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# Evaluating the efficacy of a newly developed palm-based process aid on nitrile rubber composites

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## Abstract

The exploration of bio-based process aid is still ongoing in the effort to replace petroleum-based process oil. In this study, the efficacy of a newly developed palm-based process aid (Bio) on nitrile rubber (NBR) composites was investigated by varying its concentration from 0.0 to 10.0 phr. A comparison was made against a commercial oil (CO), i.e. naphthenic oil in terms of Mooney viscosity, cure characteristics, morphology, physical, and chemical properties. Results revealed that the use of Bio as process aid improves the processability of rubber by lowering the Mooney viscosity, enhancing filler dispersion, and lowering loss tangent values. Tensile properties and compression sets of CO-NBR composites were slightly better compared to Bio-NBR composites. Interestingly, Bio-NBR composites showed better ageing properties, especially at higher process aid content. Higher retentions of tensile strength and elongation at break and lower compression sets of aged Bio-NBR compared to CO-NBR composites were observed, which are possibly contributed by the good compatibility of Bio with aged rubber. Thermogravimetric analysis showed that the thermal stability of Bio-NBR composites were slightly better than CO-NBR composites. Based on these findings, the use of Bio ($\geq 7.5$ phr) as process aid significantly improved the processability of rubber with some enhancement in ageing properties of composites that is desirable and ideal for various applications such as for the production of automotive components.

## Introduction

In recent years, rubber goods' production has shifted towards a more eco-friendly and sustainable approach. The implementation of regulations on limits of toxic substances such as the REACH regulation has driven the search of alternative raw materials that are safe and renewable to produce eco-friendly products. One of the most widely used ingredients in rubber compounding is process aid. Process aid or plasticiser is used to improve the flexibility and processing of rubbers or polymers by lowering the glass transition temperature ($T_g$) [1], reducing the viscosity and elasticity of rubber and also helps in dispersing fillers. By improving the processability of rubber, the power consumption to process the rubber is reduced and consequently, the operational cost.

Petroleum-based process oil has been used widely as an extender and process aid for rubber goods and tyres. Aromatic oil particularly is preferred by most industries due to its excellent compatibility with most elastomers [2, 3]. Nonetheless, many studies have reported that aromatic oil contains a high amount of polycyclic aromatic hydrocarbon compounds (PAH), which are carcinogenic. The emission of PAH into the environment, such as from the wear of tyre tread material, could be due to the PAH being not chemically bonded into the rubber matrix [2]. In December 2009, restrictions of PAH rich extender oils by the European countries had given rise to the search for alternatives for the oil and rubber industries [2]. Furthermore, these additives are rather expensive, and the price is unstable since they were derived from petrochemical sources [4]. They are also expected to become depleted in a few decades [5, 6]. Although some industries have opted for petroleum-based process oil with lower PAH content such as paraffinic oil, naphthenic oil and treated distillate aromatic extract as replacements for aromatic oil, there have been claims that these oils also contain carcinogens as they come from the

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same resource. Therefore, there has been an increasing demand and trend on the exploration and development of green plasticisers or additives that are renewable and safe.

Alternatively, process aid can be derived from renewable sources such as plants. Some plants have few molecules with unique structural features that are suitable as a plasticiser and process aid of low cost [3, 4]. A few examples of common plant or vegetable oils used for this purpose are soybean oil [6–9], palm oil [5], rubber seed oil [4], and castor oil [10]. Most researchers and plasticiser manufacturers used the epoxidation technique on extracted oil to improve its compatibility with rubber [11], which is influenced by the level of unsaturation of the oil. There is only a handful of studies using the derivatives of these oils, which could provide better control in terms of properties and structure of the process aid. Furthermore, despite their potentials in replacing petroleum-based process oil, the benefits and properties of resulting composites with the addition of these bio-based process aids are still below synthetic plasticisers [11].

In this study, the effects of a newly developed palm-based process aid (Bio) were investigated on nitrile rubber (NBR) composites in terms of Mooney viscosity, cure characteristics, morphology, physical, chemical, and ageing properties. Bio is a palm-based polyester that is non-toxic, environmentally friendly, and renewable. Prior studies have shown that palm-based plasticiser has improved the processability of rubber during mixing [5, 12] and offers higher heat resistance compared to other vegetable oils [13]. NBR was used due to its excellent chemical, oil, heat ageing, abrasion, water, and gas resistances [14], which can be used for many applications such as corrosion inhibition [14], fuel hose [15] and automotive component [16]. A commercial petroleum-based naphthenic oil (CO) was also used for comparison purpose. The findings of this study will help in developing alternatives to commercial petroleum-based process aid, especially for nitrile rubber-based composites.

## Materials and methods

### Materials

Nitrile rubber used was Nipol DN3350 (50.2 MU, 33.0% acrylonitrile content), which was produced by Zeon Chemicals. The carbon black of grade N550 was supplied by Cabot (M) Sdn. Bhd. Accelerators such as N-tertiary butyl-2-benzothiazole sulfenamide (TBBS) and tetramethyl thiuram disulfide (TMTD) were purchased from Galin Enterprise. Anti-degradant, 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ) was purchased from CSPL (M) Sdn. Bhd. Sulphur and activators such as stearic acid and zinc oxide were obtained from Centre West Sdn. Bhd. For comparison purposes, a naphthenic oil (Nytex 840) from
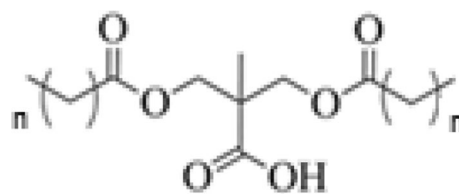


**Fig. 1** The proposed structure of the palm-based polyester, Bio [18]

**Table 1** Formulation recipes

| Ingredients | Composition |
| --- | --- |
| Nitrile rubber, Nipol DN3350 | 100.0 |
| Carbon black FEF, N550 | 40.0 |
| Zinc oxide | 5.0 |
| Stearic acid | 2.0 |
| TMQ | 2.0 |
| Bio-based process aid (Bio) or Naphthenic oil (CO) | 0.0–10.0 |
| Sulphur | 2.5 |
| TMTD | 1.0 |
| TBBS | 1.0 |

Nynas was used. The synthesis and properties of the palm-based processing aid (Bio) were as reported in previous literature [17, 18]. Figure 1 shows the proposed structure of palm-based process aid used in this study [18].

### Sample preparation

Table 1 shows the formulation used to produce NBR composites. Bio and commercial oil (CO) content were varied at 0.0, 2.5, 5.0, 7.5 and 10.0 phr. Semi-efficient curing system was used in the formulations. Compounding was done with a two-roll mill at room temperature. Mooney viscosity at 100 °C and curing characteristics at 150 °C were obtained from Mooney viscometer (MV 2000, Alpha Technologies) and rheometer (MDR 2000, Alpha Technologies), respectively. Cure Rate Index (CRI) of each compound was also calculated as per the following equation where $T_{90}$ and $T_{s2}$ are the curing time and scorch time, respectively [19]:

$$\text{Cure Rate Index} = \frac{100}{T_{90} - T_{s2}}. \tag{1}$$

Rubber compounds were cured with a hot press at 150 °C and tested for tensile properties, dynamic mechanical analysis (DMA), compression set, ageing properties, morphology, and chemical analysis such as Fourier transform infrared (FTIR) spectroscopy and thermogravimetric analysis (TGA).

## Physical testing

At least five replicates of each blend were used to determine the tensile properties. The tensile tests were conducted according to ISO 37 standard using an Instron universal testing machine. Another set of tensile specimens were put in an oven at 100 °C for 70 h to study the ageing properties. The ageing resistance was expressed as a percentage of retention, as shown in the following equation [19]:

$$\text{Retention} (\%) = \frac{\text{Value after ageing}}{\text{Value before ageing}} \times 100. \quad (2)$$

Triplicates of each sample were also put under 25% constant compression for 24 h at room temperature and 70 °C following ISO 815. The percentage of compression set was calculated as in the following equation:

$$\text{Compression set} (\%) = \frac{\text{Original thickness} - \text{Recovered thickness}}{\text{Original thickness} - \text{Spacer thickness}} \times 100. \quad (3)$$

## Dynamic mechanical analysis

Dynamic properties such as storage modulus and loss tangent were measured using a Mettler Toledo Dynamic Mechanical Analyser (DMA1 Start system) at temperatures from −100 to + 100 °C in tension mode at 10 Hz, 0.1% strain in accordance to ISO 4664-1.

## Attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy

ATR-FTIR spectra were obtained at room temperature using a Nicolet iS50 Instrument in the range of 700–4000 cm$^{-1}$. The ATR crystal used was germanium.

## Thermogravimetric analysis

Thermal stability of process aids and vulcanisates were determined using a Perkin Elmer STA 6000 thermal analyser. Specimens were weighed at $10 \pm 1$ mg and heated from 50 to 800 °C at 10 °C/min.

## Transmission electron microscopy

Rubber samples were cut using a microtome to an approximate thickness of ca. 100 nm and viewed under FEI Tecnai G2 transmission electron microscope (TEM).

## Results and discussion

### Fourier transform infrared spectroscopy

Figure 2 exhibits the FTIR spectra of NBR composites without any process aid, i.e. control (0.0 phr), with 10.0



**Fig. 2** ATR-FTIR spectra of NBR composites with and without process aids
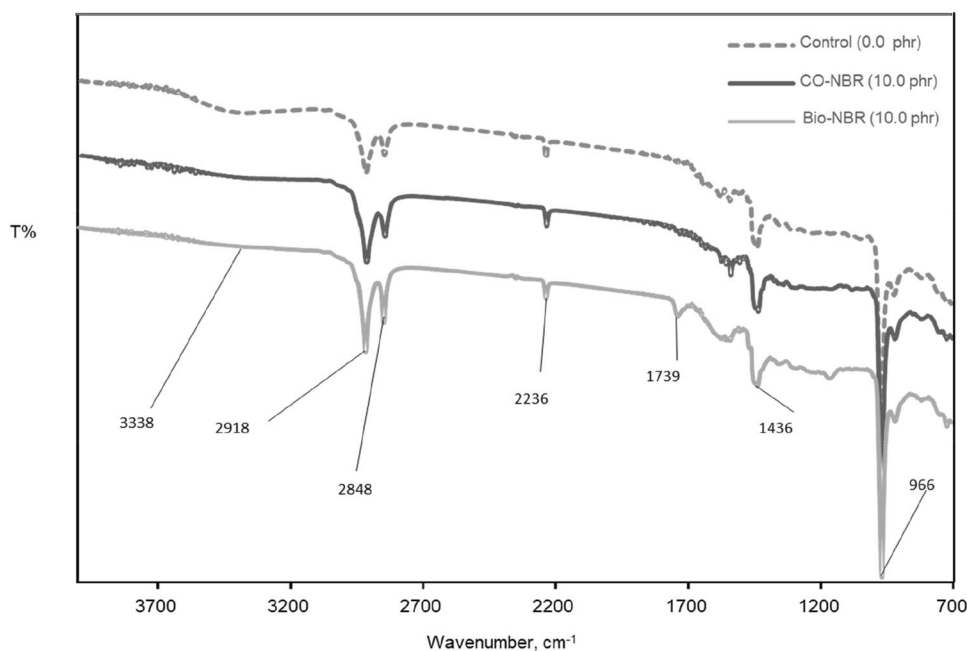
phr of CO and 10.0 phr of Bio, respectively. The band at 3338 cm$^{-1}$ represents the O–H and N–H stretches, whereas bands at 2918 cm$^{-1}$ and 2848 cm$^{-1}$ are attributed to the C–H stretches [20]. The –C≡N stretching was observed at 2236 cm$^{-1}$ [20]. Band at 1436 cm$^{-1}$ corresponds to C–H bending vibration of –CH$_2$, whereas band at 966 cm$^{-1}$ refers to C–H deformation vibration of the trans-1, 4 structure of butadiene part [16, 20]. With the addition of Bio, band characteristic of carbonyl groups C=O appeared at 1739 cm$^{-1}$, which is attributed to the presence of ester in Bio, as depicted in Fig. 1. In comparison, a similar band was not clearly visible in CO-NBR composite or composite with no process aid. The difference in functional groups will determine the compatibility of processing aid with rubber hence, its efficiency.

## Mooney viscosity

The primary function of process aid is to improve the processability of rubber by reducing the viscosity of rubber. It can be seen from Fig. 3 that the Mooney viscosity of rubber compounds decreased with increasing process aid loading, indicating softening or lubricating effect of the process aids [21]. The reduction of Mooney viscosity improves the processability of rubber compounds hence, increasing the processing efficiency. At the highest process aid loading (10.0 phr), the Mooney viscosity of NBR compound mixed with Bio was 37.6 MU, which is lower than the compound mixed with CO (48.5 MU). The higher reduction in Mooney by Bio could be due to the better compatibility of Bio with rubber. To achieve similar Mooney viscosity, the amount of Bio required is smaller than CO. The use of a lower dosage of process aid is essential as it reduces the production cost of the product.
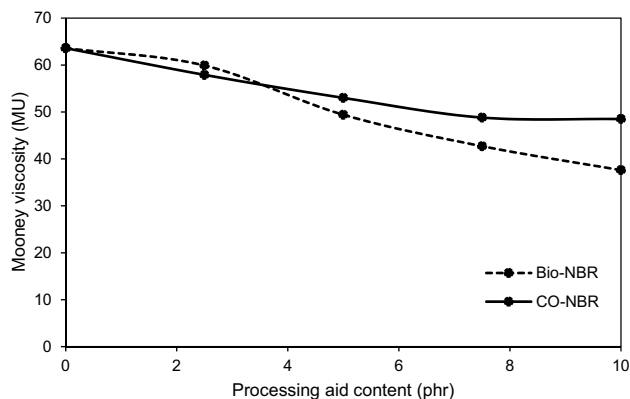
## Morphology

A comparison of TEM images showing the dispersion of carbon black particles of CO- and Bio-NBR composites at 7.5 phr of process aids is depicted in Fig. 4a, b, respectively. It can be seen that the size of carbon black aggregates in CO-NBR composite is larger than in Bio-NBR composite. This result reveals that carbon black particles are more homogeneously distributed in NBR composite with the addition of Bio implying, better processability of rubber during compounding.

## Cure characteristics

Figure 5a–c shows examples of cure curves of NBR compounds examined by the disc rheometer. The cure curve exhibits three distinct stages, which are induction, crosslinking, and post-crosslinking (in this case, a plateau) that are commonly adopted for all rubber materials with double links in their backbones [22]. The effects of process aids on the cure characteristics of carbon black-filled NBR compounds were extracted from the cure curves and displayed in Table 2. Delta torque is the difference between maximum (M$_H$) and
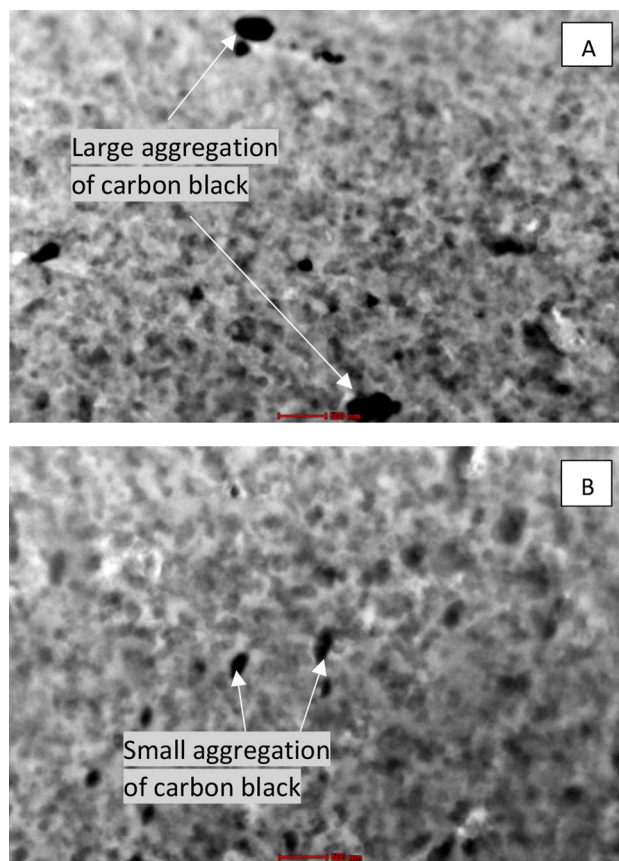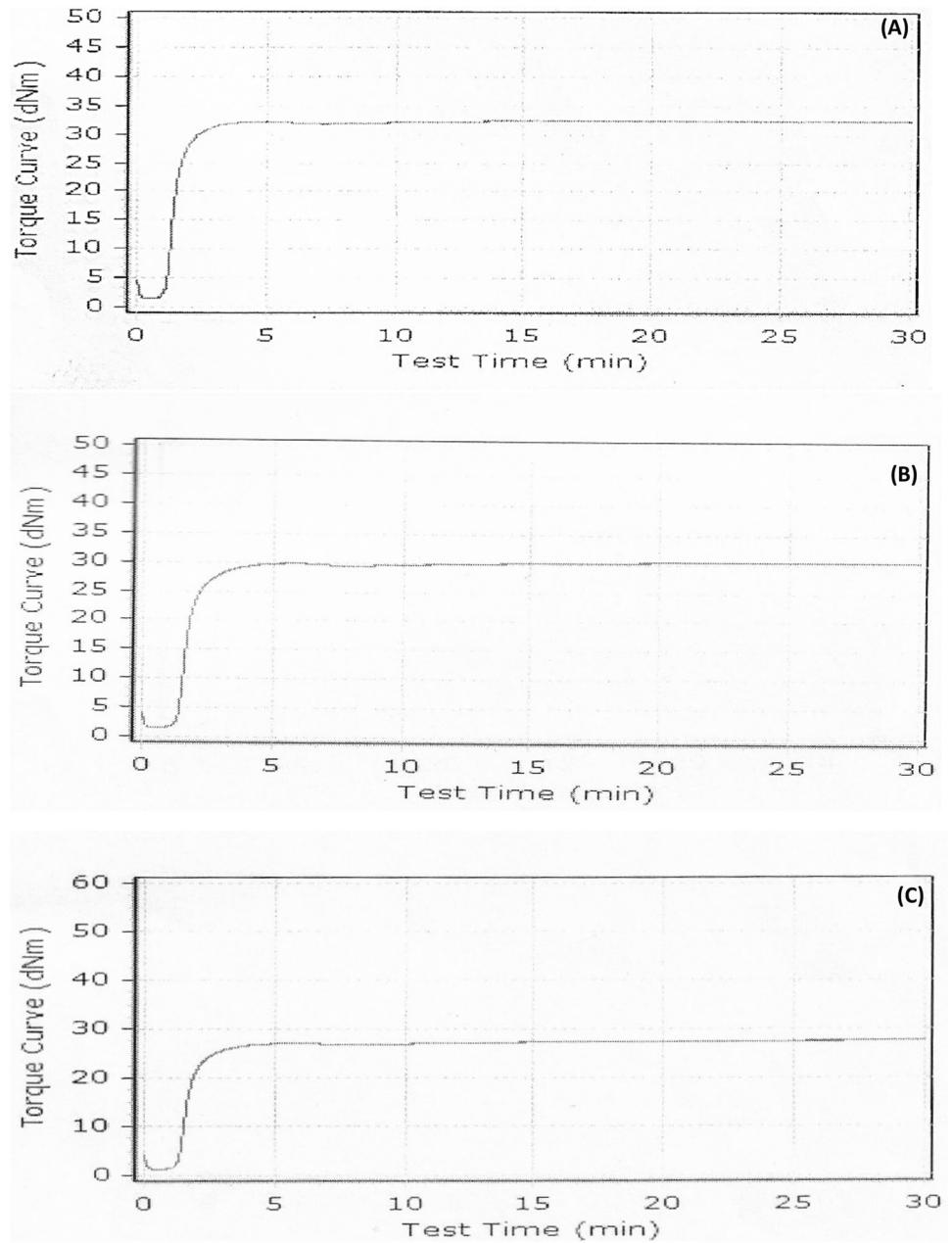


Fig. 4 TEM images of **a** CO- and **b** Bio-NBR composites



Fig. 3 Mooney viscosity of NBR compounds

**Fig. 5** Rheometer cure curves of **a** control, **b** Bio-NBR, and **c** CO-NBR compounds



**Table 2** Curing characteristics of NBR compounds

| Process aid loading (phr) | $M_L$, dNM | $M_H$, dNM | Delta torque, dNm | $T_{S2}$, min | $T_{90}$, min | CRI, $min^{-1}$ |
|---|---|---|---|---|---|---|
| Control (0.0) | 1.4 | 32.7 | 31.2 | 1.1 | 2.2 | 90.9 |
| Bio-NBR (2.5) | 1.6 | 32.6 | 31.1 | 1.2 | 2.5 | 76.9 |
| Bio-NBR (5.0) | 1.5 | 30.5 | 29.0 | 1.4 | 2.8 | 71.4 |
| Bio-NBR (7.5) | 1.2 | 29.2 | 28.0 | 1.5 | 3.2 | 58.8 |
| Bio-NBR (10.0) | 1.3 | 28.0 | 26.8 | 1.6 | 3.6 | 50.0 |
| CO-NBR (2.5) | 1.5 | 31.2 | 29.7 | 1.2 | 2.4 | 83.3 |
| CO-NBR (5.0) | 1.2 | 29.1 | 27.9 | 1.3 | 2.5 | 83.3 |
| CO-NBR (7.5) | 1.3 | 27.6 | 26.3 | 1.3 | 2.7 | 71.4 |
| CO-NBR (10.0) | 1.4 | 27.4 | 26.0 | 1.3 | 2.7 | 71.4 |

minimum torque ($M_L$), which indirectly indicates the total crosslink density of a rubber compound [23]. In general, the delta torque decreased with increasing loading of process aids, which is in line with previous literature [23–26]. Process aids could form layers between rubber chains that coat and absorb curatives and fillers, which reduce the number of available curatives and fillers for crosslinking [23]. The delta torque was slightly higher with the addition of Bio compared to CO possibly contributed by the better compatibility of Bio with NBR due to its polarity. From Table 2, the scorch time ($T_{S2}$) and cure time ($T_{90}$) increased with further addition of process aids. Changes in $T_{S2}$ and $T_{90}$ were more prominent with Bio. A similar observation was seen with the cure rate index (CRI), whereby the reduction in CRI was more prominent with Bio. These findings suggest that Bio offers safer processing time and slower curing process compared to CO. Ismail et al. [27] also found that scorch times increased with increasing palm oil fatty acid concentration. One possible reason is due to the formation of chelates between zinc ion complex and the accelerator, immediate reaction products, or crosslink precursors during vulcanisation process [27], which retard the curing process.

## Dynamic mechanical analysis

Dynamic mechanical analysis is important in determining the viscoelastic behaviour of the composites. Figure 6 shows the variation of the storage modulus ($E'$) of NBR composites as a function of temperature. Storage modulus is influenced by the interactions in the polymer [28] and also the molecular mobility of chains above the $T_g$ [29]. It is apparent that in the glassy region, the storage modulus decreased with increasing process aid. This softening effect may be attributed to the weakening of intermolecular interactions of rubber chains by process aid. All NBR composites showed a single transition implying the absence of immiscible phase.
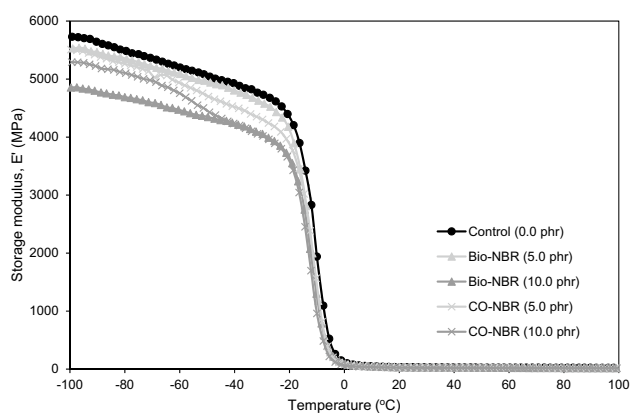
**Fig. 6** Storage modulus of NBR vulcanisates as a function of temperature

It is a well-known fact that process aid improves the processability of rubber by lowering the $T_g$. $T_g$ is usually the temperature taken at the maximum peak of loss tangent curves. Figure 7 displays the loss tangent of NBR composites as a function of temperature and shows that the $T_g$ of NBR composites with process aids slightly shifted to a lower temperature compared to the control sample. Nonetheless, there was no significant difference in $T_g$ between composites with process aids. The maximum loss tangent values of control and CO-NBR composites were similar, whereas the maximum loss tangent value of Bio-NBR composites was reduced as the concentration of Bio increases. Due to the presence of esters and carboxyl groups, Bio can interact with carbon black and NBR due to polar-polar interactions. This could cause micro-plasticisation of rubber–filler, which reduces the amount of heat generated due to shearing at the rubber–filler interface, hence, leads to better dispersion and rubber–filler interaction and lower loss tangent values [25].

## Tensile properties

The results of tensile properties are depicted in Table 3. Tensile test is usually used in many comparative studies
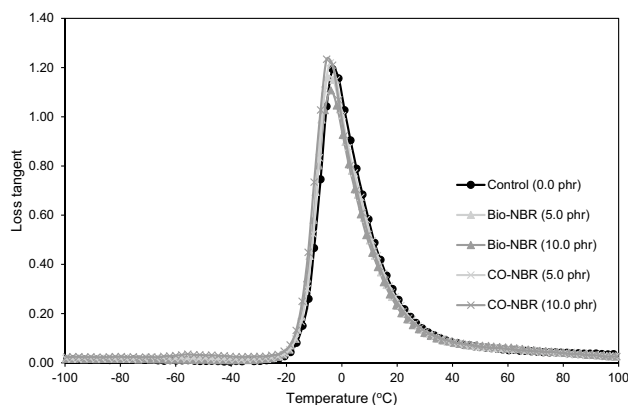
**Fig. 7** Loss tangent of NBR vulcanisates as a function of temperature

**Table 3** Tensile properties of unaged NBR composites

| Process aid loading (phr) | M100 (MPa) | TS (MPa) | EB (%) |
|---|---|---|---|
| Control (0.0) | $5.8 \pm 0.2$ | $14.8 \pm 1.4$ | $220 \pm 16$ |
| Bio-NBR (2.5) | $5.0 \pm 0.1$ | $13.8 \pm 1.0$ | $228 \pm 31$ |
| Bio-NBR (5.0) | $4.9 \pm 0.1$ | $14.3 \pm 0.7$ | $241 \pm 13$ |
| Bio-NBR (7.5) | $4.8 \pm 0.1$ | $12.1 \pm 1.2$ | $221 \pm 19$ |
| Bio-NBR (10.0) | $4.8 \pm 0.1$ | $12.4 \pm 0.5$ | $224 \pm 13$ |
| CO-NBR (2.5) | $5.4 \pm 0.5$ | $14.5 \pm 1.8$ | $228 \pm 32$ |
| CO-NBR (5.0) | $4.8 \pm 0.1$ | $14.4 \pm 0.8$ | $252 \pm 25$ |
| CO-NBR (7.5) | $4.3 \pm 0.2$ | $13.6 \pm 1.1$ | $258 \pm 13$ |
| CO-NBR (10.0) | $4.5 \pm 0.1$ | $13.6 \pm 0.6$ | $250 \pm 11$ |

to evaluate the properties of composites. Modulus at 100% elongation (M100) indicates the stiffness of rubber composites. It can be seen from Table 3 that a further increase in process aids decreased the M100 of rubber composites, which is due to the reduction in crosslink density. Significant discrepancies of M100 can be observed after 7.5 phr of process aid loadings where the Bio-NBR composite showed higher M100 values compared to CO-NBR composite. These results are expected since the crosslink density of Bio-NBR composite was higher than CO-NBR composite, possibly due to the better compatibility of Bio with NBR rubber. Higher modulus of Bio-NBR composite suggests that the composite is stiffer, which also explains the lower stretching ability or elongation at break (EB) and tensile strength (TS) of Bio-NBR composite compared to CO-NBR composite. Nonetheless, it is worth noting that the differences in tensile strength and elongation at break of Bio- and CO-NBR composites at the same process aid content were not significant.
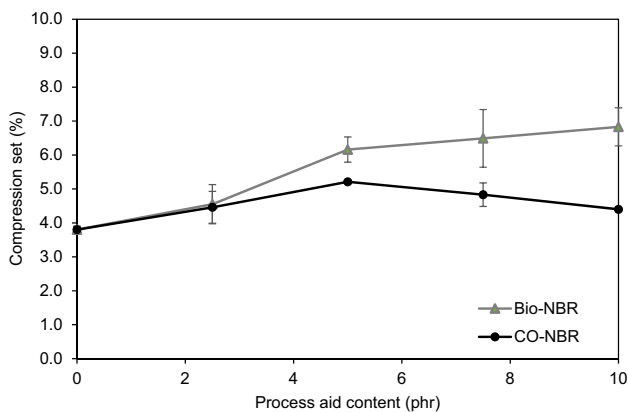
## Compression sets

Compression set measures the ability of rubber to recover to original thickness after prolonged compression at a specific condition. Lower compression set implies better resistance to deformation. It is an important parameter to evaluate the service period of a product. The compression sets of carbon-black NBR composites are depicted in Fig. 8. The compression sets increased with increasing process aid content, which is attributed to the reduction in crosslink density. The compression sets of Bio-NBR composites were also higher than CO-NBR composites. In other words, the addition of Bio resulted in higher deformation of rubber compared to CO. This could be because of the higher plasticising effect of Bio compared to CO on the NBR composites.

## Ageing properties

Ageing properties are important as they provide an insight into the performance of the composites during service. Table 4 compares the retention percentage of tensile properties and compression set of aged NBR composites using CO and Bio as process aids. The results in Table 4 could be interpreted in several ways. First, in general, the M100, TS, and compression sets of all NBR composites increased while EB decreased, suggesting stiffening of rubber after ageing. These could be due to the dominance of crosslinking reaction over chain scissions of NBR chain during accelerated ageing. It was reported that the crosslinking effect is commonly observed in NBR [26]. When oxygen reacts with rubber, chain scission and crosslinking occurred. Chain scission of a polymer will deteriorate the tensile strength and increase elongation at break, whereas the crosslinking of the polymer leads to stiffening of the rubber in which the modulus and hardness increase [26]. Second, the fluctuation of tensile properties as a function of process aid content can also be observed. These inconsistencies occurred as a result of the balancing between crosslinking and chain scissions reaction during ageing of rubber [30]. Another interesting



**Fig. 8** Compression sets of NBR composites after 24 h at room temperature

**Table 4** Retention percentage of tensile properties and compression sets of aged NBR composites

| Process aid loading (phr) | Retention of M100 (%) | Retention of TS (%) | Retention of EB (%) | Compression set (%) |
|---|---|---|---|---|
| Control (0.0) | 187.9 | 94.6 | 55.9 | 14.5 ± 1.0 |
| Bio-NBR (2.5) | 193.8 | 99.9 | 58.3 | 12.2 ± 1.5 |
| Bio-NBR (5.0) | 192.3 | 90.5 | 53.1 | 11.8 ± 0.6 |
| Bio-NBR (7.5) | 195.6 | 107.8 | 59.3 | 10.9 ± 0.2 |
| Bio-NBR (10.0) | 182.1 | 108.4 | 63.4 | 11.4 ± 1.1 |
| CO-NBR (2.5) | 212.0 | 106.5 | 55.7 | 8.1 ± 1.4 |
| CO-NBR (5.0) | 207.6 | 101.2 | 53.2 | 11.0 ± 0.3 |
| CO-NBR (7.5) | 217.1 | 102.7 | 53.1 | 13.1 ± 0.6 |
| CO-NBR (10.0) | 193.5 | 96.7 | 54.8 | 14.7 ± 1.1 |

finding is at 2.5 phr concentration of process aids, the compression set markedly improved, especially with CO-based composite. This could be attributed to the higher crosslink density, as indicated by the M100 value at the same concentration. Nonetheless, with increasing process aid content, the compression sets of CO-NBR composites deteriorated, whereas the compression sets of Bio-NBR composites continued to improve. The contradicting trends of Bio- and CO-NBR composites on compression sets could be related to the compatibility of the process aid with aged rubber and filler. The oxidation of nitrile rubber produced carboxyl and hydroxyl species in the rubber chains [20] that may improve its compatibility with Bio hence, resulting in a lower compression set.

## Thermogravimetric analysis

Thermogravimetric analysis describes the thermal and oxidative stabilities of materials and their compositional properties. As depicted in Figs. 9 and 10, the weight loss and derivative (DTG) curves of control composite showed only one degradation stage in the temperature range of 250–500 °C, which was due to the degradation of the rubber. In contrast, CO- and Bio-NBR composites exhibited two degradation stages, which are at 250–300 °C and 350–500 °C. Degradation of these NBR composites at the first stage could be attributed to the degradation of process aids. It is also apparent that composites with process aids had slightly higher weight losses compared to control. An increasing amount of process aids resulted in a further increase in weight loss. Process aids lower the molecular interactions between rubber chains hence, the energy needed to break the interactions leading to a higher degradation. From Fig. 9, the CO-NBR composite with 10.0 phr process aid had the lowest thermal stability compared to other NBR composites, suggesting a slightly better thermal stability of Bio-NBR composites.
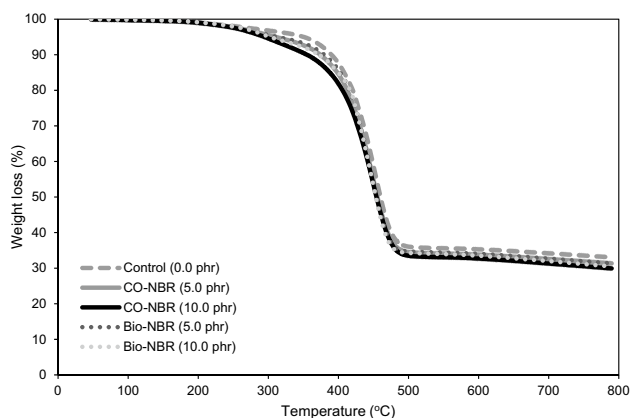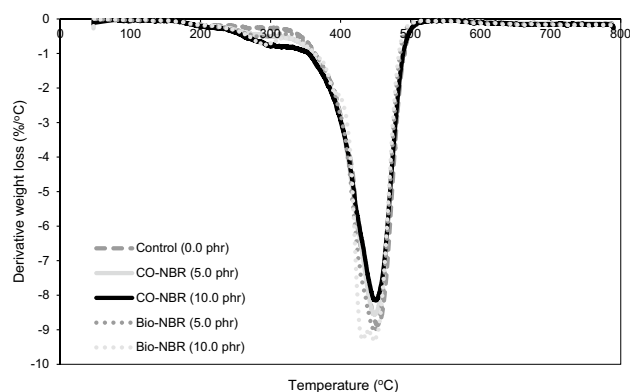


**Fig. 10** DTG curves of NBR composites

## Conclusion

This study sets out to investigate the efficacy of a palm-based (Bio) process aid by comparing its performances with a commercial oil (CO), i.e. naphthenic oil in terms of processability, physical properties, and chemical properties of nitrile rubber composites. The findings of this study show that the use of Bio increases the processability of NBR compounds by having a lower Mooney viscosity, better filler dispersion (as shown in TEM images), and lower loss tangent values compared to CO-NBR composites. In contrast to unaged samples, Bio-NBR composites showed better retention of tensile properties and elastomeric property after accelerated ageing, especially at higher concentrations of Bio compared to CO-NBR composites, possibly attributed to the better compatibility of Bio with aged rubber. The use of Bio also resulted in better thermal stability compared to CO-NBR composites. Based on these findings, Bio ($\geq 7.5$ phr) could potentially replace petroleum-based process oil, in this case, naphthenic oil, as it offers better processability of rubber compounds and improved ageing resistance of composites, which is desirable in various applications such as in the automotive industry.

## Compliance with ethical standards

**Conflict of interest** The authors have no conflict of interest.



**Fig. 9** Weight loss curves as a function of temperature

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