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RESEARCH ARTICLE

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Eco-friendly rubber compounds: Utilizing animal lard as a plasticizer oil

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Abstract

Melt-mixing technique was used to compound eco-friendly plasticizer, pork fat oil (PF) and natural rubber (NR)-carbon black (CB) and studied systematically. The results obtained for the PF-compounds (PFC) were compared to petroleum oil compounds (POC) and Shea-butter oil compounds (SBC). While some properties of the PFC were similar and favored those of the POC and SBC, the PFC generally demonstrated superior physico-mechanical properties. For instance, the optimum cure time (T_{90}) of PF5 and PF40 reduced to about 225 and 276% when compared to POC5 and POC40 and 150 and 183% relative to SBC5 and SBC40, respectively. The bound rubber content (BRC %) (filler-matrix interactions) of PF5 was over 15% and 5% greater than POC5 and SBC5, respectively. Also, the PF5 outperformed POC5 by 12% in crosslinking density and by 7% in rebound resilience (%). The PF5 and PF40 further showed improvements in fatigue life cycle, exceeding POC5 and POC40 by 40% and 75%, respectively. These enhancements were attributed to the effective distribution of CB particles facilitated by compatible PF within the NR matrix. Therefore, PF has been demonstrated to be an environmentally friendly, cost-effective, and compatible plasticizer for rubber compounding compared to POC and vegetable oil, predominantly as an activator for lowering production time.

Highlights

- Natural rubber was compounded with carbon black, with pork fat as a plasticizer.
- The pork fat compounds (PFC) showed superior physico-mechanical properties.
- The properties were better than aromatic and vegetable oil (VO) compounds.
- The fat oil was demonstrated to be a suitable and eco-friendly plasticizer.
- The fat can substitute carcinogenic oils and prevent using VO as a plasticizer.

KEYWORDS

aromatic oil, composites and fatigue resistance, natural rubber, plasticizer, pork fat oil

1 | INTRODUCTION

Natural rubber (NR), poly (2- methyl- 1,3- butadiene) is a vital raw material in many industries, with applications ranging from sports equipment, laboratory devices, automobile parts (tires, seals, tread, shock absorbers), and high-pressure applications in the oil and gas industry, and many more.[1–4] This is due to its high resilience and oil resistance, etc. However, the unsaturated sites (—C=C—) in the main chain of NR make it vulnerable to degradation accelerated greatly by heat, humidity, ozone radiation, etc.[5] Another drawback of NR is its high cost and poor rolling and traction performance.[6] To overcome some of these challenges, NR with synthetic elastomers has been explored nowadays Examples of these blends include acrylonitrile butadiene rubber (NBR),[7,8] ethylene-propylene-diene monomer rubber (EPDM),[9] Styrene-butadiene rubber (SBR),[8,10] and poly-butadiene rubber (BR)[2,11] to form dual or ternary blends. In many cases, these blends are formed for a combination of good properties from each blended component. However, in the virgin state of these blends (whether dual or ternary blends), they are not really useful for certain applications requiring high physico-mechanical properties. Therefore, the pure matrix has to be reinforced with high filler content (≤5–70 phr), sometimes single or blended fillers, in order to achieve high physico-mechanical properties.[12–15] Some of these fillers used include nanoclays, carbon blacks (CB) and nano-fiber, graphenes, and carbon nanotubes (CNT).[16–20] Nevertheless, reinforcing elastomers (single, dual, or ternary blend matrix) at high concentrations leads to high viscosity and high hardness, resulting in poor dispersion of fillers and consequently in high energy consumption during the compounding process.[21–23] Efforts have been made to address these challenges by including softeners or plasticizers in order to reduce the viscosity and ensure effective filler dispersions, thereby lowering the energy required for compounding.[24–27]

Plasticizers are characteristically derived from mineral oils and are often classified into aromatic, naphthenic, and paraffinic structures.[24,25] Plasticizers improve the processability and flexibility of rubber compounds by reducing intermolecular interactions in rubber chains and subsequently lowering the glass transition temperature.[28] Petroleum-based oils (PO), also known as extender oils, are the most commonly used plasticizer in the rubber processing industry owing to their low cost, good compatibility with most rubbers, as well as their effectiveness in improving the matrix-filler interactions.[1–4] However, the PO have been documented to pose significant environmental risks; they exhibit toxicity and possess carcinogenic properties that can adversely impact human health.[24,25,29,30] A sustainable and economically viable alternative to PO for rubber processing is urgently needed to address these threats.

In this regard, there has been extensive research into vegetable oils (VO) like soybean oil,[6,31,32] palm oil,[27,33–35] castor oil,[36] and sunflower oils[37] for compounding elastomers, due to the fact that they are environmentally friendly, have low cost, and are biodegradable.[38,39] For example, Pechurai et al.[39] used castor and jatropha oils to replace aromatic oils in the processing of Styrene Butadiene Rubber (SBR) with improvement in the rebound resilience and abrasion resistance properties. The Mensah group also confirmed recently that Shea butter oil[26] and palm oil[33] were suitable for compounding NR—S_x—CB compounds with incredible physico-mechanical properties, as compared to PO. Future competition in the food chain is, however, of greater concern should VO become widely adopted for the rubber processing industry. Therefore, alternative oils, like unused animal fats or lard, could be part of the plan. Animal fats, especially pork lard (PF) consumption is on the decline as people have become more health conscious about food intake and quality these past few decades.[40] The carcasses of pigs can contain about 10%–30% fat in the adipose tissues.[41] A study conducted in the 1990s indicated the production of Lard (rendered pork fat) rising to about 5 million metric tonnes per year, most of which go to waste.[41]

Despite extensive research on rubber compounds, literature specifically addressing the use of pork fat oil as a plasticizer for compounding remains limited, indicating a potential knowledge gap. Thus, this present study seeks to provide an alternative rubber processing aid in the form of unused pork fats (PF) and study its effectiveness compared to the traditional Treated Distillate Aromatic Extract (TDAE) and vegetable oil (Shea-butter-based compounds). To do this, varying concentrations of pork fat oil (PFO) were incorporated into natural rubber (NR)-reinforced carbon black (CB) prepared by an internal mixer and two-roll mill approach. The compounds were cured with a hot-press machine in a rectangular mold, a day after rheo-curing the samples at 160°C. The physico-mechanical properties of pork fat compounds (PFC), which include thermodynamics of mixing, vulcanization properties, tensile strength, crosslinking density, and fatigue life cycle, etc., were all conducted and the results obtained were compared with those of petroleum oil compounds and those of Shea-butter oil compounds (SBC).

2 | EXPERIMENTAL

2.1 | Chemicals and compound formulation

Natural rubber (NR), (SMR-CV60, Standard Malaysian Rubber) was sourced from Doo Man Co. Ltd. in South Korea. Carbon black N550 (specific surface area: 95–105 m^2/g) and aromatic oil (TDAE) were procured from IDONG TECH. The curing ingredients, activators: zinc oxide (ZnO), stearic acid (SA), crosslinking agent: sulfur (S), and accelerators: tetramethyl thiuram disulfide (TMTD) and N-cyclohexyl-2-benzothiazole sulfonamide (CZ), were obtained from Info Chem Company Ltd. Waste pork fat-containing meat fragments were collected from a local market in Ghana.

2.1.1 | Preparation of pork fat oil and compound formulation

The collected Pork fat (PF) was heated into oil in the lab at ≥100°C by using a heating system for about ~10 min to ensure it was moisture free. The oil was sieved to ensure the PF oil was clean from any impurities. The PF oil was then allowed to harden overnight. The absence of water moisture at the surface of the oil was used as evidence of complete dryness. The pork fat oil (PFO) content in the prepared samples was expressed as parts per hundred parts of rubber (phr), as detailed in Table 1. The composition, which includes sample prepared without oil (PF0), Shea-butter oil compounds (SBC), and petroleum-based oil compounds (POC), was all collected from the previous report by Mensah et al.[26] to be compared with those of PFC.

2.2 | Processing of the rubber compounds

The samples were compounded using a kneader (model: QPBV-300, QMESYST, South Korea) at 90°C and 30 rpm. The elastomers were initially masticated for approximately 1 min, followed by the addition and mixing of other processing aids for approximately 2 min. Carbon black (CB) filler was then incorporated and mixed for another minute. Pork fat oil (PFO) was gradually added to facilitate mixing and to ensure effective dispersion of CB within the rubber compound. Sulfur was the final ingredient, added and mixed for an additional 30 s. The compounded materials were subsequently re-mixed using a two-roll mill (QM300, QMESYSTEM) to achieve better filler dispersion mixing. By repeatedly passing the samples through the mill for approximately 8 minutes, the desired compounded targets were met, and the samples were sheeted out. Optimum curing conditions, determined using a cure rheometer machine (MDR), were employed to vulcanize the compounds in a hot press machine (model: TO-200, TESTONE. South Korea) operating at a pressure of 25 tons and 160°C. Rectangular-shaped metallic Molds with dimensions of 15 cm × 2 mm were used. After cooling for a day, the vulcanized samples were cut into standard sizes for characterization, which included Fourier transform infrared

TABLE 1 Compound formulation measured in (phr)[a] and their respective designation.

Chemicals	PF0[a]	PF5	PF10	PF20	PF40	SBC[b]	POC[c]
NR	100	100	100	100	100	100	100
ZnO	5	5	5	5	5	5	5
TMTD	1	1	1	1	1	1	1
CZ	0.5	0.5	0.5	0.5	0.5	0.5	0.5
S/A	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Sulfur	2.1	2.1	2.1	2.1	2.1	2.1	2.1
N550	50	50	50	50	50	50	50
PF	-	5	10	20	40	-	-
SBO*	-	-	-	-	-	5 & 40	-
TDAE oil**	-	-	-	-	-	-	5 & 40
Wax	1	1	1	1	1	1	1

Note: phr[a] (parts per hundred parts of rubber), PF (pork fat oil). The SBO* (Shea butter oil) and TDAE oil[b] (treated distillate aromatic extract) Mensah et al.[26a–c] spectroscopy (FTIR), vulcanization analysis, crosslinking density, tensile strength, flex-fatigue life, resilience, and hardness (Shore A) analysis.

3 | CHARACTERIZATION

3.1 | FT-IR of pork fat oil plasticized NR-CB compounds

The Fourier transforms infrared spectrometer (FTIR) (Brucker Alpha) spectra of the pork fat oil plasticized NR—S_x—CB compounds were recorded. The scan rate used was 0.1 V·s^{-1} at a spectral range of 400–4000 cm^{-1} and 32 number of scans. The various chemical groups present were interpreted and presented '1'.

3.2 | Vulcanization properties of pork fat oil plasticized NR-CB compounds

The influence of pork fat oil (PFO) plasticizer on the vulcanization behavior of NR—S_x—CB compounds was evaluated using a cure rheometer (MDR, model: PDR2030, TESTONE. Ltd., South Korea) operating at 160°C. The cure rheometer recorded various curing properties for the tested compositions, including: the maximum torque (M_H), minimum torque (M_L), difference in torque ($\Delta M = M_H - M_L$), start of cure time (t_{s2}), optimum cure time (t_{90}), and cure rate index ($CRI = 100/[t_{90}\text{-}t_{s2}]$).

3.3 | Thermodynamics parameters

3.3.1 | Gibbs free energy

The change in Gibbs free energy (ΔG) was calculated using the Flory-Huggins equation.[42]

$$\Delta G = \left[In(1-\nu_r)+\nu_r+\chi_1\nu^2{}_r\right] \tag{1}$$

where R is the universal gas constant, V_r, T is the absolute temperature, and χ^1 is the polymer-solvent parameter.

3.3.2 | Change in entropy

From the statistical theory of rubber elasticity, the conformational entropy ΔS is calculated by the following:

$$\Delta S = -\frac{\Delta G}{T} \tag{2}$$

where ΔS is the entropy, T is temperature(K), and the d change in Gibbs' s free energy is G.

3.4 | Mechanical Characterizations

3.4.1 | Tensile properties of pork fat oil plasticized NR-CB compounds

The ultimate tensile strength (UTS) and elongation at break, E_{br} (%) are two important mechanical properties of materials, often measured in tensile tests. The vulcanizates prepared with pork fat oil (PFO) plasticizer were evaluated for their tensile properties according to ASTM D412 standards using a universal testing machine (QM100s, QMESYSTEM, South Korea) at a crosshead speed of approximately 500 mm/min and a temperature of approximately 25°C. The ultimate tensile strength (UTS) was determined as the stress at fracture, while the corresponding strain was recorded as the elongation at break, E_{br} (%). To ensure accuracy and reliability, three samples were tested for each composition. The obtained results were then averaged to provide a representative value for each property.

3.4.2 | Crosslinking density of pork fat oil plasticized NR-CB compounds

The various vulcanizates were equilibrated in toluene at ~25°C for ~48 h. The degree of swelling (Q_r) of the vulcanizates was calculated by using Equation (3)

$$Q_r = \frac{W_s - W_i}{W_d} \tag{3}$$

The initial weight of the sample was denoted as W_i, while W_s and W_d represent the weights in the swollen and oven-dried states, respectively (drying conditions: 80°C, 8 h). The network chain density (N_c) was determined using the Flory–Rehner Equation (4).[43]

$$-\left[In(1-\nu_r)+\nu_r+\chi_1\nu^2{}_r\right] = \nu_s\left[\nu_r{}^{1/3}-\nu_r/2\right]N_c \tag{4}$$

where V_r is the volume fraction of the polymer matrix in the equilibrium swollen gel denoted as V_s. The solvent's molar volume (toluene) was 106.3 mL/mol, and the polymer-solvent parameter (χ1) was calculated to be 0.374 using the Bristow-Watson Equation (5).[44]

$$\chi_1 = \beta_1 + (\nu_r/RT)\left[\delta_s - \delta_p\right]^2 \tag{5}$$

The lattice constant (β) is 0.34, R represents the universal gas constant, T is the absolute temperature, and δ is the solubility parameter. The subscripts *s* and *p* denote the solvent and polymer sample, respectively. The solubility parameters of NR and toluene are 8.4 and 8.1 $(\text{cal/cc})^{1/2}$, respectively.

3.4.3 | Bound rubber analysis of uncured PF oil-plasticized NR-CB compounds

The study investigated the impact of pork fat oil (PF) plasticizer on the bound rubber content (BRC) of uncured NR—S_x—CB systems. Unbound rubber was extracted using toluene solvent. Approximately 1.0 g of uncured NR—S_x—CB composite enclosed in fabric was submerged in 300 mL of toluene for a week at 25°C. The solvent was replaced every two days. After seven days, the swollen samples were removed, oven-dried at 70°C for 12 h, and weighed when cool. The BRC (%) entrapped by the reinforcements in each NR—Sx—CB composite was calculated using the widely employed Equation (6).[45,46]

$$BRC(\%) = \frac{w_{fg} - \left[w_t\left(\frac{m_f}{m_f + m_r}\right)\right]}{w_t\left[\frac{m_r}{m_f + m_r}\right]} \times 100 \tag{6}$$

where BRC (%) is the content of bound rubber, W_{fg} is the weight of CB and gel, and W_t is the weight of composite. The m_f and m_r are the phr of CB and NR rubber in each composition, respectively.

3.4.4 | Flex-fatigue behavior

The flex-fatigue life of different pork fat oil-filled NR—S_x—CB compounds was evaluated using a De-Mattia flexing machine (QM 650D) according to ASTM D430 specifications. The thickness of the samples was measured near the groove. The test specimens had dimensions of 150 × 25 × 6.35 mm with 2.39 mm radius grooves, similar to those previously described by Mensah et al.[2] The samples were conditioned at 23 ± 1°C for at least 12 h and subjected to 300 ± 10 cycles/min. The average flex-fatigue life value for three tested samples was recorded for each composition. Flex-fatigue life was measured until a noticeable grade-one crack appeared.

3.4.5 | Rebound resilience

The elastic properties (rebound resilience, Rb%) of pork fat oil (PF) plasticized NR—S_x—CB were investigated using a DIN resilience device (DIN 53512 and ISO 4662), QMESYS, obtained from YUNTECH Co. Ltd., South Korea. The cylindrical samples had a thickness of approximately 12 mm and a diameter of approximately 16 mm. The methods used are described in detail in the previous report by Mensah et al.[2] The hammer part of the instrument was initially used to strike the sample surface five times to eliminate internal defects. As per DIN 53512 and ISO 4662 standards, hits after the fifth may be used to represent rebound resilience. In this study, the seventh hit was recorded as the rebound resilience value.

3.4.6 | Hardness (Shore A) test

The influence of pork fat oil (PF) as a plasticizer on the hardness of NR—S_x—CB vulcanizates was evaluated using a constant load hardness tester (Shore A, QMESYS, YUNTECH Co. Ltd., South Korea) with a 5 kg load, according to ASTM D2240 and ISO 48-4 standards for rubbers. Three samples of approximately 5 mm thickness were tested for each composition, and the average hardness was recorded.

3.5 | Thermal degradation property

Thermal gravimetric analysis (TGA) of pork fat oil (PF) plasticized NR—S—CB vulcanizates was conducted using an SDT Q600-TA instrument from the Department of Material Science and Engineering, University of Ghana. The analysis was performed under nitrogen, at an equilibrium temperature of approximately 25°C, and with a heating rate of 10°C/min to a maximum temperature of approximately 800°C. The weight residue (%) and derivative temperatures (DTG) were used to assess the thermal resistance properties of the compounds.

4 | RESULTS AND DISCUSSION

4.1 | FTIR spectrum of pork fat compounds

The FTIR spectrum of animal lard (Pork, cow, chicken, etc.) was earlier reported by Man and Guntarti et al.[47,48] and recently by Sahar et al.,[49] who recently also reported on the physicochemical properties of pork lard, beef, and chicken by using the FTIR technique.

Usually, peaks around 3009 cm^{-1} were linked to Cis C=CH vibrations; this was not observed when the pork lard was incorporated into NR-CB vulcanizates, as shown in Figure 1. The bands from 2953 to 2910 cm^{-1} are

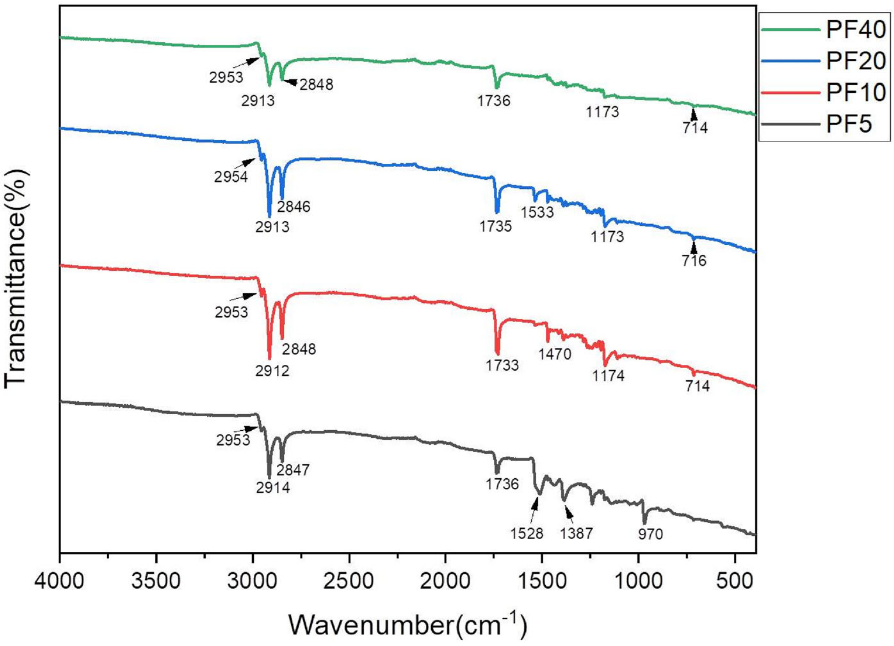

FIGURE 1 FTIR spectra of the effect of PF loading on NR—CB compounds.

related to the C–H bonds in the isoprene chain and symmetric stretching vibrations in the methylene group (-CH_2) in the lard.[48] The band at 2848 cm^{-1} represents the symmetric and asymmetric stretching vibrations of the methylene group (-CH_2).[48] The 1735 cm^{-1} band represents the C=O carbonyl group of esters (probably coming from the stearic acid) or the esters connected to triacylglycerol in the lard.[47,48] This has another stretch of C-O at 1174 cm^{-1}. Vibration bands at 1538 cm^{-1} are associated with amide II.[47,48] The band at 1460–1470 cm^{-1} comes from the bending vibration of the aliphatic CH_2 and CH_3 clusters.[48] The overlap vibrations of CH_2 with vibrations out of the field on cis-substituted olefins are represented by the bands at 714–720 cm^{-1}.[48]

Previous studies have confirmed the presence of numerous fatty acids in PF; these include oleic acid (40%–60%), stearic acid (20%–50%), linoleic acid (3%–11%), palmitic acid (2%–9%), linolenic acid (<1%), and arachidic acid (<1%).[50–52] These moieties are likely to act as activators in speeding the vulcanization reaction process, as stearic acid is generally recognized for enhancing the cure reactions and consequently the physico-mechanical properties of rubber vulcanizates.[53–55]

4.2 | Bound rubber analysis

The bound rubber content, BRC(%) analysis is a commonly used method to understand the filler-rubber matrix interactions in the composite's uncured state.[45] The BRC (%) plays an important role in vulcanization and other physico-mechanical properties of the resulting vulcanizates. The BRC (%) in the uncured compounds for PFC is compared with those of SBC and POC earlier reported[33] in Figure 2.

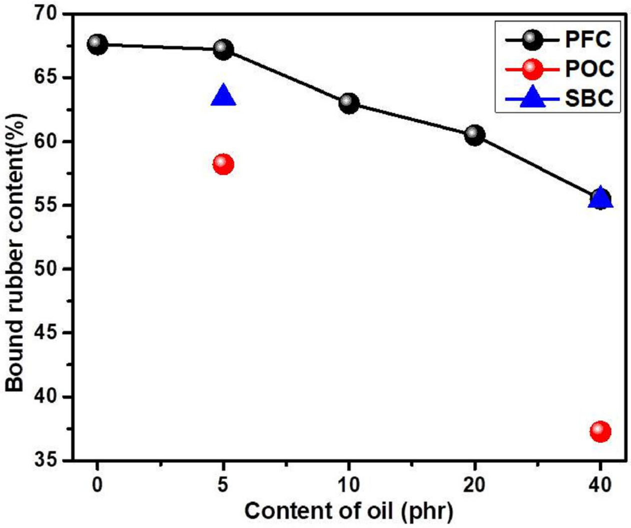

FIGURE 2 Effect of plasticizer oil type and loading on BRC (%) of the composites.

Although plasticizer improves filler dispersions within the rubber matrix, a reduction in the BRC (%) is observed, especially as the plasticizer content is elevated from 0 to 40 phr for all the compositions, depicting the weakening of structures like NR—S_x—CB and CB—CB present in the composites. A comparable observation was earlier made for rubber-CB-Soybean oil[56] and rubber-CB-epoxidized oil[57] respectively, where filler-matrix declined by the incorporation of the plasticizer oils into the matrices. Even so, the PF5, although slightly lagged behind the PF0 by ~0.7 BCR%, however, recorded over 15 and 5% BRC (%) than POC5 and SBC5, respectively, while at 40phr of plasticizer, the PF40 obtained about ~49 and ~0.2% higher BRC than its counterparts POC40 and SBC40 respectively.[26] The BRC (%) is dependent on several conditions such as the surface area of reinforcing fillers, their structure and surface activity, cure ingredients used, rubber matrix involved, and the solvent used for the BRC experiment.[45] However, it was interesting to report that PFC, POC, and SBC have demonstrated superior BRC (%) than NR-CB (50phr) (660c) and NR-CB (50phr) (550c) composites, which obtained BRC (%) of ~11.3% and ~ 16.7% respectively.[58] The current composition has also shown better BRC (%) than BR-CB(N330) and SBR-CB(N330) with ~23% and ~25% values of BRC (%) respectively.[59] Therefore, based on the BRC%, the PF has demonstrated a high capability of promoting improved compatibility between CB and NR as compared to its counterparts (POC and the SBC)[26] and those earlier reported. The BRC% of PFC could also influence other physico-mechanical properties.

4.3 | Thermodynamics of mixing: Gibb's free energy and entropy

There was a scattering of data for the results obtained for the entropy (J/mol/K) as shown in (Figure 3A). The general trend of decrease is written as follows: PF5 > PF0 > SBC5 > PF10 > POC5 > SBC40 > POC40 > PF20 > PF40. Thus, it can be seen that lower level (0 ~ 10 phr) of the plasticizer (whether animal lard, vegetable, or petroleum oil) yielded higher degree of randomness (). This may be associated with high mixing among the chains, among the fillers (CB) and between the chains and the CB than the compounds with highly loaded plasticizers (10 ~ 40 phr). Among the compositions, the attained by PF5 was the, highest and in magnitude, PF5 obtained 28% and 43% higher than its counterparts; SBC5 and POC5 respectively.[26] Meanwhile, the values obtained for the present care positions is higher compared to those reported by Doma et al.[42] for SBR/NBR blend, which recorded values of 0.06 ~ 0.19 (J/mol/K). A high disorder may lead to an effective blending of NR and CB and their interactions, especially in the presence of small amount of the plasticizers. In terms of stability, a more stable thermodynamic system can be the observed when free energy value (ΔG) < 0.[42] In Figure 3B, all the compounds have generally exhibited stable phases and ΔG is seen increasing with increasing pork fat oil (PF5 > PF10 > PF20 > PF40); with the pork lard based compounds (PF40 and PF20) showing the more negative ΔG or spontaneous phase than the rest of the compounds. Thus, higher loading of the plasticizer oil (20 ~ 40phr) seems to have produced a more spontaneous phase than the lower content of oil compounds.

4.4 | Curing properties of compounds

4.4.1 | Cure time and cure rate index (CRI)

The representative rheo-curves of the plasticized NR—S_x—CB compounds are shown in Figure 4A, while the effects of Pork fat (PF), Shea-butter oil (SBO) and aromatic or petroleum-based oil (PO) on the vulcanization properties, cure times (t_{s2}, & T_{90}) and (CRI) $[100/(T_{90}\text{-}t_{s2})]$

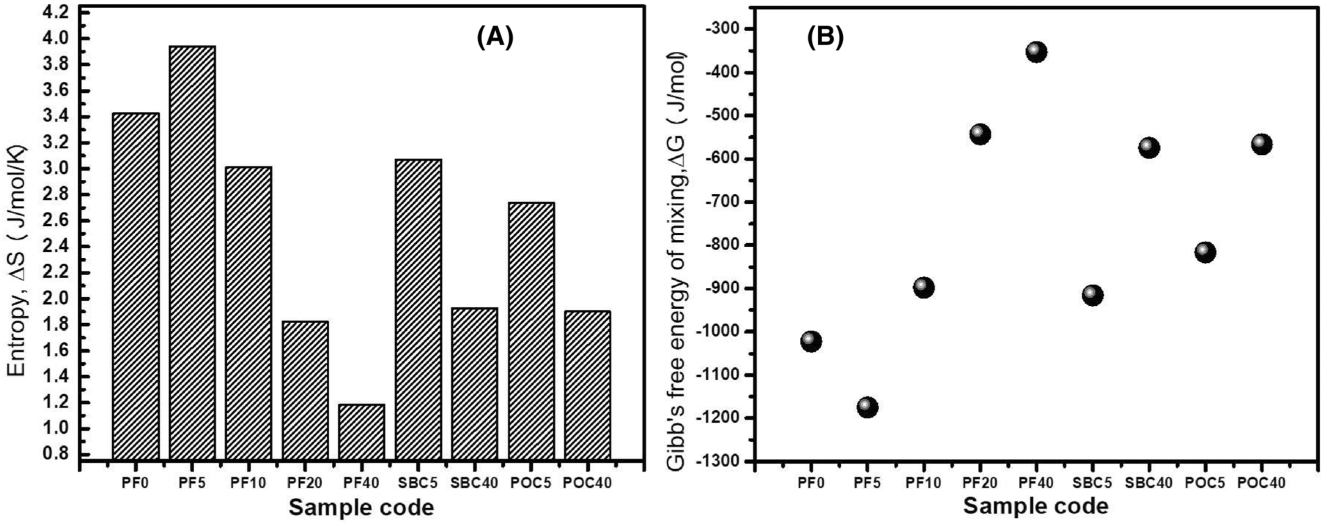

FIGURE 3 Thermodynamic parameters; (A) entropy of mixing, ΔS, and (B) Gibbs' s free energy ΔG.

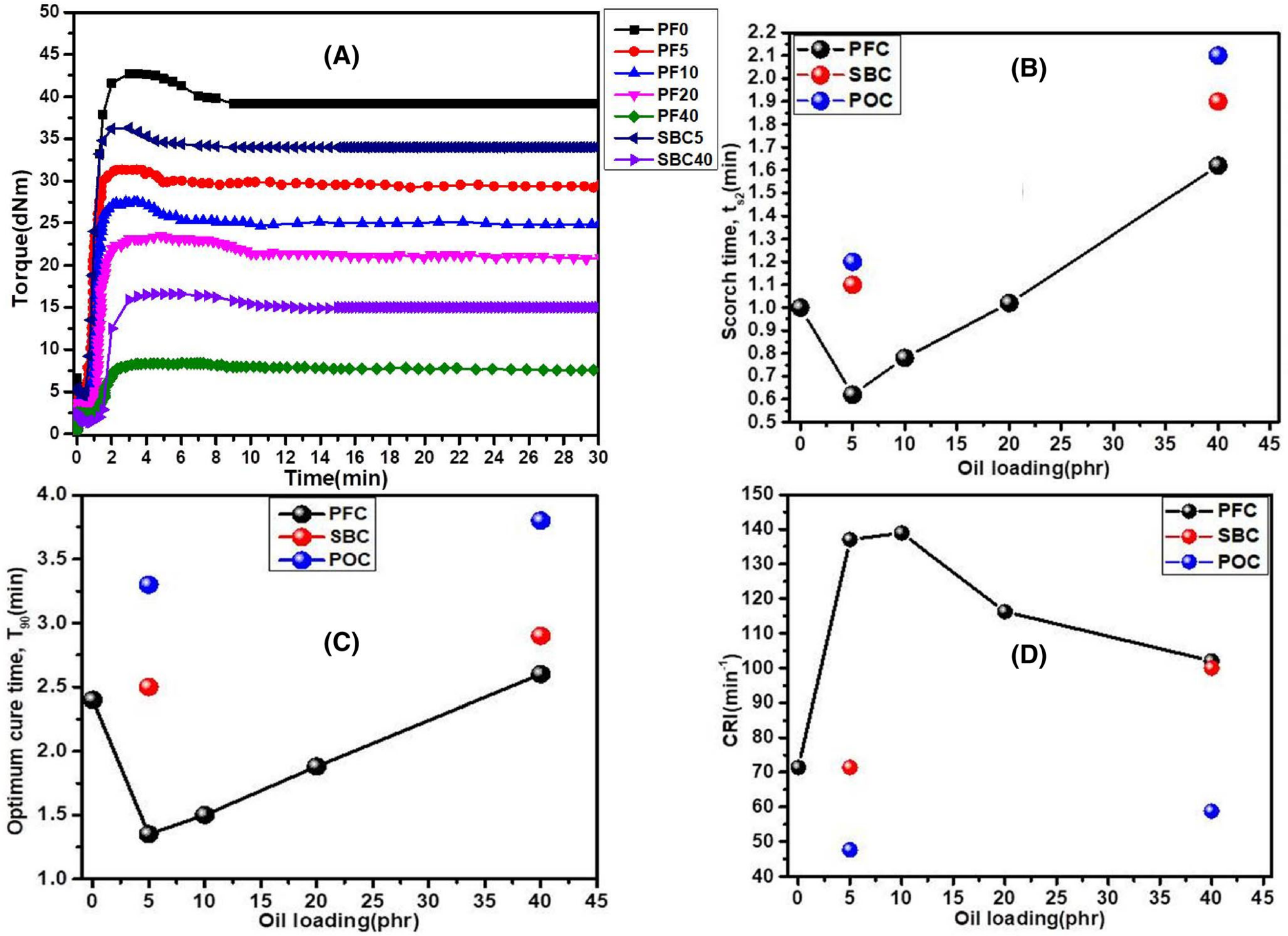

FIGURE 4 The cure time properties; (A) representative rheo-curves (B) t_{s2} (v) T_{90} and (D) cure rate index (CRI) of the oil plasticized NR—S—CB vulcanizates.

deduced from the respective rheo-curves are shown in Figure 4B–D, respectively.

As illustrated in Figure 4B,C, at elevated levels of the plasticizer oils (PFO, PO, and SBO), the curing times (t_{s2} and T_{90}) were generally seen increasing with a corresponding rise in the curing rate index (CRI) relative to the reference sample (PFO) as seen in Figure 4D. Chandrasekara and Ortega's group also reported a similar reduction in rubber compound curing when PO was present.[57,60] The oil phases within the rubber compounds may have hindered the interfacial diffusion and dispersion of curatives within the matrix, delaying their interaction with the unsaturated sites (-C=C-) of NR for rapid crosslinking reactions. Interestingly, the PFC was seen showing extremely lower T_{90} and higher CRI compared to the rest of the compounds. The retardation of the curing reaction was more pronounced for petroleum oil-based compounds (POC).[26] This suggests that the curatives were effectively dispersed in NR—S_x—CB composites assisted by PFO compared to POC and SBO.[26] Additionally, the presence of numerous fatty acids, like the stearic and oleic acid groups in PF, could have contributed to the faster curing in PFC compared to POC, since fatty acid (stearic acid) is an effective vulcanizing activator for rubber compounds.[61,62]

4.4.2 | Viscosity and torque properties

The minimum torque (M_L) or viscosity index, maximum torque (M_H), and torque difference (ΔM) are shown in Figure 5A,B, respectively, for the NR—S_x—CB compounds in the presence of the different plasticizers. The M_L dropped at higher plasticizer content. Yet, the pork fat (FC) compounds (PFC) generally recorded higher M_L values, significantly at lower PF loading (PF5) compared to the rest. A similar decreasing trend was recorded for the crosslinking density index (ΔM) and mechanical strength index (M_H). These observations exhibited by PFC are typical characteristics of plasticizer oils like PO and vegetable oils used in rubber compounding.[56,57,63]

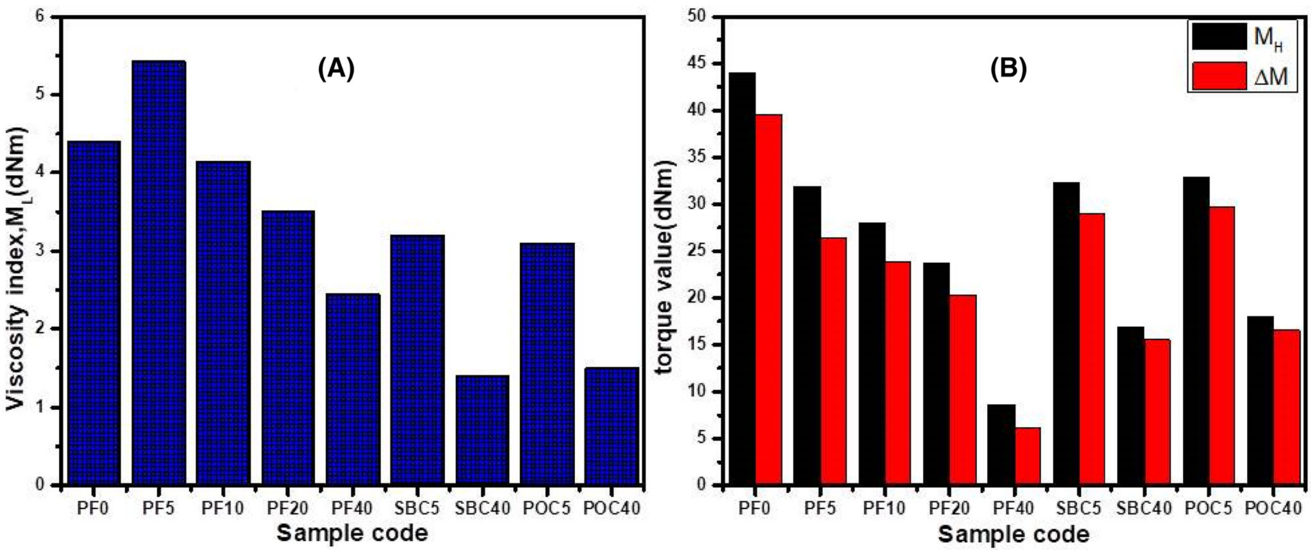

FIGURE 5 Comparing the minimum torque (M_L), maximum torque (M_H), and torque difference (ΔM) of PF in NR—S_x—CB compounds with, PO and SBO in NR—S_x—CB compounds.[26]

In terms of M_H and ΔM, the PFC slightly lagged behind the Shea-butter compounds (SBC) and the petroleum-oil compounds (POC), as reported earlier by Mensah et al.[26] interactions between natural rubber (NR) and carbon black (CB) compared to aromatic and shea-butter oil,[26] particularly at lower PF content level.

4.5 | Crosslinking density

The dispersion of fillers within a rubber matrix and the interactions between fillers and the matrix can be investigated using the equilibrium swelling ratio (Q_r) and cross-linking density (N_c) methods.[2] The values of Q_r and N_c for the various vulcanizates are compared in Figure 6A,B respectively. Q_r is seen increasing while N_c drops significantly by increasing the amount of oil. The result of N_c matches well with those obtained for the rheometric crosslinking density index (ΔM) (see Figure 5B). When compared, NR—S_x—CB compounds without oil exhibited lower equilibrium swelling ratios (Q_r) and correspondingly higher cross-linking densities (N_c) than those containing oil. The plasticizing effect of soybean oil on rubber has been previously investigated by Petrović et al.[32] The addition of soybean oil into the rubber-filled compound resulted in a significant decrease in cross-linking density (N_c), leading to a subsequent reduction in other properties such as modulus, strength, and compression set. The behavior exhibited by the PFC, shea-butter-based (SBC), and aromatic oil-based compounds (POC) was consistent with the typical effects of plasticizers.[26,56,57,60,63,64] When compared, the cross-linking density (N_c) of PF5 was significantly higher than POC5 by 36 and SBC5 by 130%. This indicates that in the presence of pork fat oil (PF), there may be stronger

4.6 | Tensile properties and reinforcement index

The tensile properties of the various vulcanizates are presented in Figure 7A, and the reinforcing index is shown in Figure 7B. In Figure 7A, although the tensile strength (TS) of the vulcanizates is scattered, lower loading of the plasticizer oils (5 phr) seems to favor the TS of petroleum oil compounds (POC), shea-butter oil compounds (SBC),[26] palm oil compounds[33] and that of soybean oil compounds reported by Jayewardhana et al.[65] Usually, higher plasticizer loading is associated with poor TS properties.[26,33,65] It is, however, interesting to see that even at 20phr of PF loading, the TS exceeded the rest of the compounds. For instance, the PF20 is over 8% and 13% higher than the TS of the palm oil compound (NR-50phr CB-20phr Palm oil)[33] and shea-butter compound (NR-50phr CB-20phr shea-butter oil)[26] respectively. The PF20 also showed improved and comparable TS values with those reported for soybean and palm oil compounds.[65] Further, the PF20 obtained higher TS values than compounds designed for tire treads by Mensah et al.[2] Here, for instance, sample TP5 (SBR/BR-35phr CB-10phr Silica-10phr TDAE oil) which obtained the highest TS of ~16 MPa, lags behind PF20 and PF40 by 41.8% and 12.5%, respectively. It is fascinating to also observe that the TS of PF compounds has demonstrated

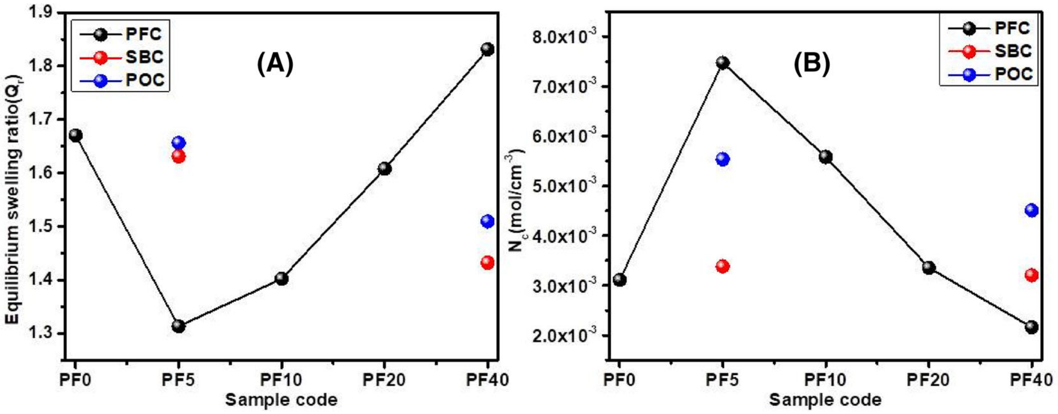

FIGURE 6 Effect of plasticizer oil loadings on the equilibrium swelling properties of NR—S_x—CB composites (A) equilibrium swelling degree (Q_r) (B) crosslinking density (N_c).

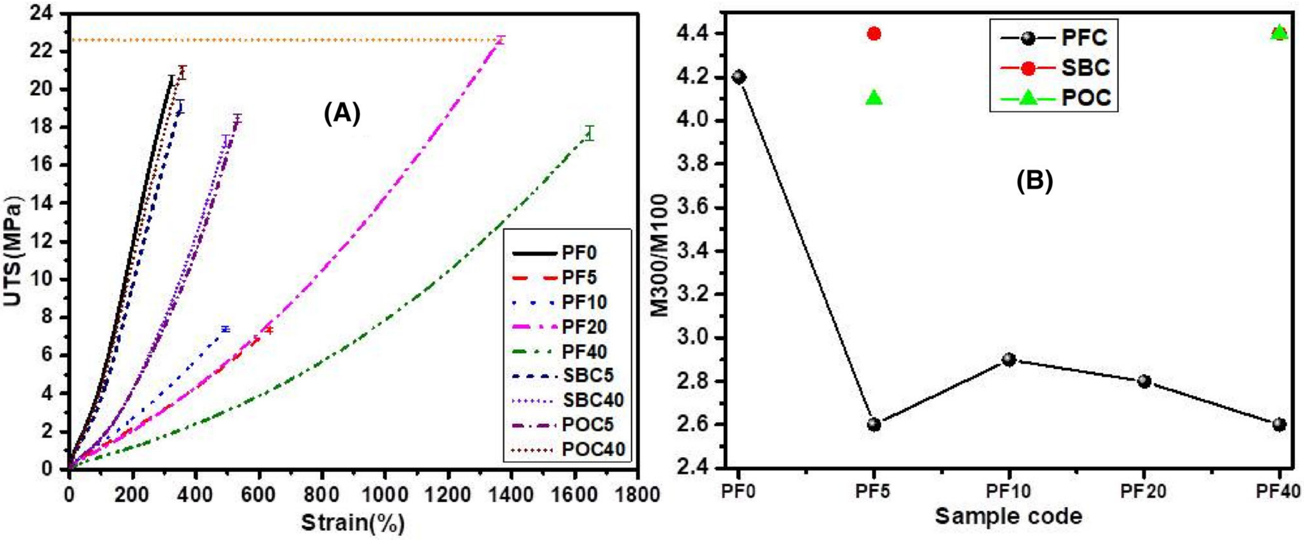

FIGURE 7 Tensile properties of NR—S_x—CB compounds plasticized with PF, PO, and SBO; (A) stress–strain curves (B) reinforcement factor.

comparable tensile strength (TS) properties with the truck-based tread compounds containing 75 phr NR/25 phr BR-50 phr CB-10 phr Silica, and 75phr NR/25phr BR-40phr CB-20phr Silica-10phr TDAE, as reported earlier by Ramin Zafarmehrabian et al.[29] In terms of elongation at break, EB (%), the PFC showed extremely higher values, especially at elevated PF content. For example, at lower 5phr of oil loading, PF5 was over 21% and 85% higher than SBC and POC,[26] respectively. At higher oil loading, the EB (%) of PF20 was 230% and 371% higher than SBC and POC,[26] and tremendously higher than the tread compounds reported earlier by Ramin Zafarmehrabian et al.[29] as well as those reported earlier by Mensah et al.[2]

The scattering in data observed for TS and EB (%) is a characteristic of rubber compounds filled with plasticizer oils, as earlier observed by Chandrasekara and Petrović et al.[56,57]

The reinforcing effect of carbon black (CB) on the gum in the presence of plasticizer oils was evaluated using the ratio of modulus at 100% and 300% (M300/M100), shown as reinforcement index. This approach is commonly employed to study the strengthening action of fillers on rubber chains, as shown in Figure 7B. Clearly, the SBC and POC are seen exhibiting higher reinforcement effects than the PFC, associated with higher stiffness. Noticeably, the PF increases the flexibility and mobility of the rubber chains as compared to PO and SBO.

4.7 | Fatigue life and hardness (Shore A)

Figure 8A compares the fatigue life of NR—S_x—CB compounds in the presence of pork fat oil (PFO), sheabutter oil (SBO), and aromatic oil (PO). As shown, increasing the oil content generally enhances fatigue behavior. For instance, the samples PF5, PF40, POC5, and POC40 demonstrated significant improvements of over 70%, 439%, 22%, and 434%, respectively, compared to the NR—S_x—CB compound without oil. The pork fat compounds (PFC) demonstrated relatively superior fatigue behavior compared to the POC and SBC, notably at higher oil loading. At 5 phr oil loading, PF5 achieved a 75% and 40% increase in fatigue life cycle compared to the compound without oil and POC5, while PF40 showed over a 21% increment in fatigue life cycle compared to SBC40 and POC40, respectively. The enhanced dispersion of carbon black (CB) and curatives within the PFC with desired tighter network structures may have hindered crack growth and delayed fatigue failure.

In general, a decreasing trend in hardness is seen as oil content increases. At 5 phr oil loading, POC and SBC compounds showed higher values compared to PFC (PF5). Also, the SBC and POC recorded relatively higher hardness values than the PFC, as shown in Figure 8B. Thus, the reason for the lower hardness could be associated with the higher fatigue life, higher EB (%), and lower reinforcement index for the PFC. Typically, in the presence of PF oil, the level of wetting of rubber chains with PF was higher due to viscosity differences, leading to the lowering of interfacial frictions between entangled rubber chains, as well as between CB particles that have adhered to themselves or to the rubber chains. This effect could cause a dramatic increase in mobility and flexibility, thereby extending the EB (%), lowering viscosity (see Figure 5A), reducing stiffening tendency, and increasing fatigue life.[14,15,33]

4.8 | Rebound resilience and flex-fatigue

The rebound resilience (R_b%) behavior of the various compounds is presented in Figure 9. It can be seen that increasing the content of pork fat oil (PFO) generally enhances resilience properties. As shown in Figure 9, the pork fat compounds (PFC) exhibit a significant improvement in resilience compared to the reference sample (SBC) and the petroleum oil-based compounds (POC). For example, the R_b% of PF5 and PF40 was approximately 7% and 5% higher than POC5 and POC40, respectively, while lagging behind SBC. When compared with the previously reported industrial tread compounds: NR/SBR-57 phr CB, NR/SBR-57 phr CB, and NR/SBR-55 phr CB, which recorded R_b% values of 38.9%, 38.4%, and 34.5%, respectively,[66] the PFC, like SBC and POC,[26] has demonstrated higher resilience properties. The current sample, PFC, has also demonstrated improved R_b% behavior compared to the ternary blends: (SBR/BR/NR-70 phr CB) and

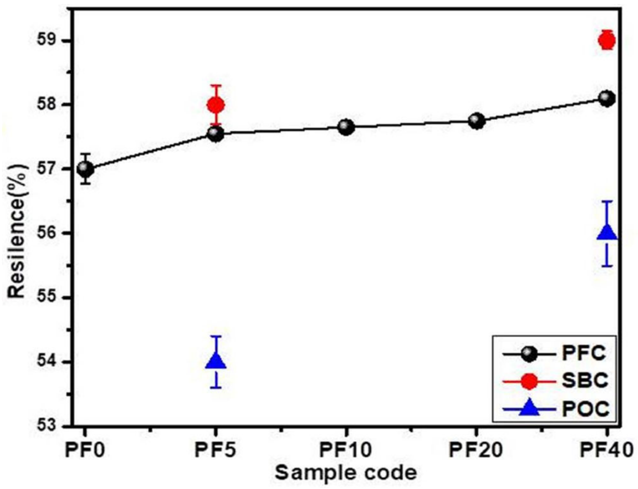

FIGURE 9 Effect of plasticizer oil loadings on the Resilience (%) properties of NR—S_x—CB compounds.

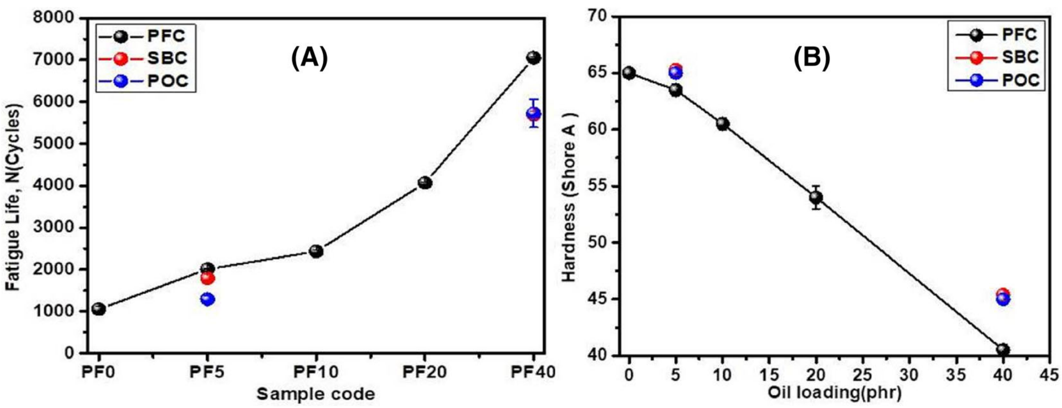

FIGURE 8 Comparing the (A) fatigue life and (B) hardness (Shore A) properties of PFC with representative samples of SBC and POC.[26]

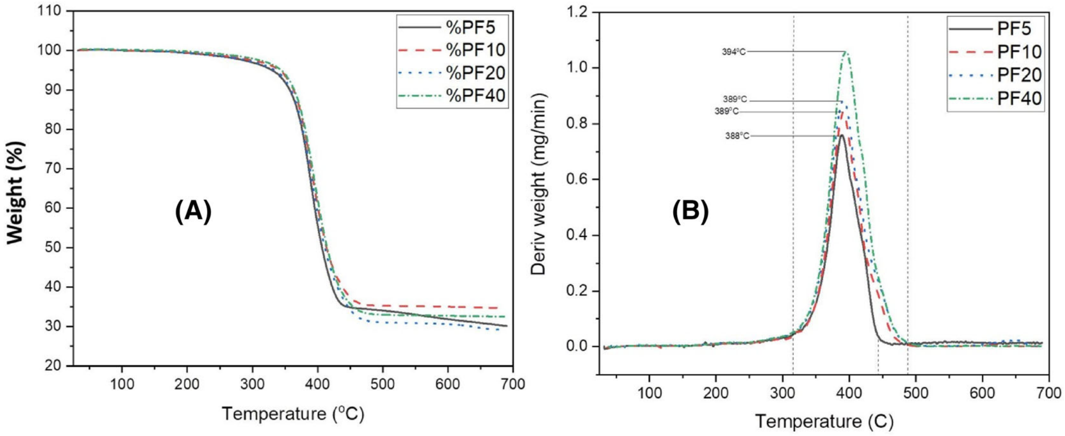

FIGURE 10 Thermal degradation behavior of NR—S_x—CB containing pork lard as plasticizer. (A) TGA thermograph; and (B) DTG thermograph.

(SBR/BR/NR-50 phr CB/20 phr silica) which recorded R_b% of 15% and19%, respectively.[30] Therefore, the PFO has been demonstrated to be a suitable plasticizer oil for compounding rubber intended for developing tread compounds with higher rebound resilience behavior.

4.9 | Thermo-gravimetric analysis (TGA)

The TGA of pure natural rubber (NR) was reported earlier by Yahya et al.[67] and Sasitaran et al.[68] which recorded a maximum degradation temperature of T_{max} of 380 and 382.9°C, respectively. Peterson et al.[69] also studied the TGA of NR-CB composites, where the NR-CB(40phr) recorded ~4% weight residue (%). In this present work, Figure 10A,B show the respective plots of the TGA and the derivative thermogravimetry (DTG) thermographs of representative vulcanizates of natural rubber-carbon black (NR—S_x—CB) compounded with pork fat oil. In Figure 10A, the decomposition stages seen around the temperature range of 100–250°C can be linked to the breakdown of low molecular weight structures, including water moisture and remains of unreacted cure ingredients, while the decomposition in the 350–520°C range could be an indication of the thermal degradation of the main chains of NR. In terms of weight residue (%), there is no clear trend seen; the order of decreasing weight residue (%) is observed as: PF10 > PF40 > PF5 < PF20, which is a typical behavior of plasticizers incorporated into rubber compounds.[26,33] Even so, the minimum weight residue (%) reported presently in this work is slightly lower than 30%, which is extremely higher than the one reported for NR-CB and NR-CB containing calcium carbonate and biochar.[69]

However, the order of increasing T_{max} (°C) is given by PF5 < PF10 = PF20 < PF40 as shown in Figure 10B. Clearly, increasing the pork fat/lard seems to increase the T_{max} (°C) and sample PF40 demonstrated the highest of these properties. The huge content of the pork fat incorporated could have wetted, coated, and insulated the rubber molecules, thereby slowing down the thermal degradation of the pork oil-filled compounds. The results reported pork fat-filled compounds are higher than unfilled NR cured conventionally at 160°C.[67,68] Therefore, for increased thermal stability, optimization of the pork fat content in the rubber compound can be done to maintain the overall physico-mechanical behavior.

5 | CONCLUSION

Natural rubber (NR) filled with carbon black (CB) in the presence of edible and eco-friendly pork fat oil (PFO) was systematically investigated and compared to compositions prepared with petroleum-based oil (TDAE or PO). Properties like crosslinking density, via equilibrium swelling, viscosity index (M_L), torque properties (M_H & ΔM), tensile strength, elongation at break, hardness (Shore A) and fatigue, etc. were all studied. It was interesting to find out that while these properties were advantageous for the pork fat compounds (PFC), others were comparable to the petroleum oil-based compounds (POC) and Shea-butter-based compounds (SBC). For example, the PFO shortened optimum curing time, and caused a dramatic increase in elongation at break (%), with high fatigue resistance and rebound resilience properties. For example, the optimum cure time (T_{90}) of PF5 and PF40 was reduced to about 225% and 276% when compared to POC5 and POC40 and 150% and 183% relative to SBC5 and SBC40, respectively. In terms of bound rubber content (BRC %), a measure of filler–matrix interactions, PF5 was over 15% and 5% higher than POC5 and SBC5, respectively. Also, the PF5 led POC5 by 12% in crosslinking density and by 7% in rebound resilience (%). Furthermore, PF5 and PF40 showed improvements in fatigue life behavior by exceeding POC5 and POC40 by 40% and 75%, respectively. These findings confirm that PFO is a promising, economical, and environmentally friendly plasticizer that can serve as a viable alternative to vegetable and petroleum-based oils in the rubber processing industry.

AUTHOR CONTRIBUTIONS

Bismark Mensah (A), Winston Amobeng (B), Vitus Apalangya (C), Emmanuel Essien (D), Elijah Nyakudya (E), Emmanuel Essien (F), Kan-Dapah Kwabena (G), and Tsatsu Nukunya (H): A: Project administration, resources, methodology, investigation, formal analysis, data curation, conceptualization, and writing—original draft. B: Methodology, investigation, data analysis, and writing—review/editing. C–G: Writing—review/editing. H: Resources, support, writing—review/editing, and supervision.

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CONFLICT OF INTEREST STATEMENT

The authors declare no competing financial interest. Corresponding author has consent from all co-authors.

DATA AVAILABILITY STATEMENT

The data used to support the findings of this study are available from the corresponding author upon request.

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