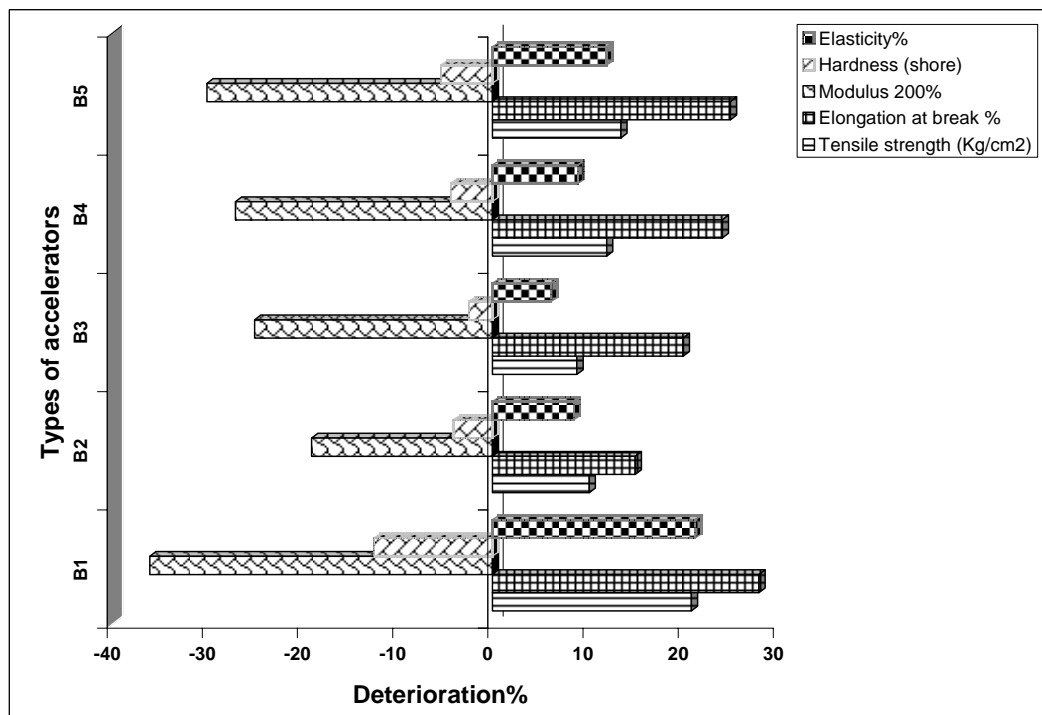


Figure 14. Effect of thermal ageing on elasticity % of NBR containing different types of accelerators



$$D\% \text{ (deterioration \%)} = (\text{value before ageing} - \text{value after ageing}) / \text{value before ageing} \times 100$$

Figure 15. Deterioration effect due to subjecting NBR mixtures to weathering ageing