

Some factors affecting the flex life of polybutadiene rubber vulcanisate*

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Abstract

Flex life of three different grades of polybutadiene rubber (BR) with highly linear chains, linear chains and long-branched chains were measured. The rubbers were reinforced with precipitated silica nanofiller the surface of which had been pre-treated with sulphur-bearing bis(3-triethoxysilylpropyl)-tetrasulphane (TESPT) coupling agent. The rubbers were cured by reacting the sulphur in TESPT with the rubber chains to produce vulcanisates. The mechanical properties of the rubber vulcanisates such as tensile strength, Young's modulus, elongation at break, stored energy density at break and tear energy were subsequently determined. The flex life of the rubber vulcanisates was also measured at a constant maximum strain amplitude and a test frequency of 3.17 Hz at ambient temperature. Additionally, the flex life of some unfilled rubber vulcanisates of similar Mooney viscosities cured with elemental sulphur was also measured.

For the silica-filled rubber vulcanisate, the rubber with the highly linear chains had the longest flex life and the one with long-branched chains, the shortest flex life. It seemed that a correlation between the flex life and the molecular chains structure might exist despite the crosslink density of the rubber vulcanisates being different and the compounds having silica in them. Also, for the unfilled rubber vulcanisates, the rubber with highly linear chains had the longest flex life and the one with long-branched chains the shortest flex life. Therefore, it was concluded that the flex life of the rubber vulcanisate was determined, to a large extent, by the molecular chains structure of the raw rubber, irrespective whether the rubber had reinforcing silica filler, different crosslink densities and different initial viscosities or not. A similar trend was also observed for some of the mechanical properties. For example, the elongation at break was lower and Young's modulus higher for the silica-filled rubber vulcanisates with long-branched chains than those measured for the silica-filled rubber vulcanisate with highly linear chains.

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INTRODUCTION

Mechanical testing like tensile testing and tear testing involves the application of static loads to the sample while flex testing involves the application of dynamic loads i.e., repeated stresses or strains to the sample.¹ Fatigue phenomenon can be observed in the form of cracks developing at particular locations in the material structure.² Materials under repeated cyclic loads can undergo accumulating damage which can be observed by the propagation of cracks.³ This damage is called fatigue and is shown by a loss of resistance with time. The physical effect of a repeated loads on material is different from the static loads. Fatigue failure always is brittle fracture irrespective of whether the material is brittle or ductile.⁴ Mostly fatigue failure occurs at stress well below the static elastic strength of the material.⁵ Increase in frequency decreases the flex life of an elastomer but it should be kept below 5Hz to avoid an increase in the temperature of the elastomer which decreases the flex life itself.⁶ Number of other factors affect the flex life of rubbers. These factors are strain amplitude, minimum stress, temperature, filler loading and extent of crosslinks in the rubber.⁷ For example, increase in filler loading increases the flex life.⁸ Increase in strain amplitude decreases the flex life.⁹ One factor which is of interest to rubber technologist's and has received little or no attention is the influence of the molecular structure of the raw elastomer chains on the flex life of rubber vulcanizate. In this study, we will investigate and determine effect of three different chains structures on the flex life of polybutadiene rubber (BR). The raw rubbers used had highly linear chains, linear chains and long-branched chains.

The rubbers were subsequently reinforced with silane pre-treated precipitated silica nanofiller and then cured with sulphur to produce rubber vulcanisates for further tests.

EXPERIMENTAL

Materials

Three grades of polybutadiene rubber were used. They were: BRCB22 (highly linear chains), BRCB24 (linear chains), and BRCB25 (long-branched chains). The rubbers were supplied by LANXESS in Germany. The reinforcing nanofiller was Coupsil 8113 (Evonik Industries, AG, Germany). Coupsil 8113 is a precipitated amorphous white silica-type (Ultrasil VN3), the surfaces of which had been pretreated with a bis-(3-triethoxysilylpropyl)-tetrasulphane (TESPT) coupling agent, also known as Si69. It has 11.3 % by weight TESPT and 2.5 % by weight sulphur (included in TESPT). The surface area of the filler was 175 m²/g (as measured by N₂ adsorption) and the particle size was 20-54 nm. In addition to the raw rubbers and filler, the other ingredients were N-tert-butyl-2-benzothiazole sulphenamide (TBBS; a safe-processing delayed action accelerator with a melting point of 109⁰C) (Santocure TBBS, Flexsys, Dallas, TX, USA), zinc oxide (ZnO; as an activator, ACROS ORGANICS, Belgium), and elemental sulphur (curing agent; Solvay Barium Strontium, Hannover, Germany). The melting temperatures of ZnO and silanised silica are above 1000 ⁰C. The cure system consisted of TBBS, ZnO and sulphur, which were added to fully crosslink the rubbers. To protect the rubbers against environmental ageing, N-(1,3-dimethylbutyl)-N'-phenyl-*p*-phenylenediamine (Santoflex 13, Brussels, Belgium) (antioxidant 6PPD with a melting point of 45-51⁰C) was used.

Mixing

Raw rubber, solid filler and curing agents were mixed in a Haake Rheocord 90, a small size laboratory mixer with counter-rotating rotors. Banbury rotors were used to carry out the mixing

of the rubber compounds at room temperature ($\sim 24^{\circ}\text{C}$). The rotor speed was set at 45 rpm and the total mixing time was 16 minutes. The volume of the mixing chamber was 78cm^3 and it was 57% full during mixing.¹⁰ First, raw rubber and then immediately solid filler were placed in the mixing chamber and mixed for 10 minutes to disperse the silica particles well in the rubber. After 10 minutes elapsed, ram was raised and TBBS, ZnO and sulphur were added and mixed for another 6 minutes. The compound was then removed from the mixer, cooled down to ambient temperature and placed in a clean plastic bag. The rubber compounds were then stored at room temperature for at least one day, milled on two roll-mill to produce sheets of about 3 mm thick for further work (Compounds 1-3, Table 1). The cure properties of the rubber compounds were then measured. This mixing procedure was utilized for all the rubber compounds prepared in this study. For the second part of this study, some unfilled rubber compounds (Compounds 4-6, Table 1) were prepared. The initial viscosity of the raw rubbers before mixing with the chemicals were BRCB22: 62 Mooney Units (MU); BRCB24: 46 MU, and BRCB25: 48 MU. To ensure that the rubber viscosities were similar before the chemical additives were added, the raw BRCB22 rubber was mixed for 133 min, and BRCB24 and BRCB25 rubbers for 3 min, respectively and then the chemical additives were added and mixing continued for another 6 min to produce Compounds 4-6 in Table 1. The idea was to produce rubber compounds which had very similar viscosities at the end of the mixing process. In fact, that was the case, since the viscosity of Compounds 4-6 was somewhere between 43-47 MU as shown in Table 1. Note also that the cure systems were the same for these compounds.

Measurement of the viscosity and cure properties of the rubber compounds

The viscosity of the rubber compounds was measured at 100°C in a single speed rotational Mooney viscometer at 2 r.p.m.¹¹ The results were expressed in Mooney units (MU). The scorch

time t_{s2} , which is the time for the onset of cure and the optimum cure time t_{95} , which is the time for the completion of cure, were determined from the cure traces generated, using an oscillating disc rheometer (ODR) (Monsanto, Swindon, UK). The angular displacement in the ODR tests was $\pm 3^\circ$ and test frequency 1.7 Hz. The cure rate index, which is a measure of the rate of cure in the rubber, was calculated using the following expression:

$$[100/(t_{95} - t_{s2})] \quad (1)$$

Results from these tests are summarised in Table 1. Δ Torque which is the difference between the maximum and minimum torque values on the cure trace of a rubber compound and is an indication of crosslink density changes in the rubber was calculated (Table 1).

Curing of the rubber compounds

After measuring the cure properties and viscosity of the rubber compounds, the compounds were cured in a compression mold 2.8 mm thick in an electrically heated hydraulic press at 160°C under 40 MPa pressure, according to the optimum cure time of the compounds shown in Table 1. Approximately 190g of the uncured rubber compound was placed in the center of the compression mold to allow it to flow in every direction to prevent anisotropy from forming in the sheets. After the rubber was cured, the mold was taken out of the press and the rubber removed and left in air to cool down to ambient temperature. Finally, the cured rubber sheets were placed in clean plastic bag and stored at ambient temperature for at least 24 hours before their mechanical properties and flex life measured. Standard dumbbell-shaped and trouser test pieces were subsequently cut from the cured sheets of rubber for measuring the flex life and mechanical properties.

MECHANICAL TESTING

Hardness

A Shore A hardness device was used for measuring the hardness of the rubber vulcanisates. For these tests, cylindrical samples, 12.5 mm thick and 25 mm in diameter, were cured in a compression mold in the same way as curing the rubber sheets. The samples were placed in the hardness tester and readings were taken from 3-5 positions on each sample at ambient temperature. The median values were then recorded. For each rubber vulcanisate, three samples were used in these tests.¹²

Tensile properties

Tensile testing of the rubber vulcanisates was carried out at a crosshead speed of 100 mm/min at room temperature, using Lloyd's mechanical testing machine. Standard dumbbell-shaped specimens, total length 75 mm with a gauge length of 25 mm, were used to carry out the tensile testing.¹³ The tensile strength, Young's modulus, modulus at different strain amplitudes, elongation at break and stored energy density at break were subsequently measured.

Tear strength

To measure the tear strength of the rubber vulcanisates, rectangular strips, 100 mm long and 30 mm wide, were prepared from the cured sheets of rubber. A sharp cut, approximately 35 mm, was inserted along the length of the sample, half way along its width to produce two legs of the same dimensions to form trouser test pieces. The rubber samples were then tested at a cross-head speed of 100 mm/min in a mechanical testing machine to produce tear force versus cross-head separation from which an average tear force was measured. Finally, the average tear force, F_a , was placed in the equation below to calculate a tear energy for the rubber sample:

$$T = 2F_a/t \quad (2)$$

where F_a is the average tear force and t the sample thickness. For each rubber vulcanisate, five specimens were used and median values recorded.¹⁴ Extension in the legs during tearing was considered to be very small and hence equation 2 was used with no correction. This was in accordance with the procedure described in reference 14.

Flex testing of the rubber vulcanizates

Using standard dumbbell-shaped test pieces, the flex tests were performed in uniaxial tension in a house-built dynamic testing machine at a constant maximum strain amplitude of 100% and at a test frequency of 3.71Hz at room temperature (24.5°C). Eight samples were tested for each rubber vulcanisate and the number of cycles to failure, N , for each sample was recorded. The average number of cycles to failure for each vulcanisate was then calculated.¹⁵ Note that measuring the flex life of the rubber vulcanisates in this study was the first part of an on-going study which will produce power-law relationships for the vulcanisates. The flex life measurements will then be used in combination with the power-law relationships to derive theoretical equations for the flex life prediction of the rubber vulcanisates. Results from the new study will be reported in due course.

Crosslink density measurement

The crosslink density of the rubber vulcanisates was measured by swelling in a laboratory reagent grade of toluene (Fisher Scientific, UK). Cylindrical samples, similar in size to the ones used in the hardness measurement, were placed in toluene in small glass bottles. The increase in weight of each sample was measured frequently over time until it reached equilibrium. Once the equilibrium weight was reached, the samples were removed from toluene and their weight measured and then placed in a vacuum oven for 2-3 days at 80°C to fully extract the solvent. The samples were then removed from the oven and placed in a fume cabinet with flowing air for an

extra day at ambient temperature and weighed again to determine the final weight of the sample. Having recorded the initial weight of the dry samples before the tests began, the volume fraction of the rubber in the swollen gel was determined and finally, the crosslink density was calculated.¹⁶ To calculate the crosslink density of the rubber vulcanisates, the following information was used. For compound 1, the volume fraction of the rubber in the swollen gel, V_r was 0.099 and the interaction parameter χ , 0.32. For compound 2, V_r was 0.091 and χ , 0.31 and finally for compound 3, V_r was 0.17 and χ , 0.4.

RESULTS AND DISCUSSION

Viscosity and cure properties of the rubber compounds

Table 1 shows the viscosity and cure properties of the rubber compounds tested. For Compounds 1-3 (silica-filled compounds), the minimum torque, which is an indication of the uncured rubber compound viscosity, shows a similar trend to the Mooney viscosity as expected. For Compounds 1, 2, and 3, the minimum torque is 32, 25 and 27 dNm, which matches the trend recorded for the Mooney viscosity, which is 61, 47 and 51 MU, respectively. The maximum torque, which represents the extent of crosslink density in the rubber, shows an increasing trend from 92 to 118 dNm for Compounds 1-3, respectively. In fact, the Δ torque values, which indicate crosslink density changes in the rubber, for compounds 1, 2 and 3 are 60, 82, and 91 dNm, respectively. For Compounds 1, 2 and 3, the crosslink density was calculated to be 24 mol /m³, 22 mol /m³, and 46 mol /m³, respectively (Table 2), which does not match the trend observed for the Δ torque numbers. The scorch times of Compounds 1 and 2 were similar at 5.1-5.5 min but the scorch time of Compound 3 was noticeably longer at 8.2 min. The optimum cure time of Compounds 1 and 2 were 24.8 and 30.1 min, respectively and that of Compound 3, 28.5 min. The rate of cure as indicated by CRI, was similar for the three compounds at about 4.1-5.1 min⁻¹ (Table 1). Recall

that Compounds 1-3 were cured by reacting the sulphur in TESPT with the rubber chains by adding TBBS and ZnO curatives and no elemental sulphur was used in the curing reaction. The TBBS and ZnO requirements for these compounds were different. For Compound 1, they were 2.5 phr and 0.2 phr, for Compound 2, 5 phr and 0.2 phr, and for Compound 3, 5.5 phr and 0.5 phr, respectively (Table 1). The double bond concentrations for the raw BR rubbers were determined with Nuclear Magnetic Resonance Spectroscopy (¹HNMR) technique and found to be BRCB22: 49.9%; BRCB24: 49.8%; BRCB25: 49.5%, respectively, which were similar. All the indications are that there is no direct correlation between the concentration of the double bonds in the rubber chains and the TBBS and ZnO requirement for full cure at least for these compounds. The procedure for measuring the optimum amounts of the TBBS and ZnO curatives for curing the silica-filled BR rubbers was described in a previous publication¹⁷

For Compounds 4-6 (unfilled compounds with the same cure system), the minimum torque was 13 dNm. The Mooney viscosity of these compounds was somewhere between 43-47 MU. But the maximum torque was somewhere between 60-78 dNm. This produced Δ torque values from 47 to 65 dNm, respectively, which indicated different crosslink densities in the rubber despite the compounds having the same cure system. It must be mentioned that Δ torque is influenced by stable covalent sulphur chemical bonds between the rubber chains as well as contribution from the physical interactions between the rubber chains, e.g. due to mechanical entanglement/interaction and attractive Van der Waals forces. The exact contribution from the chemical bonds and physical bonds are not easily understood and this may vary from one sample to another, causing variation in the overall crosslink density of the rubber vulcanizate.^{18,19} The scorch time of the compounds was somewhere between 9.2 to 12.2 min and the optimum cure

time between 46.8-50.3 min. Notably, the rate of cure was almost the same for the three compounds, with the CRI being at 2.6-2.7 min⁻¹ (Table 1).

Hardness, mechanical properties and flex life measurements of the silica-filled rubber vulcanisates

The hardness of the rubber vulcanisates (Compounds 1-3, Table 2) increased from 55 to 64 Shore A, respectively. This trend was consistent with an increase in the crosslink density as indicated by the Δ torque values, which rose from 60 to 91 dNm, respectively. Clearly, as the crosslink density increased, the rubber became harder as expected.

The rubber vulcanisates had very different mechanical properties despite having the same loading of silica, i.e. 30 phr, which could be due to different crosslink densities as indicated by the Δ torque values in Table 1. The highest tensile strength and elongation at break were measured for Compound 2 at 11 MPa and 769%, respectively, whereas Compounds 1 and 3 had similar tensile strength at about 7.5-8 MPa and elongation at break at 697% and 414%, respectively. A similar pattern was also observed for the stored energy density at break. Compound 2 had a 38 MJ/m³ stored energy density at break, whereas Compounds 1 and 3, had 24 and 17 MJ/m³ stored energy density at break, respectively. The Young's modulus increased progressively from 2.3 MPa for Compound 1 to 4.6 MPa for Compound 3. This trend matched that of the hardness, which also increased progressively from 55 to 64 Shore A for the Compounds. The modulus at 100%, 200% and 300% strain amplitudes showed a similar trend. The tear energy of Compounds 1 and 2 were similar at 7-8 kJ/m² whereas the tear energy of Compound 3 was slightly lower at 5.8 kJ/m².

In a highly linear chains rubber such as Compound 1, when the rubber is stretched, the chains slide past each other much easier than they do in the much tighter long-branched chains network

like Compound 3, affecting the tensile properties of the rubber. In fact, when the results in Table 1 are re-examined, that seems to be the case. For example, Compound 1 has a much higher elongation at break than Compound 3. As expected, Compound 3 has a much higher Young's modulus than Compound 1 because it has a much stiffer network due to the physical entanglements of the rubber chains. The same applies to the modulus at 100, 200 and 300% strain amplitudes where Compound 3 shows a much higher modulus than Compound 1.

Results from the flex tests on the silica-filled rubber vulcanisates (Compounds 1-3; Table 1) are presented in Fig.1. As the figure shows, the flex life of Compound 1 is somewhere between 35510 and 4 million cycles, which gives an average value of 2.2 million cycles. Compound 2 had flex life between 7340 and 1.7 million cycles, which gives an average value of about 0.24 million cycles. The flex life of Compound 3 was much shorter between 2108 and 6184 cycles, which gives an average value of 3955 cycles. Evidently, Compound 1 has the longest and Compound 3 the shortest flex life. It is interesting that Compound 1, the rubber with the highly linear chains, has the longest flex life and that of Compound 3 with long-branched chains, the shortest life. It seems that a correlation between the flex life and the molecular chains structure of the rubber may exist despite the crosslink density of the rubber vulcanisates being different and the compounds having silica in them. The crosslink density of Compounds 1 and 2 were 24 and 22 mol/m³ and that of Compound 3, 46 mol/m³, respectively (Table 2). Note that the Δ torque values for these compounds are not the same and increase from 60 dNm for Compound 1 to 91 dNm for Compound 3 (Table 2), which further indicates that these rubber vulcanisates have different internal structures. To further investigate effect of the molecular chains structure on the flex life of the rubber, Compounds 4-6 (Table 1) were prepared. These compounds had the same cure system and no silica filler in them.

Flex life measurements of the unfilled rubber vulcanisates

Figure 2 shows the flex life of the unfilled rubber vulcanisates (Compounds 4-6, Table 1). As mentioned earlier, these compounds had the same cure system and no silica filler in them. Furthermore, the rubbers had very similar viscosities, somewhere between 43-47 MU (Table 1), though had different crosslink densities as indicated by the Δ torque values, which increased from 47 dNm for Compound 1 to 61 dN m for Compound 3.

For Compound 1, the flex life increases from 933 to 3093 cycles. For Compound 2, there is a noticeable reduction in the flex life, which rises from 509 to 1008 cycles. Interestingly, the flex life of Compound 3 is very similar to that of Compound 2, which is from 471 to 1605 cycles. The trend is similar to the one observed for the silica-filled rubber vulcanisates (see Fig. 1). In both cases, the rubber with the highly linear chains has the longest flex life and that with the long-branched chains the shortest flex life. It seems that the flex life of the rubber vulcanisate is determined, to a large extent, by the molecular chains structure, irrespective whether the rubber has reinforcing silica filler and different crosslink densities or not.

There are numerous factors which affect the flex life of a rubber vulcanisate. For example, molecular structure and the number of chains free ends, chains branching which may interfere with the crosslinking process and affect the uniformity of crosslinks distribution in the rubber, molecular chains entanglement and molecular chain slippage. It is not immediately clear to what extent these factors have affected the flex life of the rubber vulcanisates and hence further work will be needed to study the internal structure of the rubbers in more detail using for example dynamic mechanical analysis (DMA). Clearly, the results show that the molecular chains structure is an important factor in determining the flex life of a rubber vulcanisate and therefore this topic merits further investigation.

CONCLUSIONS

This study examined effect of the molecular chains structure on the flex life of a polybutadiene rubber. The following conclusions are reached.

- For the silica-filled rubber vulcanisate, the rubber with the highly linear chains has the longest flex life and the one with long-branched chains, the shortest flex life. It seems that a correlation between the flex life and the molecular chains structure may exist despite the viscosity of the rubber compounds and crosslink density of the rubber vulcanisates being different and the compounds having silica in them.
- Similarly, for the unfilled rubber vulcanisates, the rubber with highly linear chains has the longest flex life and that with long-branched chains the shortest flex life. For these compounds, the rubber viscosities were comparable, and the cure system was the same although the crosslink densities were different.

Thus, it seems that the flex life of the rubber vulcanisates is determined, to a large extent, by the molecular chains structure of the raw rubber, irrespective whether the rubber has reinforcing silica filler, different crosslink densities and different viscosities or not. A similar trend is also observed for some of the mechanical properties. For example, the elongation at break was lower and Young's modulus higher for the rubber vulcanisates with long-branched chains than those measured for the rubber vulcanisate with highly linear chains.

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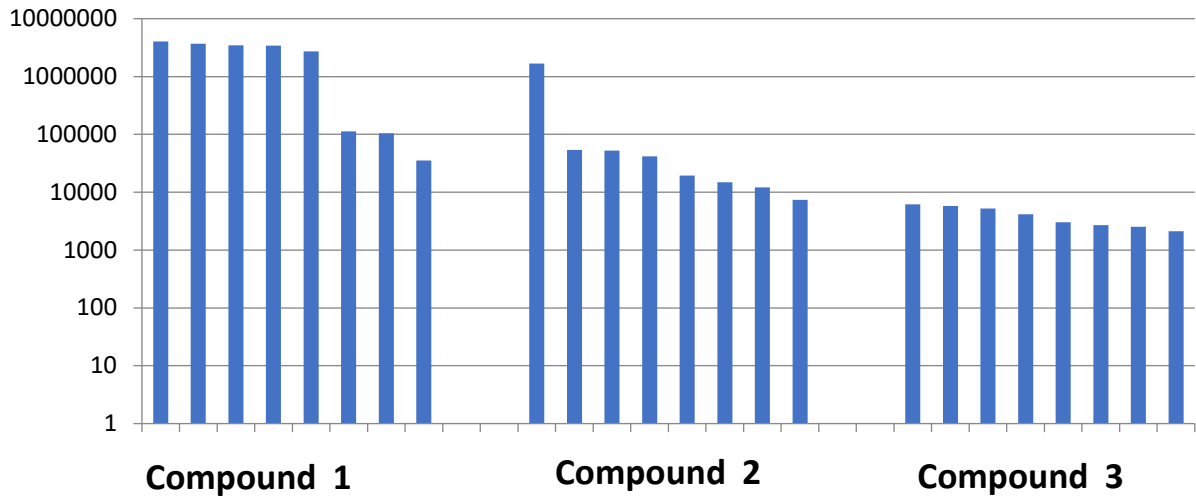


Fig 1 - Flex life of the silica-filled rubber vulcanisates

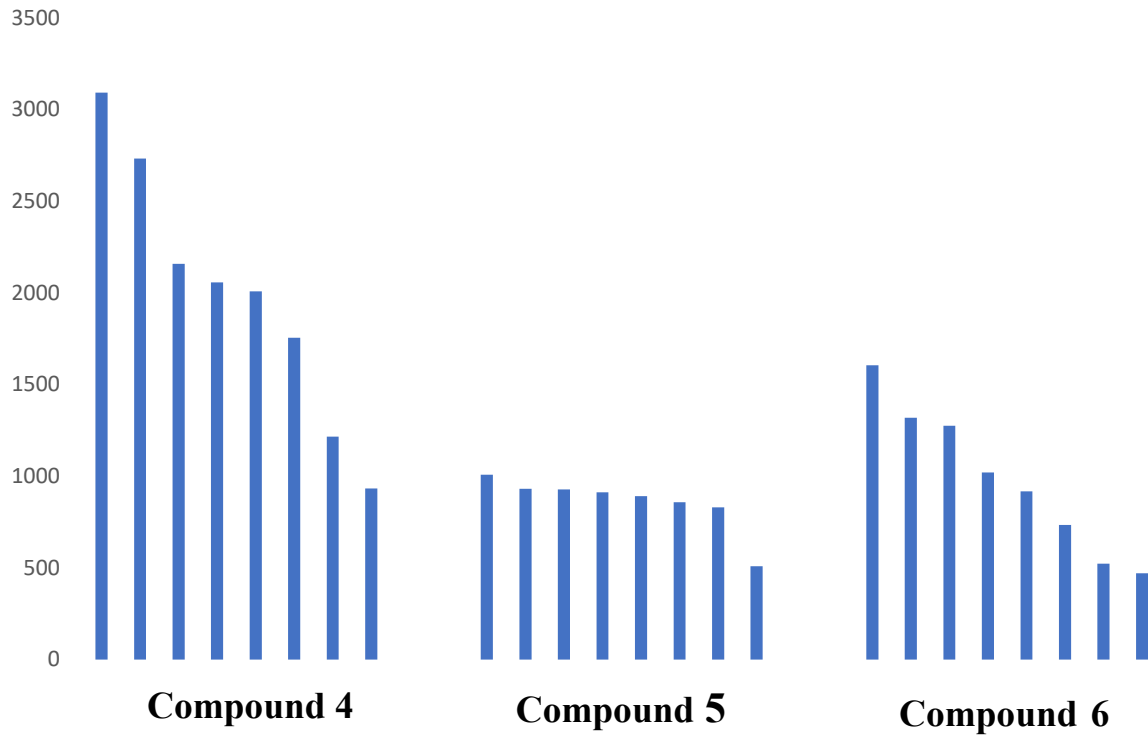


Figure 2 – Flex life of the unfilled rubber vulcanisates

Table 1 – Formulations, Mooney viscosity and cure properties of the rubber compounds

Formulation (phr)	Compound 1	Compound 2	Compound 3	Compound 4	Compound 5	Compound 6
BRCB22	100	-	-	100	-	-
BRCB24	-	100	-	-	100	-
BRCB25	-	-	100	-	-	100
Silanized silica	30	30	30	-	-	-
TBBS	2.5	5	5.5	1.75	1.75	1.75
ZnO	0.2	0.2	0.5	0.2	0.2	0.2
Sulphur	-	-	-	0.5	0.5	0.5
Santoflex-13	-	-	-	1	1	1
Minimum torque, M _L (dNm)	32	25	27	13	13	13
Maximum torque, M _H (dNm)	92	107	118	60	78	74
ΔTorque (dNm)	60	82	91	47	65	61
t _{s2} (mins)	5.1	5.5	8.2	9.2	12.1	12.2
t ₉₅ (mins)	24.8	30.1	28.5	46.8	50.3	49.8
CRI (min ⁻¹)	5.1	4.1	4.9	2.7	2.6	2.7
Viscosity (MU)	61	47	51	47	44	43

Table 2 Mechanical properties of the rubber vulcanisates (Compounds 1-3, Table 2).

Properties	Compound 1	Compound 2	Compound 3
Hardness (Shore A)	55	58	64
Tensile Strength (MPa)	7.53	11.01	8.05
Elongation at break (%)	697	769	414
Stored Energy density at break (mJ/m ³)	24.3	37.92	17.24
Tear Strength (kJm ⁻²)	8.03	7.03	5.77
Young's Modulus (MPa)	2.34	2.73	4.55
Modulus at 100% Strain (MPa)	0.6	0.88	1.54
Modulus at 200% Strain (MPa)	0.68	0.9	1.5
Modulus at 300% Strain (MPa)	0.9	0.93	1.55
Crosslink density (mol/m ³)	23.69	22.24	46