Effect of Activated Palm Kernel Shell Nanoparticles as a Bio-filler on the Mechanical Properties Vulcanized Natural Rubber

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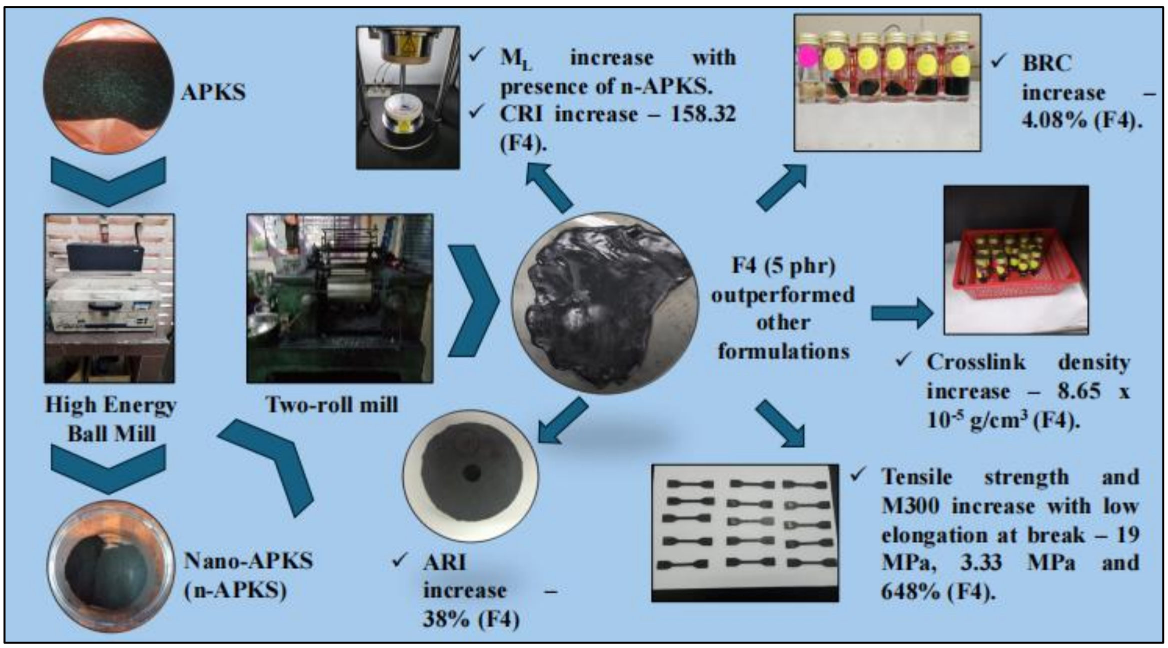
GRAPHICAL ABSTRACT



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Oil palm wastes, such as palm kernel shell (PKS), can be beneficially used as value-added additive for natural rubber. Converting PKS into activated palm kernel shell (APKS) and transforming into nano size APKS (n-APKS) can enhance the performance of vulcanizates natural rubber (NR). Performance of n-APKS incorporated NR has been evaluated with different loadings (0 to 10 phr) in several tests, such as cure characteristic, bound rubber content (BRC), swelling, tensile, and abrasion resistance index (ARI). The presence of n-APKS decreased minimum torque from 0.54 dN.m to 0.31 dN.m, indicating enhancement processability. The cure index peaked at n-APKS loading of 5 phr (F4), which indicated the effective crosslinking in NR matrix. The F4 (5 phr) formulation outperformed others, featuring a BRC of 4.08%, a crosslink density of 8.65 x 10^{-5} mol/cm^3, a tensile strength of 19 MPa, a M_{300} at 3.33 MPa, a reinforcement index of 3.78%, and an ARI of 38% with relatively low elongation at break (648%). The optimum loading of 5 phr is deemed a promising bio-filler option for the rubber industry and it also helps to reduce environmental waste disposal issues.

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Keywords: Natural rubber; Nano-activated palm kernel shell; Bio-filler; Performance; Rubber industry

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INTRODUCTION

Palm oil production is a key contributor to Malaysia's economy, with oil palm plantations covering approximately 5.65 million hectares. In 2023, Malaysia produced 18.55 million tonnes of crude palm oil and 0.98 million tonnes of palm kernel oil (Parveez *et al.* 2024), showing a slight increase from 2022 as the oil palm industry recovered post-pandemic (Parveez *et al.* 2023). However, large-scale production generates substantial waste biomass, including oil palm trunks (OPT), oil palm fronds (OPF), empty fruit bunches (EFB), mesocarp fibers (MF), palm kernel shells (PKS), and palm oil mill effluent (POME) (Onoja *et al.* 2019). Improper management of this biomass can lead to environmental issues, such as pollution and global warming (Abbas *et al.* 2019).

To address these challenges, research has focused on converting this renewable biomass into value-added products such as bio-fillers (Khalil *et al.* 2012; Yeo *et al.* 2020; Jafri *et al.* 2021). Natural fillers, such as palm oil, coconut husk, rice husk, and other renewable materials, are gaining attention due to their environmental friendliness, biodegradability, cost-effectiveness, availability, and low density (Zhou *et al.* 2015). In rubber applications, fillers are essential for enhancing properties such as strength, ductility, hardness, impact resistance, and fracture toughness (Zafirah *et al.* 2020; Siti *et al.* 2024). The performance of fillers is influenced by factors such as porosity, carbon content, surface area, and loading (Romli and Mamauod 2017; Zafirah *et al.* 2023). Among natural fillers, palm kernel shell (PKS) has shown promise as a bio-filler for rubber (Aisyah Ar-Raudhoh *et al.* 2024; Daud *et al.* 2016; Syazwani Aqilah *et al.* 2024). However, PKS faces challenges such as low surface area, low carbon content, and a lack of pore development, limiting its effectiveness. To overcome these limitations, PKS can be subjected to a carbonization-activation process, resulting in activated palm kernel shell (APKS). Previous studies have shown that micro-sized APK particles can serve as semi-reinforced fillers, enhancing the properties of carboxylated nitrile butadiene rubber (XNBR) (Syazwani Aqilah *et al.* 2024).

This study focuses on nano-sized activated palm kernel shell (n-APKS) as a bio-filler for natural rubber (NR), representing a significant advancement over previous research using micro-sized fillers. The smaller particle size of n-APKS increases the surface area, improving rubber-filler interaction and potentially enhancing mechanical properties. Additionally, n-APKS, like activated carbon, possesses unique properties such as a higher surface area and greater porosity, which are expected to improve its interaction with the rubber matrix. These characteristics are crucial for enhancing the overall performance of the rubber composite. This approach, which has not been extensively explored, is expected to outperform micro-sized fillers (Marlina *et al.* 2020).

The research aimed to evaluate the performance of n-APKS as a bio-filler for NR compounds with varying filler loadings (1.0 phr to 10 phr). The study assessed the properties of n-APKS-filled NR vulcanizates through various tests, including cure characteristics, bound rubber content, swelling measurements, tensile behavior, and abrasion resistance. This study presented an innovative approach to improving natural rubber performance by utilizing nano-sized sustainable fillers, offering valuable insights into the potential of bio-based materials to enhance rubber properties.

EXPERIMENTAL

Materials

The NR grade SMR-10 was sourced from Vistec Industries Sdn. Bhd., Puchong, Malaysia, and the Activated Palm Kernel Shell (APKS) depicted in Fig. 1a, was supplied by the Malaysian Palm Oil Board (MPOB), Bangi, Malaysia. Compounding ingredients, including zinc oxide (ZnO), stearic acid (HST), Benzothiazyl Disulfide (MBTS), 1,3-Diphenyl-Guanidine (DPG), Tetramethylthiuram Disulfide (TMTD), 2,2,4-Trimethyl-1-2-Dihydroquinoline (TMQ), a dispersion agent, sulfur, and processing oil, were obtained from Airelastic Industries Sdn. Bhd., Klang, Malaysia. The APKS was transformed from micro to nano-size using a High Energy Ball Mill (HEBM), as depicted in Fig. 1b.

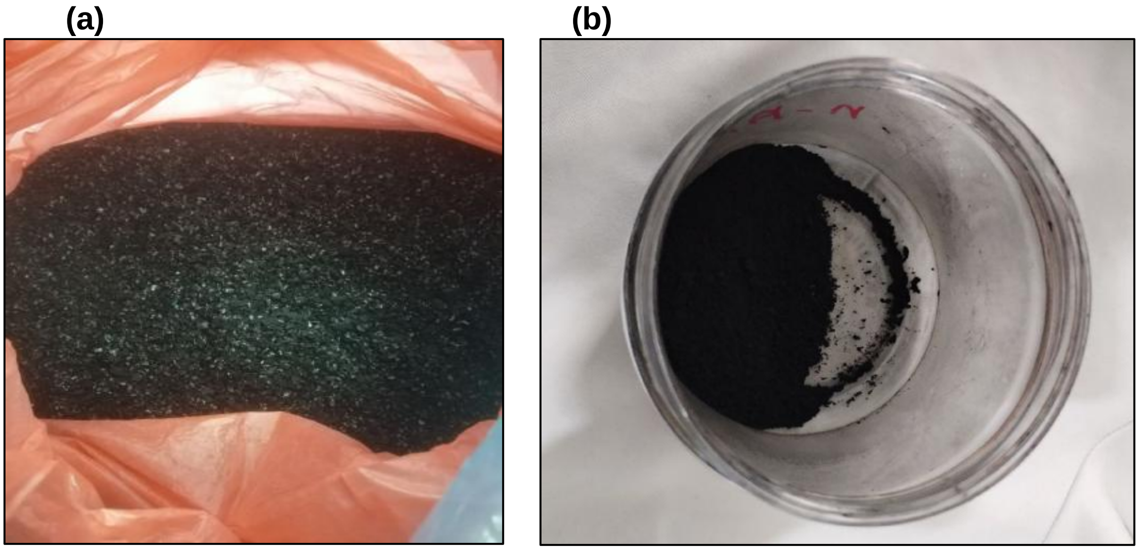

Fig. 1. (a) APKS; (b) nano-APKS

Preparation of Nano-Activated Palm Kernel Shell Filled Natural Rubber Compounds

Six different formulations of n-APKS filled NR compounds were prepared with varying filler loadings, as shown in Table 1. The compounding process was conducted using a two-roll mill, following ASTM D3184-11 (2018) standards. The rubber was pre-masticated for 1 to 2 min until it softened and formed a band, making it easier to mix during the compounding process. All ingredients were thoroughly blended to ensure uniformity. After mixing, the compound was allowed to rest overnight at room temperature. The rubber was then molded at its respective cure time (t_{90}) at approximately 160 °C using a hot press machine, a temperature that ensures optimal vulcanization and good flow for NR compounds to fill the mold. The resulting rubber compound was used for mechanical testing, including bound rubber content, swelling, tensile strength, and abrasion resistance. The flowchart for preparing n-APKS with the NR compound is shown in Fig. 2.

Table 1. Formulations of n-APKS filled NR Compounds

Ingredient (phr)*	Formulation No.					
	1	2	3	4	5	6
NR	100	100	100	100	100	100
n-APKS	0	1	3	5	7	10
ZnO	3	3	3	3	3	3
HST	2	2	2	2	2	2
Dispersing Agent	2	2	2	2	2	2
TMQ	0.8	0.8	0.8	0.8	0.8	0.8
Processing Oil	5	5	5	5	5	5
DPG	0.7	0.7	0.7	0.7	0.7	0.7
Sulphur	1.3	1.3	1.3	1.3	1.3	1.3
Accelerator	2.05	2.05	2.05	2.05	2.05	2.05
* (Parts per hundred rubber)						

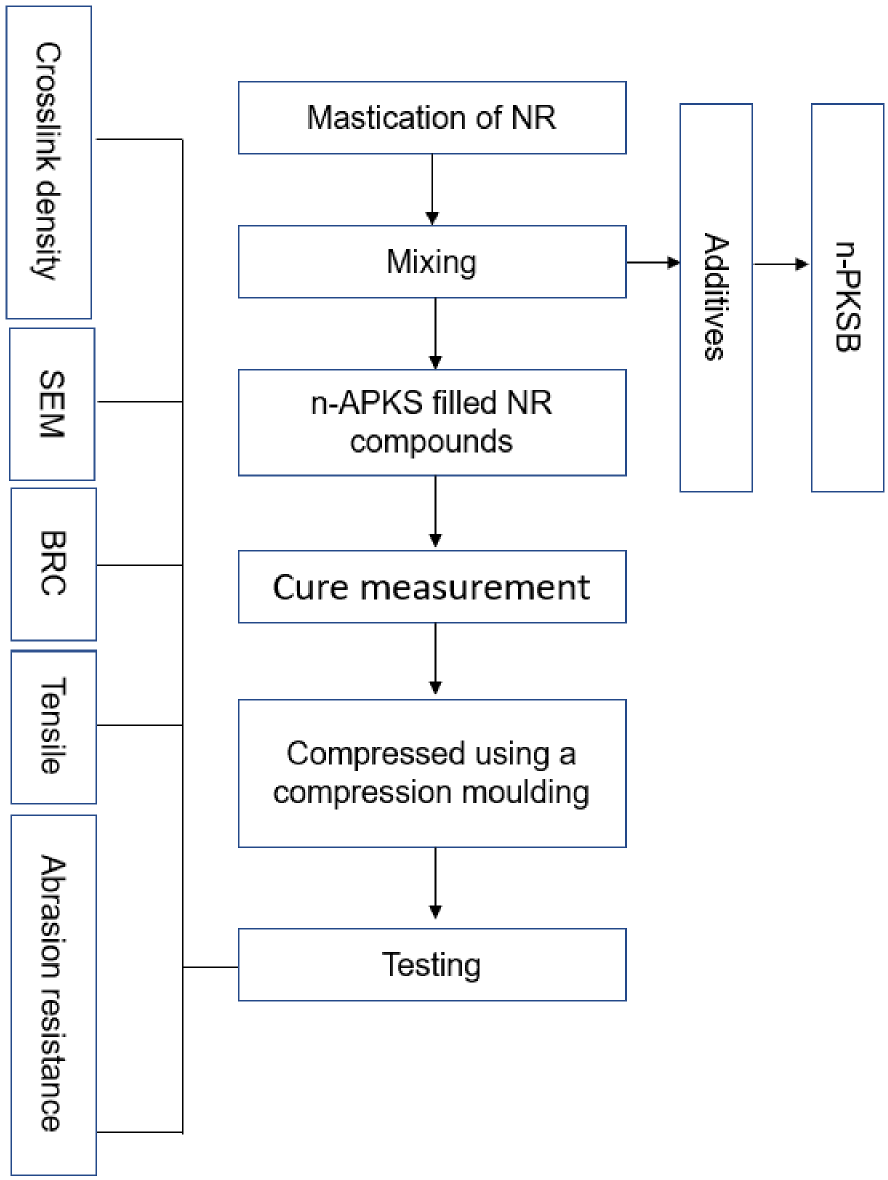

Fig. 2. Flowchart for the preparation of n-APKS incorporated into the NR compound

Characterization of Nano-Activated Palm Kernel Shell Filled Natural Rubber Compounds

Cure characteristics

The cure characteristics were determined to assess the scorch time (t_{s2}), optimum time (t_{90}), minimum torque (M_L), maximum torque (M_H), delta torque (ΔM), and cure rate index (CRI) of each formulation of n-APKS-filled NR compounds. This analysis was performed using the Moving Die Rheometer (M-3000AU) (Gotech Testing Machine Inc., Taichung City, Taiwan) according to ASTM D2048-19a (2019). Approximately 5 g of each formulation of n-APKS filled NR compounds was prepared and cut using a rheometer cutter. The sample was then placed between the pair of rotating disks, with the temperature set at 160 °C.

Bound rubber content (BRC)

The BRC analysis was conducted according to ASTM D5775-95 (2019). For this analysis, approximately 0.3 g of each formulation of the uncured n-APKS-filled NR samples were prepared and cut. Each sample was immersed in 10 mL of toluene solvent inside a sealed glass bottle and kept in a dark environment for 7 days. After the 7-day immersion period, the sample was weighed then dried for 1 h at 70 °C in a fume chamber to allow the solvent to evaporate. The sample was then dried again in a drying cabinet for 24 h and weighed once more. The BRC was calculated using Eq. 1,

$$\text{BRC} = \frac{W_{fg} - W[m_f/(m_f+m_p)]}{W[m_p/(m_f+m_p)]} \times 100\% \quad (1)$$

where W_{fg} is the weight of the gel (g), W is the weight of the test piece (g), m_f is the weight of the filler (g), and m_p is the weight (g) of the polymer in the rubber compound.

Swelling measurement

The swelling measurement was performed according to ASTM D3616-95 (2019). Each formulation of n-APKS-filled NR vulcanizates was cut into dimensions of 1 cm × 1 cm, and the initial weight was recorded. Each sample was immersed in 10 mL of toluene in a small glass bottle with a tightly fitting cap for 5 days. The weight of the samples was recorded daily. After 5 days of immersion, the swollen sample was weighed and dried in an oven at 70 °C until the weight became constant. The data obtained were then tabulated, and the crosslink density ($[X]_{phy}$) was calculated using the Flory-Rehner equation, as shown in Eq. 2,

$$-\ln(1 - V_r) - V_r - XV_r^{\,2} = 2\rho V_o [X]_{phy} V_r^{\,1/3} \quad (2)$$

where V_r is the volume fraction of the polymer in solvent specimen, X is the interaction parameter of the rubber network-solvent (X of NR = 0.391), ρ is the density of rubber (NR = 0.92 g/cm^3), V_0 is the molar volume (103.11 cm^3/mol), and $[X]_{phy}$ is the crosslink density (mol/cm^3).

Tensile test

The tensile test was used to determine the tensile properties of n-APKS-filled NR vulcanizates. This testing was carried out using the Instron Universal tensile testing machine (model 5569; Instron, Norwood, MA, USA). The test was performed at a speed of 500 mm/min according to ASTM Standard D412-16 (2021). Each formulation of n-APKS-filled NR vulcanizates was cut into dumbbell shapes using a tensile rubber cutter and the thickness was measured. At least 5 rubber samples from each formulation were tested and average results were recorded. The tensile properties, including tensile strength, elongation at break, and modulus at 300 (M_{300}) values, were recorded by the software. The reinforcement index (RI) for n-APKS-filled NR vulcanizates was calculated using Eq. 3,

$$\text{RI} = \frac{M_{300}}{M_{100}} \quad (3)$$

Abrasion test

The abrasion test was conducted to determine the wear resistance of the rubber surface by applying abrasive force. This testing was performed using Abrasion Resistance Tester (GT-7012-D; Gotech Testing Machine Inc., Taichung City, Taiwan) according to ASTM D5963-22 (2022). Five samples from each formulation of n-APKS-filled NR vulcanizates were prepared and drilled into small round samples using a REXON professional bench drill press. The weight and density of each sample were recorded. After the test run was completed, the weight of the abrasive sample was recorded again to determine the mass loss. The abrasion resistance index (ARI) was calculated using Eq. 4,

$$\text{ARI } (\%) = \frac{V_s}{V_t} \text{ X } 100\% \tag{4}$$

where V_s is the volume loss of the standard rubber (cm^3) and V_t is the volume loss of the test rubber (cm^3).

RESULTS AND DISCUSSION

Cure Characteristics

The cure characteristics of NR compounds (F1) and n-APKS-filled NR compounds (F2 through F6) are presented in Table 2, with F1 serving as the control. The M_L and M_H values assess the processability and viscosity of the rubber compounds, respectively, while *M* indicates the material's stiffness and rigidity due to crosslinking (Sianturi and Surya 2018; Abdelsalam *et al.* 2021). It is evident that the M_L values showed minimal change, indicating that processability is not significantly affected. Similarly, the M_H, *M*, t_{s2}, and t_{90} values displayed only slight variations, regardless of the presence of n-APKS.

Table 2. Cure Characteristics of n-APKS-Filled NR Compounds

Formulations	t_{s2} (min)	t_{90} (min)	M_L (dN.m)	M_H (dN.m)	ΔM (dN.m)
F1	0.47	1.34	0.54	12.97	12.43
F2	1.01	1.71	0.31	13.04	12.73
F3	0.55	1.59	0.35	13.55	13.21
F4	0.51	1.44	0.34	13.89	13.56
F5	0.59	1.66	0.43	13.66	13.24
F6	1.11	1.99	0.50	13.63	13.13

Bound Rubber Content (BRC)

The percentage values of BRC for uncured n-APKS-filled NR compounds are shown in Fig. 3. F1, which contained no n-APKS, had a BRC value of 0%. As n-APKS was added, the BRC began to rise, increasing by 4% from F2 to F3 and by 8% from F2 to F4. F4 exhibited the highest BRC value (92%) compared to the other formulations, indicating strong interactions between n-APKS and the NR phase. This was likely due to the irregular shape, rough surface, and microporous structure of n-APKS (Fig. 4), which enhances mechanical interlocking between the two phases, thereby strengthening the interaction between n-APKS and the NR matrix (Kaewsakul *et al.* 2013; Hamza *et al.* 2015; Syazwani Aqilah *et al.* 2024). F5 shows a decreasing trend, likely due to poor interaction between n-APKS and NR at higher loading levels. F6 (10 phr) does not have data, as the uncured rubber compound dissolved completely after one day, resulting in no BRC value. This dissolution is attributed to weak interactions between n-APKS and the NR matrix, leading to a loosely bound rubber shell (Sarkawi *et al*. 2015).

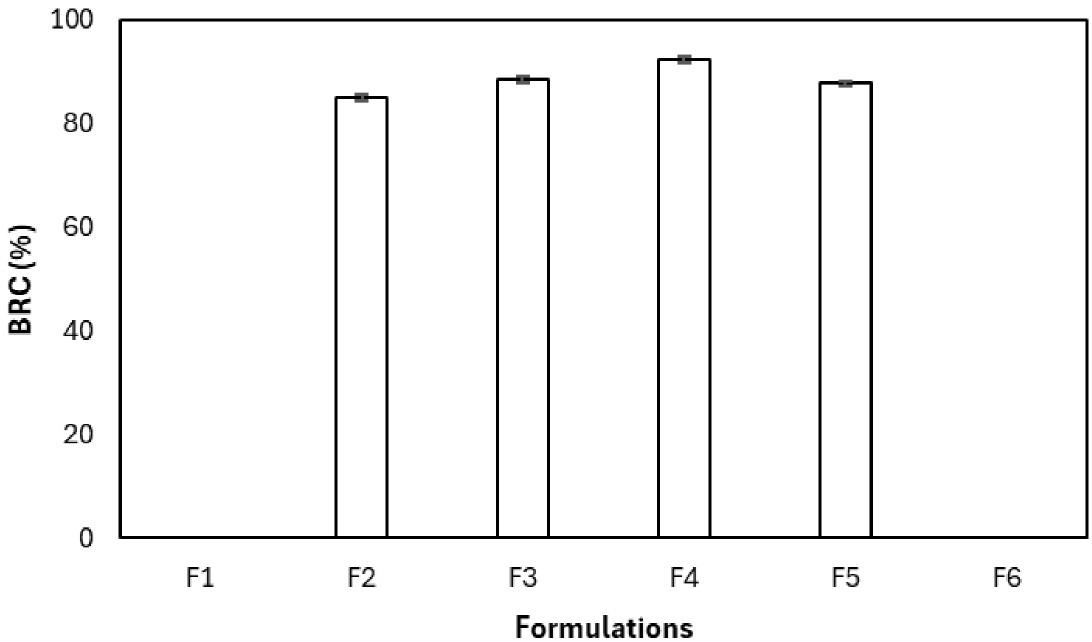

Fig. 3. Bound rubber contents of uncured n-APKS-filled NR compounds

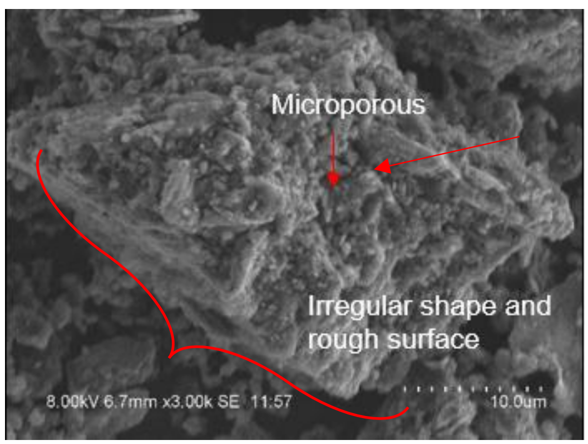

Fig. 4. SEM image of n-APKS particle

Crosslink Density

The crosslink density of n-APKS-filled NR vulcanizates is presented in Fig. 5. F1, which contained no n-APKS. It had the lowest crosslink density at 6.82 × 10^{-5} mol/cm^3. The absence of fillers allowed the rubber chains to be less constrained, enabling the toluene solvent to penetrate more easily (Ostad Movahed *et al.* 2015). The introduction of n-APKS resulted in a 1% increase in crosslink density from F1 to F2. As n-APKS loading was increased, the crosslink density continued to rise by 8% from F2 to F3 and by 16% from F2 to F4, with F4 exhibiting the highest crosslink density (8.65 × 10^{-5} mol/cm^3). This increase is attributed to the stronger interaction between n-APKS and NR phases, which restricts the mobility of rubber chains within the 3D network of the vulcanizate (Kim *et al.* 2020; Majid *et al.* 2020). This finding aligns with the higher BRC value, which is associated with the elevated crosslinking density of F4. However, as n-APKS loading continued to increase, the crosslink density began to decrease, dropping by 8% from F4 to

F5 and by 10% from F4 to F6. This decline is likely due to agglomeration from the high n-APKS content (Fig. 6), which leads to poor dispersion and weak interaction between n-APKS and the NR phase (Kam *et al.* 2018).

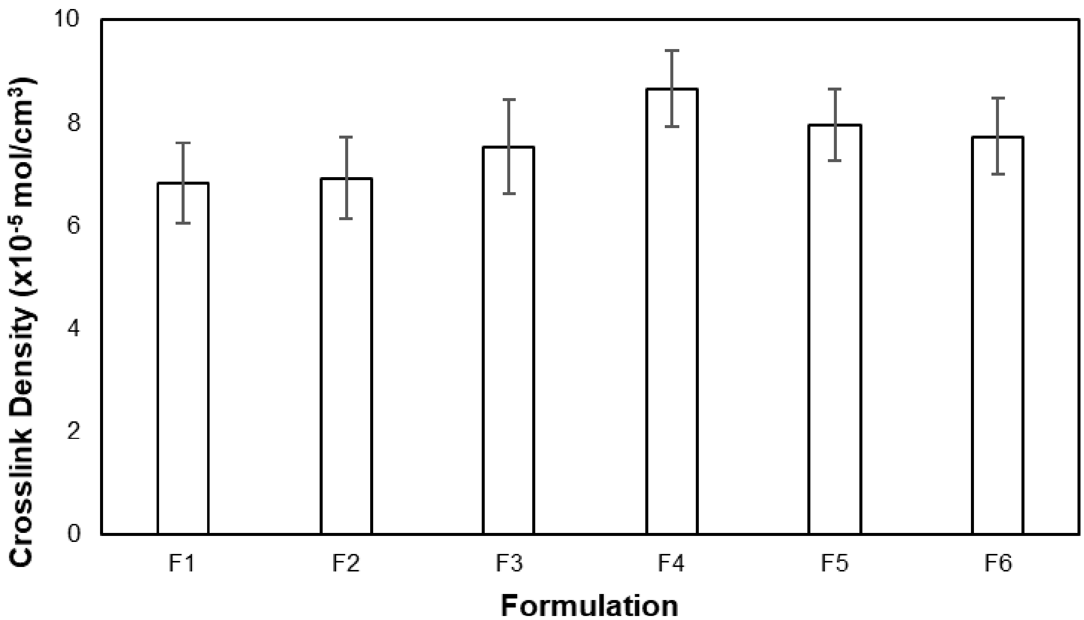

Fig. 5. Crosslink densities of n-APKS-filled NR vulcanizates

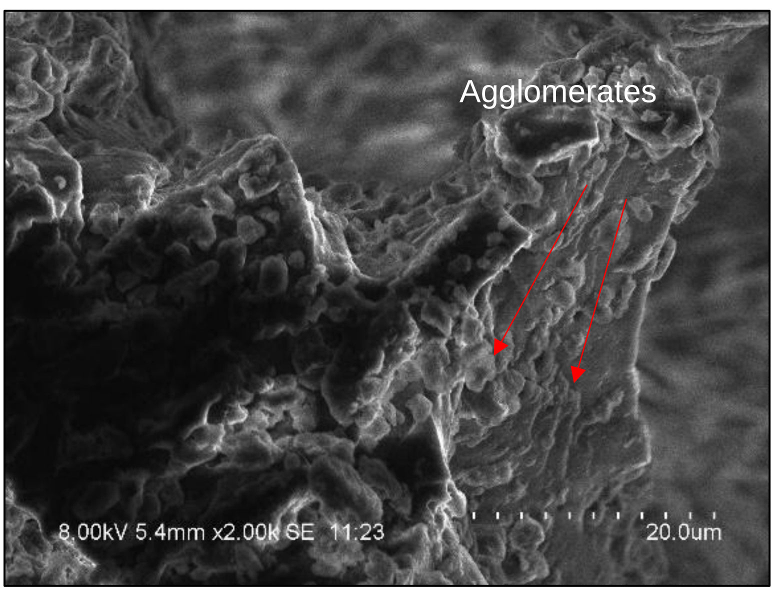

Fig. 6. Agglomeration at above 5 phr of n-APKS filled NR vulcanizate

Tensile Properties

The tensile strength of n-APKS-filled NR vulcanizates is shown in Fig. 7. F1, which lacked n-APKS, exhibited the lowest tensile strength at 8 MPa. The addition of n-APKS resulted in a significant increase in tensile strength, with a 94% rise from F1 to F2. As n-APKS loading increased, tensile strength continued to improve, with a 7% increase from F2 to F3 and a 17% increase from F2 to F4. F4 displayed the highest tensile strength at 19

MPa, which can be attributed to the stronger interaction between n-APKS and the NR phases, aligning with the BRC and crosslink density results.

The SEM image of the tensile fracture in Fig. 8a reveals a clean-cut fracture with no discontinuities, suggesting that n-APKS particles were well-bonded within the NR phase (Lay *et al.* 2020). This indicates that at 5 phr, n-APKS showed better dispersion, with its microporosity promoting improved mechanical interlocking with the NR phase (Fig. 4). However, as n-APKS loading increased, the tensile strength began to decrease, with a 13% drop from F4 to F5 and a 26% drop from F4 to F6. This decline is mainly due to excessive n-APKS loading, which leads to agglomeration, disrupting the continuity of the n-APKS-filled NR network. The fracture surface in Fig. 8b shows discontinuities, indicating weak interfacial bonding between n-APKS and the NR matrix, resulting in poor resistance to pulling forces (Kam *et al.* 2018; Abdelsalam *et al.* 2021).

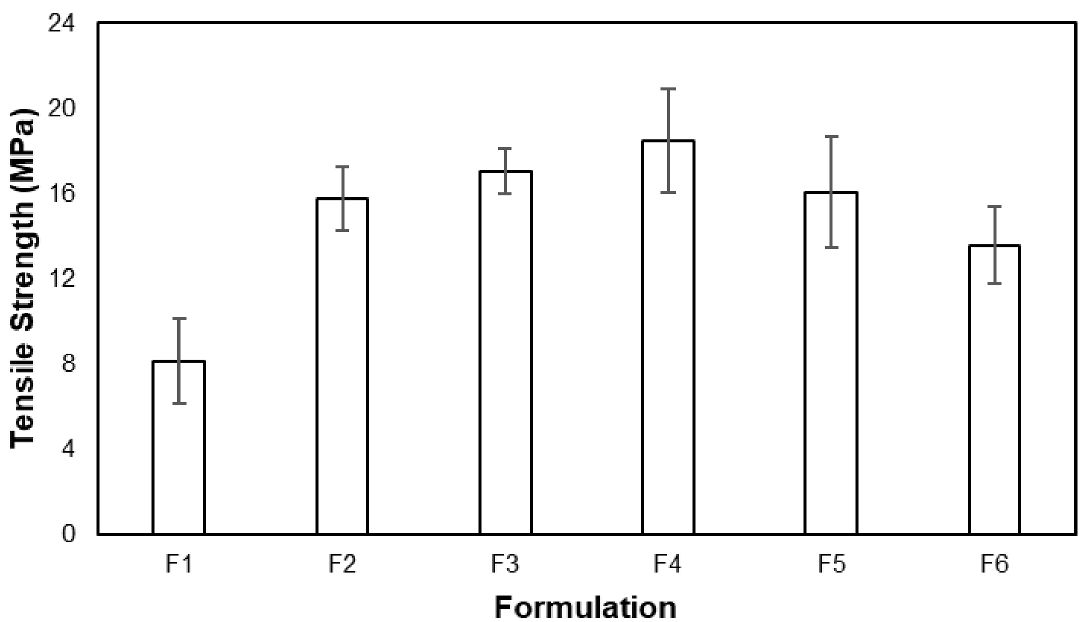

Fig. 7. Tensile strengths of n-APKS-filled NR vulcanizates

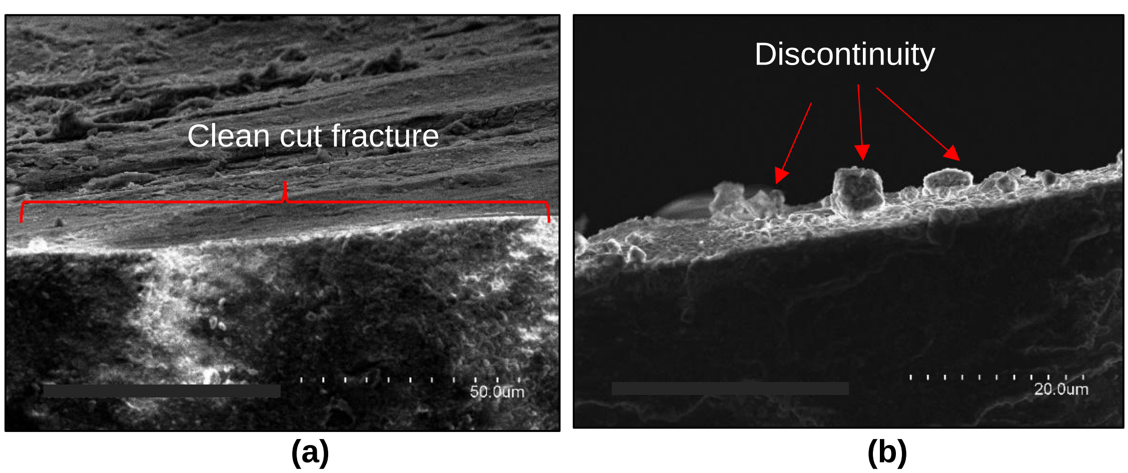

Fig. 8. SEM image of tensile fracture n-APKS-filled NR vulcanizates: (a) F4 (5 phr), (b) above 5 phr

The elongation at break (EB) of n-APKS-filled NR vulcanizates is shown in Fig. 9. A decreasing trend in EB is observed with the addition of n-APKS, dropping by 3% from F1 to F2. This can be attributed to the presence of n-APKS, which creates tie-chain molecules and entanglements within the NR matrix, thus increasing the ductility of the rubber vulcanizates as n-APKS forms mechanical interlocks with the NR matrix, as reflected in the BRC results (Nabil *et al.* 2013; Bukit *et al.* 2020). As n-APKS content increased further, the EB decreased by 0.7% from F2 to F3 and by 4% from F2 to F4, with F4 showing the lowest EB value (648%) compared to other formulations. This can be attributed to the high stiffness and rigidity of F4, which results from a higher crosslink density and BRC values. However, as n-APKS loading increased further, the EB started to rise, increasing by 12% from F4 to F5 and by 14% from F4 to F6. This increase is due to agglomeration (Fig. 6), which leads to the formation of larger clusters of particles that act as weak points within the NR phase, ultimately reducing the EB (Usha Devi *et al.* 2015).

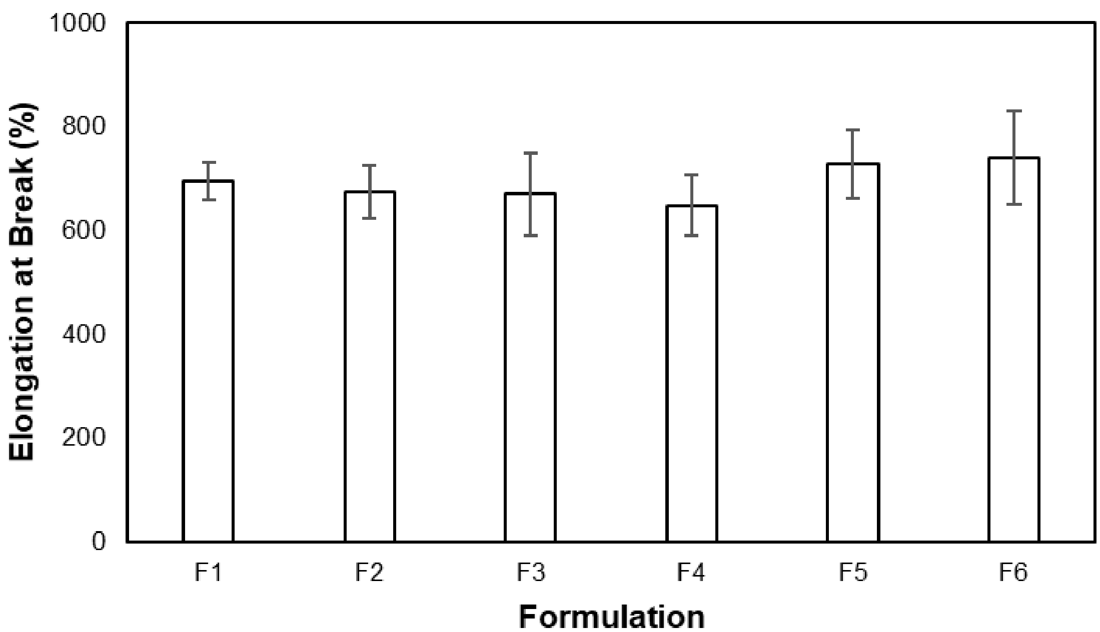

Fig. 9. Elongation at break of n-APKS-filled NR vulcanizates

The M_{300} and reinforcement index (RI) of n-APKS-filled NR vulcanizates are shown in Fig. 10. A similar increasing trend was observed in M_{300} and RI with the incorporation of n-APKS in NR. There was a drastic increase in M_{300} and RI with the presence of n-APKS, increasing from F1 to F2 by 66% and 111%, respectively. This rapid improvement indicates that the NR vulcanizates had become stiffened and more rigid. As n-APKS loading continued, both M_{300} and RI increased from F2 to F3 by 0.9% and 7% and from F2 to F4 by 5% and 19%, respectively. F4 had the highest values of both M_{300} (3.33 MPa), and RI (3.78%) compared to other formulations, indicating better interaction between n-APKS and the NR matrix, which restricted the movement of rubber chains. This results in the rubber vulcanizates becoming stiff and rigid, enhancing the mechanical interlocking between n-APKS and the NR phase (Ismail and Shaari 2010; Amoke *et al.* 2021). These results show that high crosslink density and low EB corresponded to high M_{300} and high RI. However, as n-APKS loading continued to increase, the M_{300} and RI started to decrease from F4 to F5 by 1% and 18%, and from F4 to F6 by 5% and 25%, respectively. This decrease can be explained by the poor dispersion of n-APKS particles in the NR matrix (Fig. 6), which leads to weak interfacial bonding and reduced stiffness (Salaeh and Nakason 2012; Amoke *et al.* 2021).

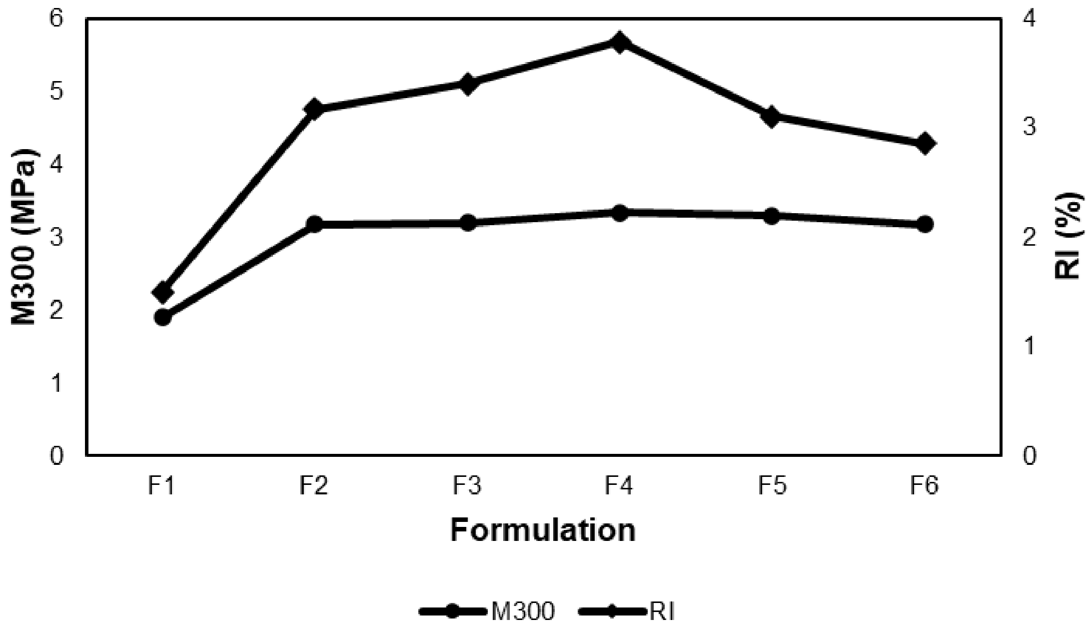

Fig. 10. M300 and RI of n-APKS-filled NR vulcanizates

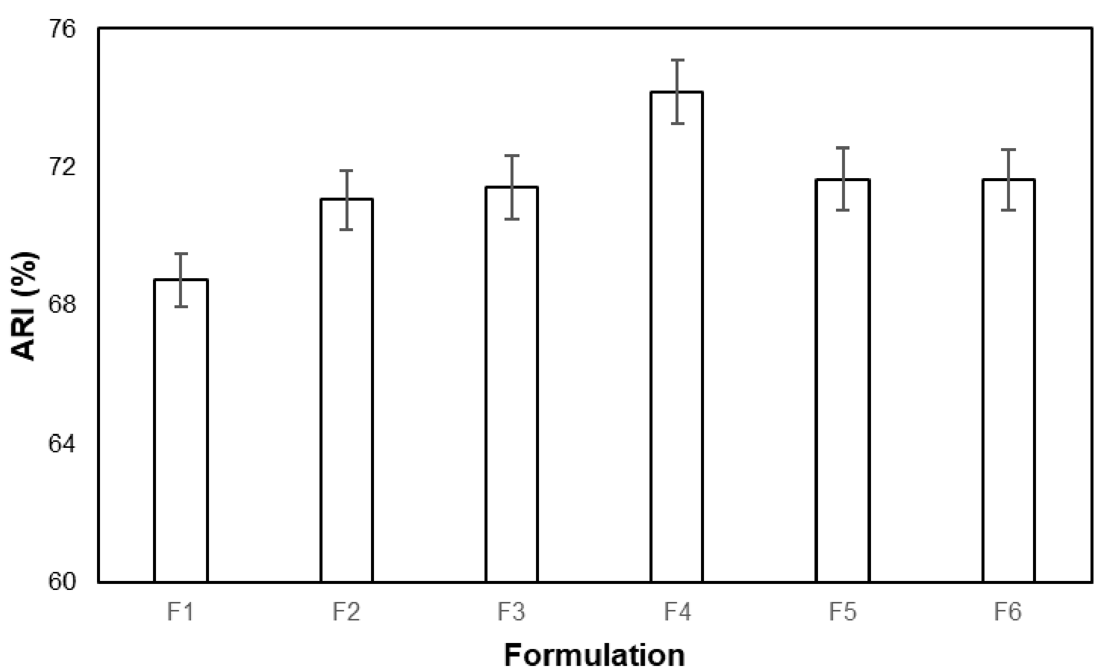

Fig. 11. ARI of n-APKS-filled NR vulcanizates

Abrasion Resistance Index (ARI)

The ARI of n-APKS-filled NR vulcanizates is presented in Fig. 11. F1 had the lowest ARI (22%) due to the absence of n-APKS, resulting in low resistance to surface damage (Zafirah *et al.* 2023). With the addition of n-APKS, the ARI started to increase from F1 to F2 by 31%, indicating the influence of n-APKS in creating interactions with the NR matrix to provide wear resistance (Daud *et al.* 2017). This increasing trend continued from F2 to F3 by 8% and from F2 to F4 by 33% with F4 exhibiting the highest ARI (38%) compared to other formulations. This is due to the better dispersion and distribution of n-APKS in the NR matrix, contributing to better mechanical interlocking and interfacial bonding between n-APKS and the NR matrix. This provides good wear resistance against surface damage of the NR vulcanizates (Sae-Oui *et al.* 2017; Malomo *et al.* 2020). These results are supported by the crosslink density and M_{300} values of F4. As n-APKS loading continued, the ARI started to decrease from F4 to F5 by 12% and from F4 to F6 by 17%. This could be explained by poor mechanical interlocking due to poor distribution and dispersion of n-APKS, leading to agglomeration (Fig. 6) and resulting in poor mechanical resistance and consequently poor abrasion resistance (Vishvanathperumal and Anand 2022).

RECOMMENDATION

A promising recommendation for future research would be to further explore the potential of n-APKS as a bio-filler in rubber formulations, particularly by investigating its usage together with other fillers such as carbon black (CB) or silica to further enhance the performance of natural rubber (NR). Future studies should focus on optimizing the loading of n-APKS in combination with other fillers to achieve a balanced improvement in both mechanical and processing properties. Additionally, conducting a techno-economic analysis could provide valuable insights into the cost-effectiveness and sustainability of using n-APKS in industrial applications. This could help assess the feasibility of scaling up the use of n-APKS in the rubber industry while addressing environmental waste management issues associated with oil palm by-products.

CONCLUSIONS

The incorporation of nano-sized active palm kernel shell (n-APKS) into natural rubber (NR) vulcanizates resulted in improved mechanical properties, including enhanced cure characteristics, bound rubber content (BRC), and resistance to swelling, tensile strain, and abrasion.

Based on the findings, the optimal loading of n-APKS was determined to be 5 phr, at which point the dispersion of n-APKS particles within the natural rubber matrix was most effective. Beyond this loading, further increases in n-APKS led to the formation of filler-filler clusters, causing a decline in properties.

At the optimal 5 phr loading, n-APKS improved interaction, mechanical interlocking, and interfacial bonding, leading to a high crosslink density (8.65 x10^{-5} mol/cm^3), tensile strength (19 MPa), M_{300} (3.33 MPa), reinforcement index (RI) (3.78%), and abrasion resistance (38%), while maintaining a low elongation at breakage (EB) (648%). Thus, n-APKS proves to be a suitable bio-filler for reinforcing rubber, significantly enhancing the properties of NR.

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