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Research Article

Strengthening Natural Rubber with Activated Carbon from Cassava Rhizome Waste: Cure Characteristics, Physical, Thermal, and Mechanical Properties

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Abstract

This study aims to develop a bio-reinforcing filler from cassava rhizome waste, a common agricultural residue in northeastern Thailand that generates a significant amount of waste annually. The waste was turned into activated carbon (AC) by activating it with potassium hydroxide (KOH) and microwave irradiation. AC was then used as a reinforcing agent in natural rubber (NR) composites. The effects of KOH concentration and AC content on the cure characteristics, as well as the physical, thermal, and mechanical properties of the composites, were investigated. At optimal AC content, scorch time decreased by 13.74%, torque difference increased by 17.20%, and cure time was reduced by 2.90%. Mechanical properties improved with higher AC content, with AC prepared at lower KOH concentration exhibiting superior performance. The swelling index decreased with increasing AC content, indicating enhanced solvent resistance. Utilizing cassava rhizome-derived AC offers significant environmental benefits by repurposing agricultural waste. Future research could optimize preparation processes to further enhance performance and explore industrial applications. This study highlights the potential of cassava waste as an eco-friendly and sustainable filler for rubber composites.

Keywords: Activated carbon, Cassava rhizome, Mechanical properties, Natural rubber, Physical properties, Thermal properties

1 Introduction

Activated carbon, also known as activated charcoal (AC), is a carbon material produced from carbonaceous sources and is characterized by its highly porous structure, which provides a large surface area. The production of AC involves two main processes: physical activation and chemical activation. Chemical activation is more commonly used due to its lower activation temperature, higher yield, shorter processing time, and greater effectiveness in producing higher porosity and surface area [1], [2]. Previous studies have shown that the effectiveness of the chemical activation process depends on both the type and concentration of the chemical activator used. For example, Chaisit *et al.* [3] demonstrated that activated carbon derived from cassava root, prepared using the chemical activation process with potassium hydroxide (KOH), exhibited an increase in surface area as the KOH concentration increased. Similarly, Sujiono *et al.* [2] found that AC produced from coconut shells using different chemical activators achieved the highest surface area (516 m^2/g) when sodium hydroxide (NaOH) was used, compared to zinc chloride ($ZnCl_2$) and phosphoric acid (H_3PO_4).

Normally, commercial AC is prepared from coke, peat, and petroleum coke [4]. However, researchers are increasingly focusing on biomass feedstock and renewable energy resources. AC can be prepared from various materials, including agricultural waste such as rice husks [5], [6], bamboo [7], [8], coconut shells [2], [9], cassava roots [3], [10], and coffee grounds [11]. The use of agricultural waste as a raw material for AC production is particularly appealing as it not only addresses waste disposal problems but also reduces the demand for non-renewable resources and helps to mitigate climate change. Moreover, producing AC from these wastes can be cost-effective and contribute to sustainable development by converting waste into valuable products. The high porosity, mechanical strength, and adsorption capacity of AC make it effective for various applications. It is widely used in water and air purification systems, where its adsorption properties help remove impurities and contaminants. As an example, bamboo-based activated carbon has a lot of micropores and a high specific surface area (1175 m^2/g), which makes it better at absorbing CO_2. It can absorb 0.98 mmol/g (43.13 mg/g) of 3000 ppm CO_2 [7]. AC is also utilized in energy storage devices such as supercapacitors, where its high surface area contributes to increased energy density [3]. Research has further shown that nanosheet-structured AC, prepared from red onion skin waste, is a promising candidate for supercapacitor and hydrogen storage applications [12]. Additionally, AC serves as a reinforcing agent in polymer composites, improving their mechanical properties and durability [13], [14]. Yurtary *et al.* [15] discovered that incorporating AC into a polypropylene matrix enhanced its tensile modulus in comparison to pure polypropylene. These diverse applications underscore the potential of AC in industrial and environmental technologies, making it a subject of significant interest for researchers and industries alike.

Natural rubber (NR) is a polymer that often requires reinforcement to enhance its mechanical, thermal, and physical properties [16], [17]. Research has demonstrated that fillers enhance the stiffness, tensile strength, modulus, and wear resistance of NR by strengthening the rubber matrix. The filler particles create a network that restricts rubber chain mobility, thereby enhancing its mechanical performance [18]. Traditionally, reinforcing agents such as carbon black [19], [20] and silica [21], [22] have been commonly added to NR to enhance these properties. However, in recent years, research has increasingly focused on using bio-fillers as sustainable alternatives to carbon black, due to their potential to reduce the use of non-renewable resources [23], [24]. Examples of these bio-fillers include rice husks [25], [26], coffee grounds [27], biochar [28], [29], and activated carbon [13], [14], which not only contribute to the circular economy by utilizing agricultural waste but also offer reinforcing properties comparable to those of traditional fillers. Moreover, bio-fillers can enhance the biodegradability of NR composites, aligning with sustainability goals. Incorporating bio-fillers can also reduce composite costs without compromising performance. Ismail *et al.* [14] studied the mechanical properties of natural rubber filled with charcoal-activated carbon and carbon black at loadings of 10 and 15 phr. They observed that both fillers increased the tensile strength and modulus. However, carbon black resulted in slightly higher density, hardness, and tensile properties, along with lower swelling compared to charcoal-activated charcoal. The study concluded that to match the efficiency of carbon black, a higher loading of activated charcoal is necessary. In another study, Lay *et al.* [13] found that activated carbon made from dead leaf biomass could serve as a substitute for carbon black. Their results showed that adding 15 phr of activated carbon increased the tensile strength of rubber composites by 8% and raised the hardness to 48 Shore A. However, these properties were still lower than those of composites with carbon black, suggesting that further optimization may be needed to match the performance of carbon black.

Eyo-Honesty and Evudiovo [30] prepared AC from the periwinkle shell using chemical activation with KOH and heat treatment at 800 °C for 2 h, finding that this AC enhanced the mechanical properties of NR. Similarly, Al-Ghamdi *et al*. [31] prepared AC from coconut shells via steam activation and used it as a reinforcing agent in NR composites, which also improved mechanical properties. However, both methods require long activation times and high temperatures. In contrast, microwave-assisted activation for preparing AC offers a safer and more efficient process, minimizing pollution and significantly reducing processing time. This method presents a unique advantage over traditional activation techniques. Optimizing mechanical properties in rubber composites depends significantly on key factors like filler content and particle size. For instance, Tan and Isayev [32] and Al-Maamori *et. al*. [33] showed that adding fillers like carbon black and silica improved the mechanical properties of rubber, including hardness, modulus, and tensile strength, with optimal performance achieved at certain filler loadings. Al-Maamori *et al*. [33] observed that these improvements peaked at an optimal filler loading of 60 phr before slightly decreasing, highlighting the importance of precise filler content for maximizing performance. Chuayjulit *et al*. [34] further found that smaller filler particle sizes enhanced mechanical properties by increasing viscosity, reducing scorch time, and improving the overall performance of rubber.

In northeastern Thailand, cassava is one of the most important economic crops, leading to a significant amount of cassava rhizome waste after harvesting [35]. This waste offers an opportunity for sustainable utilization, particularly due to the high glucose content (≥90%) in cassava starch, making it an ideal material for producing AC [3]. Therefore, the goal of this research is to produce AC from cassava rhizomes for use as NR's reinforcing filler. The cure characteristics, morphology, thermal, mechanical, and physical properties of the NR filled with cassava rhizome AC were investigated. Additionally, the research examines the effects of KOH concentration on the properties of NR composites, thereby contributing to environmental sustainability and economic efficiency by enhancing material performance and addressing agricultural waste management.

2 Materials and Methods

2.1 *Materials*

The cassava rhizome was collected from Nakhon Ratchasima Province, Thailand. Potassium hydroxide (KOH, Kemaus, 85%) was used for the activation process, while hydrochloric acid (HCl, Labo Chemie PVT. LTD., 35.4%) was used for the neutralization process. Natural rubber (STR 5L), stearic acid, zinc oxide (ZnO), and tetramethyl thiuram disulfide (TMTD) were supplied by Chareon Tut Co., Ltd., Thailand. All chemicals were used without purification.

2.2 *Activated carbon preparations*

Figure 1 shows the flowchart of the activated carbon preparation process. First, cassava rhizomes (brown in color) were washed and sun-dried for two days. They were then pyrolyzed in a homemade furnace at approximately 600 °C for 4–6 h. After cooling, the resulting cassava rhizome biochar (black in color) was milled with a high-speed blender and sieved using a stainless-steel sieve with a 270-mesh size (53 µm).

The cassava rhizome biochar (pH 7) was then soaked in a potassium hydroxide (KOH) solution (pH 14) for 24 h as part of the chemical activation process. KOH, a strong base, increases the surface area and porosity of the biochar by breaking down its carbon structure, offering greater porosity compared to other chemicals used in activation methods [36]. The mechanism of the reaction between KOH and biochar was proposed by Raymundo-Piñero *et al*. [37], as expressed in Equations (1)–(4).

$$6KOH + 2C \leftrightarrow 2K + 3H_2 + 2K_2CO_3 \quad (1)$$

$$K_2CO_3 + C \leftrightarrow K_2O + 2CO \quad (2)$$

$$K_2CO_3 \leftrightarrow K_2O + CO_2 \quad (3)$$

$$2K + CO_2 \leftrightarrow K_2O + CO \quad (4)$$

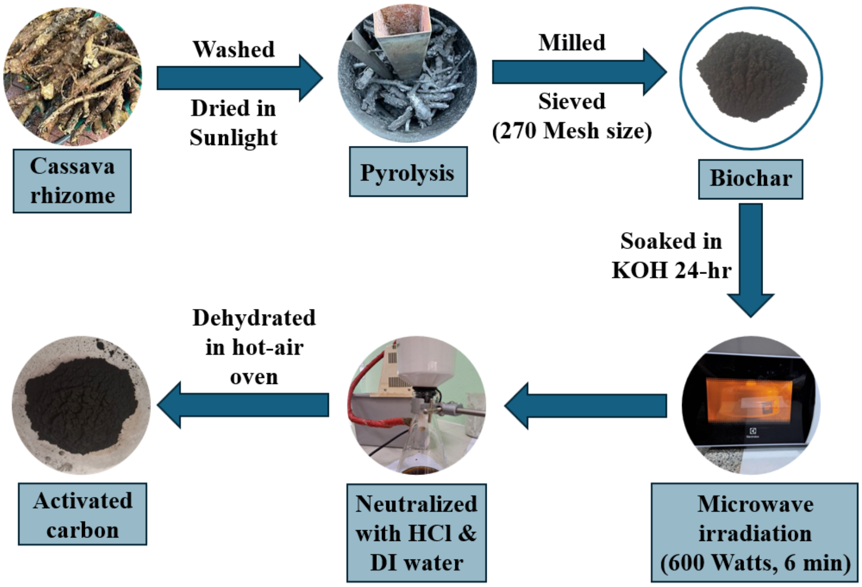

Figure 1: Schematic representation of the activated carbon preparation process.

The biochar was subsequently heated using microwave irradiation at 600 watts for 6 min. The obtained AC was neutralized with hydrochloric acid (HCl) until it reached pH 7 and washed three times with deionized water. Finally, the AC was dried in an oven for 24 h.

In this work, two types of cassava rhizome AC were prepared using different concentrations of KOH. The biochar-to-KOH ratios used were 1:1 and 1:5 by weight in 250 mL of deionized water, based on previous studies [3], which showed that biochar-to-KOH ratios varying from 1:1 to 1:5 influence the specific surface area and total pore volume. The AC samples with KOH concentration ratios of 1:1 and 1:5 were denoted as AC1 and AC5, respectively. The yields of AC1 and AC5 were 93% and 83%, respectively.

2.3 *Activated carbon characterization*

The morphology and chemical composition of the cassava rhizome AC were characterized using scanning electron microscopy (SEM, AURIGA, Carl Zeiss) coupled with energy dispersive spectroscopy (EDS). The surface area and total pore volume of AC were analyzed via nitrogen adsorption using the Brunauer-Emmett-Teller (BET) method following a degassing procedure at 300 °C for 6 h. The particle size distribution of the AC was analyzed using a laser scattering particle size distribution analyzer (HORIBA, LA-950V2).

2.4 *Natural rubber compound preparation*

The natural rubber (NR) compound was blended using a two-roll mill (ML–D6L12–INV, Chareon Tut Co., Ltd., Thailand) with a roller speed ratio of 1:4 between the front and back rolls. During the compounding process, NR was masticated for 10 min. Then, every 4 min, the rubber chemical reagent was added as follows: stearic acid 2.5 phr, zinc oxide (ZnO) 5 phr, tetramethyl thiuram disulfide (TMTD) 0.7 phr, activated carbon (AC1 or AC5) at 0, 5, 10, or 15 phr, followed by sulfur 2.5 phr. Once fully mixed, the compound formed into sheets and kept at room temperature for 24 h before the cure characteristics analysis and vulcanization process.

2.5 *Cure characteristic and thermal analysis*

The cure characteristics of the NR compounds were investigated using a moving die rheometer (MDR 3000, MonTech, Taiwan) at 160 °C. Three samples of each compound were examined.

To investigate the glass transition temperature and cure temperature of the NR compounds, a differential scanning calorimeter (DSC, Model DSC 1, Mettler–Toledo) was used. The measurement temperature was in the range of –100 °C to 250 °C with a heating rate of 10 °C/min under a nitrogen atmosphere.

2.6 *Sample preparation*

The natural rubber compounds were cured in a hot compression mold (Chareon Tut Co., Ltd., PR2DW300L350 PM–WCL–HMI) at 160 °C for 5 min. The mold was made of stainless steel, and the pressure was 1500 psi. After the vulcanization process, a sheet of vulcanized rubber was cut with a die cut. For mechanical testing, die C type, commonly used for rubber material testing according to ASTM D412, was employed to prepare the samples.

2.7 *Mechanical testing*

Tensile testing was performed on dumbbell-shaped specimens following ASTM D412–06 using a universal testing machine (LS Plus Series, Lloyd). A load cell of 10 kN and a crosshead speed of 500 mm/min were used at room temperature. Five specimens were tested for each ratio.

The tear strength was tested according to ASTM D624–98 using a universal testing machine (LS Plus Series, Lloyd) at ambient temperature. A load cell of 10 kN and a crosshead speed of 500 mm/min were used. At least five samples were tested and evaluated.

The hardness of vulcanized rubber was tested following ASTM D2240–03 using a durometer (GS–612, TECLOCK) as Shore A. Five positions of the samples were investigated and evaluated.

2.8 *Physical testing*

The swelling of vulcanized rubber was examined using toluene as the solvent. The diameter and thickness of the specimen were 1.3 mm and 6 mm, respectively. The sample was weighed before (W_o) and after soaking (W_f) in toluene for 24 h. At least five specimens were investigated for each ratio. The swelling index was calculated using Equation (5).

$$Swelling\ \ index\ \ (\%) = \frac{\left(W_f - W_o\right)}{W_o} \times 100 \tag{5}$$

The vulcanized natural rubber was weighed in ethanol (w_e) and air (w_a) to calculate the density using Equation (6). The density of ethanol is 0.79 g/cm^3. Three specimens were used in each ratio to determine the average density.

$$Density\left(g/cm^3\right) = \frac{w_a}{\left(w_a - w_e\right)} \times 0.79 \tag{6}$$

2.9 *Morphology*

The fractured surface of the tested tensile specimen was examined via a scanning electron microscope (SEM, AURIGA, Carl Zeiss). The sample was coated with gold to improve its conductivity and reduce the charging effect.

3 Results and Discussion

3.1 *Activated carbon properties*

The morphology of cassava rhizomes AC is shown in Figure 2. Both AC1 and AC5, prepared with biochar-to-KOH concentration ratios of 1:1 and 1:5, respectively, exhibited similar flake–shaped morphologies with varying sizes. The outer structures of activated carbon were observed to be somewhat porous and unfinished, as indicated by the red circles. This observation aligns with the study by Poolprasert and Chorchong [10], who reported that activated carbon derived from cassava roots exhibits porous structures.

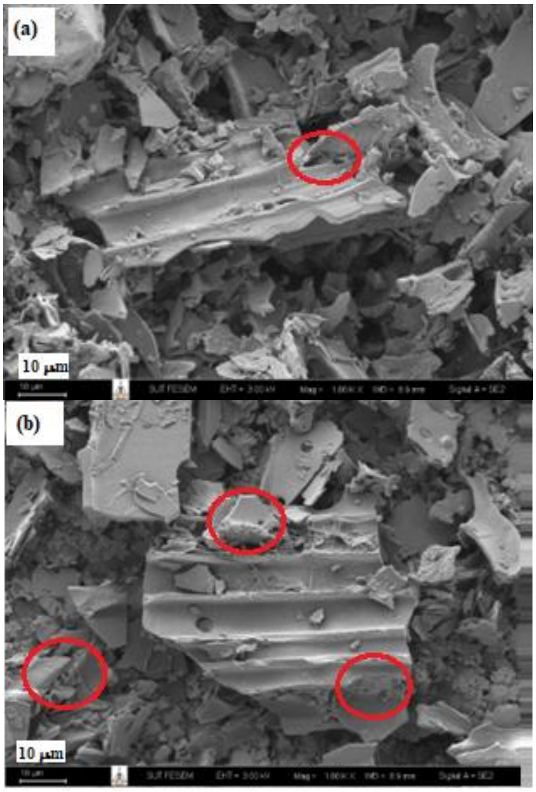

Figure 2: SEM micrographs of cassava rhizome AC; (a) AC1; (b) AC5.

The chemical compositions of AC1 and AC5 are presented in Table 1. The results indicate that carbon (C) is the predominant element, constituting 81.6% of AC1 and 72.8% of AC5, with AC1 having a higher carbon content compared to AC5. Oxygen (O) was the second most abundant element, followed by trace amounts of potassium (K), calcium (Ca), and nitrogen (N).

Table 2 shows the surface area, total pore volume, and particle size of AC. The surface area and total pore volume of AC5 were 17.62 m^2/g and 0.0125 cm^2/g, respectively, which were higher than those of AC1. This may have been due to the higher KOH concentration in AC5, likely causing a more intense reaction on the biochar surface, consistent with findings from previous studies [3]. Furthermore, the number-average particle sizes of AC1 and AC5 were 19.07 ± 12.06 µm and 23.29 ± 25.22 µm, respectively, indicating that AC1 had a smaller particle size than AC5.

However, the higher surface area of AC5 can be attributed to its significantly larger total pore volume, enhancing the accessible area for interactions. As shown in Figure 2, AC5 exhibits a more porous structure compared to AC1, which compensates for its larger particle size and results in a greater overall surface area. While particle size influences surface area, the porous structure of AC5 plays a more critical role in this enhancement [38]. This finding corresponds to the preparation of AC from lotus stalks by Yang *et al*. [39], which showed that increased KOH concentrations and higher temperatures used for chemical activation led to increased porosity and specific surface area in lotus stalk-derived AC.

Table 1: Chemical composition of cassava rhizome AC.

Element Content (%)	**Activated Carbon**	
	AC1	**AC5**
C	81.6	72.8
O	14.1	20.4
K	3.0	1.0
Ca	1.3	-
N	-	5.8

Table 2: Surface area, total pore volume, and particle size of cassava rhizome AC.

Activated Carbon	**S_{BET} (m^2/g)**	**Total Pore Volume (cm^3/g)**	**Particle Size (µm)**
AC1	6.86	0.0093	19.07 ± 12.06
AC5	17.62	0.0125	23.29 ± 25.22

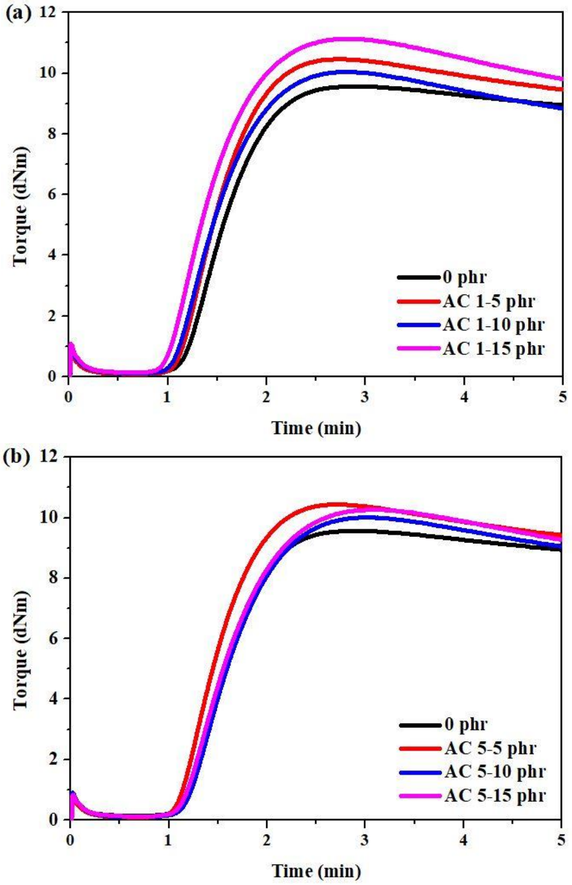

Figure 3: Vulcanization curve obtained after 5 min at 160 °C; (a) AC1; (b) AC5.

3.2 *Cure characteristics*

Figure 3 depicts the rheology of rubber composites. The effects of KOH concentration and AC content on the cure characteristics of NR compounds are shown in Table 3. Scorch time (t_{s2}) refers to the flowability of rubber before the curing process. It was found that the addition of AC tended to decrease the scorch time, suggesting a good interaction between AC and the rubber matrix [14]. The maximum torque (M_H) of the NR compound increased steadily with rising AC content, indicating an enhanced overall network structure. This finding aligns with that of Raju *et al*. [40], who also observed that higher AC content contributed to an increased maximum torque by strengthening the network structure. The torque difference (M_H – M_L) is the difference between maximum torque (M_H) and minimum torque (M_L), which refers to the magnitude of the crosslink density of rubber [41]. The results showed that the torque difference increased with an increase in the AC content. This is because the AC particles in the rubber formed a network. This network makes it harder for polymer chains to move around, which makes them less likely to deform and increases the torque during vulcanization. This behavior indicates that the addition of AC increased the crosslink density. Moreover, the addition of AC1 resulted in a slightly higher torque difference compared to the addition of AC5.

Table 3: Cure characteristics of NR/AC composites.

Sample	Amount (phr)	t_{s2} (min)	t_{c90} (min)	M_H (dN.m)	M_L (dN.m)	M_H-M_L (dN.m)	CRI (min^{-1})
NR/AC1	0	1.31	2.07	9.53	0.30	9.23	129.87
	5	1.24	2.01	10.46	0.25	10.21	129.87
	10	1.21	2.05	10.03	0.31	9.72	119.05
	15	1.13	2.01	11.17	0.35	10.82	113.63
NR/AC5	5	1.30	2.08	9.62	0.22	9.40	126.58
	10	1.33	2.21	10.02	0.28	9.74	113.63
	15	1.30	2.23	10.30	0.32	9.98	107.52

t_{s2}, t_{c90}, M_H, M_L, M_H-M_L, and CRI are scorch time, cure time, maximum torque, minimum torque, torque difference, and cure rate index respectively.

In addition to the effects on scorch time and maximum torque, the study also examined the influence of AC content on the cure time (t_{c90}) of NR compounds. Cure time refers to the period required for the full vulcanization of rubber. The results showed that the addition of AC1 decreased the cure time, while the addition of AC5 increased it. The decrease in cure time might have been due to the heat and energy generated during the process [14]. Conversely, the increase in cure time might have been a result of the higher surface area, which may have absorbed the zinc complex necessary for basic accelerators in the vulcanization process [27]. Moreover, the cure rate index (CRI) was affected by the type and amount of AC. The CRI of bare NR is 129.87 min^{-1}. The addition of AC tended to decrease the CRI, which aligns with findings from the addition of carbon black and biochar as reported by Lubura *et al*. [42]. A more significant reduction in CRI was found with AC5 than with AC1, with the lowest CRI recorded for the rubber compound containing 15 phr of AC5 (107.52 min^{-1}).

3.3 *DSC thermogram*

The DSC thermogram of the NR compound showed a glass transition temperature (T_g) of –63.0 °C, consistent with previous findings [43]. The addition of AC to the NR slightly shifted the T_g to a lower temperature, as shown in Figure 4 and Table 4, likely due to the creation of more free volume upon the addition of AC. The onset temperature of the NR began at 154.2 °C and was enhanced with the addition of AC, as indicated in Table 4. Additionally, an exothermic peak of 165 °C represents the vulcanization point, where crosslinking of the rubber chains occurs. The addition of AC tended to increase the curing temperature, suggesting enhanced thermal stability in NR. Notably, AC5 showed a greater improvement in the vulcanization peak compared to AC1. This difference can be attributed to the higher porosity of AC5, which leads to a larger surface area. This increased surface area allows AC5 to absorb more heat during the vulcanization process, thereby requiring a higher temperature for curing. This result aligns with the study by Mekbuntoon *et al*. [44], which demonstrated that a higher surface area of AC can improve the vulcanization efficiency of AC/NR composites by providing more active sites for the curing process.

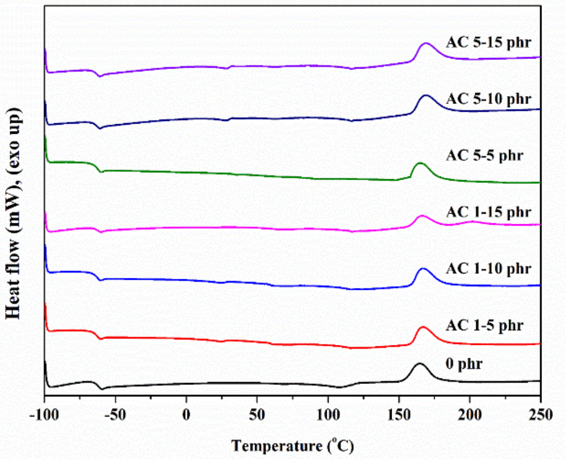

Figure 4: DSC thermograms of NR/AC composites.

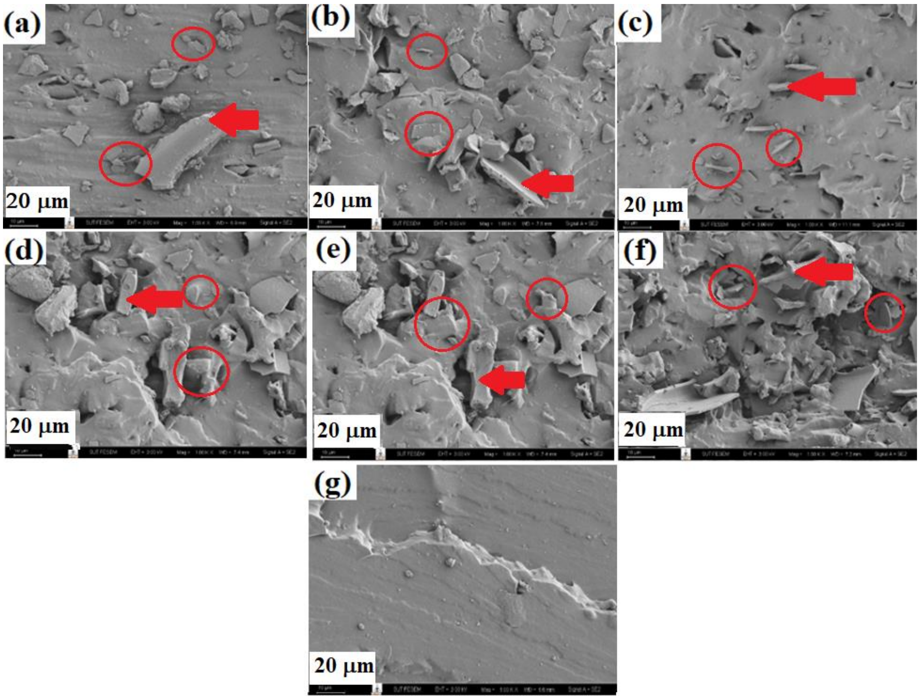

Figure 5: SEM micrographs of vulcanized natural rubber filled with cassava rhizome AC: AC1; (a) 5 phr; (b) 10 phr; (c) 15 phr: AC5; (d) 5 phr; (e) 10 phr; (d) 15 phr; (g) 0 phr. Red arrows indicate the AC. A scale bar is equal to 20 µm.

Table 4: Thermal properties of NR/AC composites.

Sample	Amount (phr)	Temperature (°C)		
		T_g	Onset curing	Peak curing
NR/AC1	0	–63.0	154.2	164.8
	5	–63.9	157.3	165.5
	10	–64.4	158.9	167.0
	15	–64.3	157.5	166.0
NR/AC5	5	–63.9	157.4	165.2
	10	–64.3	158.8	167.7
	15	–64.6	159.7	168.8

3.4 *Morphology*

The fractured surfaces of vulcanized natural rubber are shown in Figure 5. Figures 5(a)–(f) reveal that the addition of cassava rhizome AC resulted in a rougher surface and disrupted the rubber matrix. AC particles (indicated by red arrows) were well-distributed throughout the NR matrix, exhibiting various particle sizes and strong adhesion between the AC and rubber, as highlighted by the red circle. In contrast, the smooth surface of unfilled NR is illustrated in Figure 5(g).

3.5 *Mechanical and physical properties*

The mechanical properties of vulcanized rubber with varying cassava rhizome AC content are shown in Table 5. The tensile strength and 300% modulus of NR were 7.0 ± 1.8 MPa and 1.4 ± 0.1 MPa, respectively. The addition of cassava rhizome AC enhanced both the tensile strength and 300% modulus of NR, likely due to the high carbon content in AC, which improves interactions within the rubber matrix and thus strengthens both properties [45]. These results align with Ismail *et al*. [14], who observed similar improvements with charcoal AC; however, cassava rhizome AC showed even higher tensile strength and modulus at similar loading levels, making NR composites filled with cassava rhizome AC suitable for applications requiring enhanced strength and durability, such as automotive components, industrial belts, and vibration dampers.

In addition to these mechanical properties, the hardness (Shore A) of vulcanized rubber increased slightly when cassava rhizome AC was added, as AC particles restrict the mobility of rubber chains and reduce deformation under stress [46]. The hardness of

NR/AC1 at 15 phr was comparable to that of NR filled with AC derived from dead leaves (48 Shore A), as observed by Lay *et al*. [13]. AC1 exhibited a marginally greater improvement in hardness compared to AC5, likely due to AC1's smaller particle size, which enhances contact and cross-linking with the rubber matrix, leading to stronger interfacial bonding and, consequently, higher hardness [47]. This increased hardness suggests suitability for products like shoe soles and flooring materials, which require abrasion resistance and dimensional stability.

Table 5: Mechanical properties of NR/AC composites.

Sample	Amount (phr)	Tensile Strength (MPa)	300% Modulus (MPa)	Tear Strength (kN/m)	Hardness (Shore A)
NR/AC1	0	7.0 ± 1.8	1.4 ± 0.1	7.3 ± 0.3	41.4 ± 0.6
	5	9.6 ± 0.7	1.7 ± 0.1	7.2 ± 0.1	43.5 ± 0.6
	10	9.7 ± 0.8	1.8 ± 0.1	8.7 ± 0.6	43.7 ± 1.2
	15	12.6 ± 0.5	2.4 ± 0.1	10.6 ± 0.8	47.7 ± 0.4
NR/AC5	5	8.2 ± 1.0	1.4 ± 0.1	7.5 ± 0.5	40.1 ± 0.7
	10	8.8 ± 0.6	1.7 ± 0.2	9.1 ± 0.7	41.6 ± 0.3
	15	11.1 ± 0.4	2.1 ± 0.1	10.8 ± 0.5	44.5 ± 0.3

Moreover, the tear strength of NR increased with higher cassava rhizome AC content, which can be attributed to enhanced interfacial adhesion between the AC and the rubber matrix, which facilitates stress distribution and effectively hinders tear propagation [45]. In comparison, Ribeiro *et al*. [48] reported that the tear strength of bare NR increased by 27.6% with the addition of carbon black at 50 phr. In this study, the tear strength increased by 45.2% with only 15 phr of cassava rhizome AC, indicating a more significant enhancement with cassava rhizome AC. Interestingly, the addition of AC1 resulted in slightly better mechanical properties compared to AC5. This may be due to AC5's higher surface area and pore volume, which could absorb more rubber chemical reagents [27], potentially leading to a decrease in crosslink density. This improvement suggests the potential for use in applications such as tire treads and conveyor belts, where high tear strength is crucial.

Figure 6 illustrates the swelling index of NR/AC composites in a toluene solvent. The results, derived using Equation (5) and the values shown in Table 6, show that the swelling index of NR decreased with an increase in the AC content, indicating enhanced solvent resistance with added AC. This improvement is likely due to the increased interaction and cross-linking between AC particles and the rubber matrix, which restricts the movement of rubber chains and thus limits solvent penetration [49].

However, the addition of AC5 resulted in a higher swelling index compared to AC1, suggesting that AC1 provides better solvent resistance. The smaller particle size of AC1 likely contributes to a more uniform dispersion within the rubber matrix, leading to a denser network that more effectively restricts solvent uptake. Additionally, smaller particles generally have higher surface reactivity, which can promote stronger interfacial bonding and increased crosslink density [50]. In contrast, AC5, with its larger particle size and higher surface area, may absorb more rubber chemical reagents, potentially reducing effective crosslinking and decreasing resistance to swelling in solvents such as toluene.

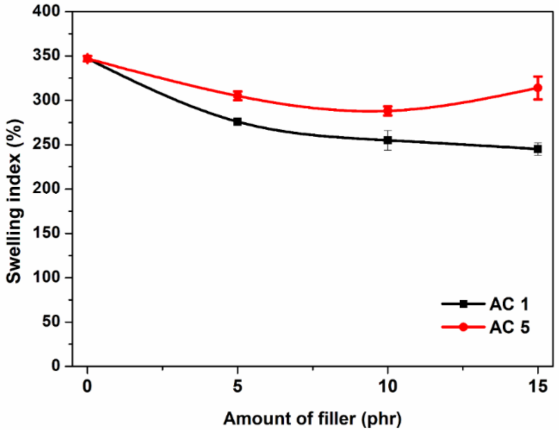

Figure 6: Swelling index of NR/AC composites (Lines are used as visual guidelines).

Table 6: Bulk density of NR/AC composites.

Sample	Amount (phr)	Swelling Index (%)	Bulk Density (g/cm^3)
NR/AC1	0	347 ± 3	0.9717
	5	276 ± 2	0.9921
	10	255 ± 11	0.9999
	15	245 ± 7	1.0152
NR/AC5	5	305 ± 5	0.9896
	10	288 ± 5	1.0184
	15	314 ±13	1.0247

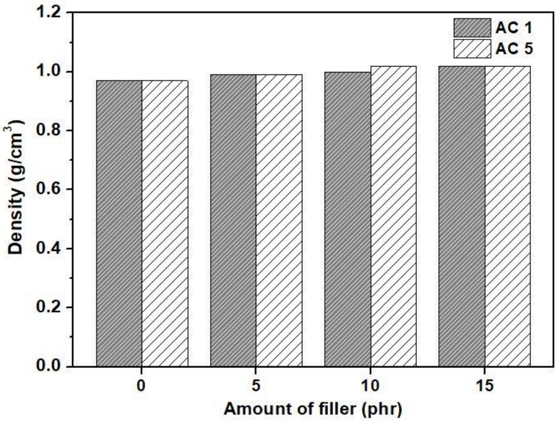

Figure 7: Bulk density of vulcanized natural rubber filled with cassava rhizome AC.

Figure 7 illustrates the bulk density of vulcanized rubber filled with cassava rhizome AC, which shows an increase in density with higher AC content, as presented in Table 6. This increase may be attributed to enhanced crosslink density in the compound, as more AC particles create additional bonding points within the rubber matrix, restricting polymer chain movement and contributing to a denser network structure [50]. This result showed a similar trend to the torque results previously discussed and is consistent with findings of Kim *et al*. [51], who reported that increasing carbon black content also led to greater density in their formulations. Additionally, the bulk density of rubber with AC5 was slightly higher than that with AC1, likely due to AC5's larger particle size and surface area, which may enable slightly greater filler-matrix interactions.

4 Conclusions

Cassava rhizome-activated carbon (AC), which is made using KOH and microwave irradiation, is more effective and better for the environment than traditional activation methods. This makes composite production more sustainable. Microwave irradiation not only reduces activation time but also yields a higher percentage of AC, making the process more productive and cost-effective. This AC is primarily composed of carbon, with higher KOH concentrations producing AC with increased surface areas. The addition of 15 phr AC, produced with lower KOH concentrations, resulted in a higher torque difference (10.82 dN.m), shorter cure times (1.13 min), and reduced curing temperatures. The cassava rhizome AC effectively enhanced the mechanical properties of natural rubber, with tensile strength reaching 12.6 MPa and hardness increasing to 47.7 Shore A. Compared to carbon black, cassava rhizome AC offers sustainability advantages, being derived from renewable agricultural waste with less energy-intensive production. While its performance may not yet fully match carbon black, its eco-friendliness and cost-effectiveness make it a viable alternative. This innovation supports the development of sustainable rubber products, such as shoe soles and mats, aligning with industry goals. Future studies will investigate the effects of higher cassava rhizome AC content beyond 15 phr on the mechanical, thermal, and solvent resistance properties of the composites, optimizing filler content for specific industrial applications.

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Author Contributions

N.P., K.N., and I.S.: carried out the experiments; N.P., P.S., I.S., W.S., N.S., P.M, P.C., and K.N.: coordinated the project; N.P., N.S., P.S., W.S., N.S., and K.N.: analyzed the data and wrote the manuscript; N.P., I.S., and K.N.: prepared the graphs. The findings were discussed, and the manuscript was reviewed by all authors.

Conflicts of Interest

The authors declare no conflict of interest.

References

[1] B. Wang, J. Lan, C. Bo, B. Gong, and J. Ou, “Adsorption of heavy metal onto biomass-derived activated carbon: Review,” *RSC Advances*, vol. 13, pp. 4275–4302, 2023, doi: 10.1039/d2ra07911a.

[2] E. H. Sujiono, D. Zabrian, Zurnansyah, Mulyati, V. Zharvan, Samnur, and N. A. Humairah, “Fabrication and characterization of coconut shell activated carbon using variation chemical activation for wastewater treatment application,” *Results in Chemistry*, vol. 4, 2022, Art. no. 100291, doi: 10.1016/j.rechem.2022.100291.

[3] S. Chaisit, N. Chanlek, J. Khajonrit, T. Sichumsaeng, and S. Maensiri, “Preparation, characterization, and electrochemical properties of KOH-activated carbon from cassava root,” *Materials Research Express*, vol. 7, 2020, Art. no. 105605, doi: 10.1088/2053-1591/abbf84.

[4] N. A. Basha, T. Rathinavel, and H. Sridharan, “Activated carbon from coconut shell: Synthesis and its commercial applications- A recent review,” *Applied Science and Engineering Progress*, vol. 16, no. 2, 2023, Art. no. 6152, doi: 10.14416/j.asep.2022.07.001.

[5] M. M. Alam, M. A. Hossain, M. D. Hossain, M. A. H. Johir, J. Hossen, M. S. Rahman, J. L. Zhou, A. T. M. K. Hasan, A. K. Karmakar, and M. B. Ahmed, “The potentiality of rice husk-derived activated carbon: From synthesis to application,” *Processes*, vol. 8, p. 203, 2020, doi: 10.3390/pr8020203.

[6] M. S. Yerdauletov, K. Nazarov, B. Mukhametuly, M. A. Yeleuov, C. Daulbayev, R. Abdulkarimova, A. Yskakov, F. Napolskiy, and V. Krivchenko, “Characterization of activated carbon from rice husk for enhanced energy storage devices,” *Molecules*, vol. 28, p. 5818, 2023, doi:10.3390/molecules28155818.

[7] S. Heo, W. Kim, Y. Jo, and A. A. Adelodun, “Fabrication of bamboo-based activated carbon for low-level CO_2 adsorption toward sustainable indoor air,” *Sustainability*, vol. 16, p. 1634, 2024, doi:10.3390/su16041634.

[8] D. A. Khuong, H. N. Nguyen, and T. Tsubota, “Activated carbon produced from bamboo and solid residue by CO_2 activation utilized as CO_2 adsorbents,” *Biomass and Bioenergy*, vol. 148, May 2021, Art. no. 106039, doi: 10.1016/j.biombioe.2021.106039.

[9] Z. Deng, S. Sun, H. Li, D. Pan, R. R. Patil, Z. Guo, and I. Seok, “Modification of coconut shell-based activated carbon and purification of wastewater,” *Advanced Composites and Hybrid Materials*, vol. 4, pp. 65–73, 2021, doi: 10.1007/s42114-021-00205-4.

[10] P. Poolprasert and T. Chorchong, “Preparation of activated carbon from cassava root for cadmium removal in aqueous solution sample,” *Journal of Renewable Energy and Smart Grid Technology*, vol. 15, no. 2, pp. 26–38, Dec. 2020.

[11] W. Singsang, J. Suetrong, T. Choedsanthia, N. Srakaew, S. Jantrasee, and N. Prasoetsopha, “Properties of biodegradable poly (butylene succinate) filled with activated carbon synthesized from waste coffee grounds,” *Journal of Materials Science and Applied Energy*, vol. 10, no. 3, pp. 87–95, Aug. 2021.

[12] N. M. Musyoka, B. K. Mutuma, and N. Manyala, “Onion-derived activated carbons with enhanced surface area for improved hydrogen storage and electrochemical energy application,” *RSC Advances*, vol. 10, pp. 26928–26936, 2020, doi: 10.1039/D0RA04556J.

[13] M. Lay, A. Susli, M. K. Abdullah, Z. A. Abdul Hamid, and R. K. Shuib, “Converting dead leaf biomass into activated carbon as a potential replacement for carbon black filler in rubber composites,” *Composites Part B: Engineering*, vol. 201, Nov. 2020, Art. no. 108366, doi: 10.1016/j.compositesb.2020.108366.

[14] S. N. S. Ismail, N. N. I. N. Ibrahim, S. N. Rasli, N. A. Majid, N. M. Abdul Wahab, S. N. Jamal, S. Zakaria, and K. Nazir, “Reinforcement of charcoal activated carbon (CAC) in natural rubber (NR) compound: In comparison with carbon black,” *ASEAN Engineering Journal*, vol. 16, no. 2, pp. 161−167, Jun. 2022. doi: 10.11113/aej.v12.17224.

[15] A. Yurtay, İ. Özkan, and M. Kiliç, “Preparation and characterization of carbon/polymer composite as a novel ethylene scavenger to preserve fruit and vegetables quality in refrigerator drawer,” *Postharvest Biology and Technology*, vol. 216, Oct. 2024, Art. no. 113032, doi: 10.1016/j.postharvbio.2024.113032.

[16] A. Fazli and D. Rodrigue, “Recycling waste tires into ground tire rubber (GTR)/rubber compounds: A review,” *Journal of Composites Science*, vol. 4, no. 3, p. 103, 2020, doi: 10.3390/jcs4030103.

[17] P. Mente, M. Te, and H. Sp, “Natural rubber and reclaimed rubber composites- A systematic review,” *Polymer science*, vol. 2, 2016, doi: 10.4172/2471-9935.100015.

[18] L. Bokobza, “Elastomer nanocomposites: Effect of filler–matrix and filler–filler interactions,” *Polymers*, vol. 15, no. 13, p. 2900, 2023, doi: 10.3390/polym15132900.

[19] E. Farida, N. Bukit, E. M. Ginting, and B. F. Bukit, “The effect of carbon black composition in natural rubber compound,” *Case Studies in Thermal Engineering*, vol. 16, Dec. 2019, Art. no. 100566, doi: 10.1016/j.csite.2019.100566.

[20] C. G. Robertson and N. J. Hardman, “Nature of carbon black reinforcement of rubber: Perspective on the original polymer

nanocomposite," *Polymers*, vol. 13, no. 4, p. 538, 2021, doi: 10.3390/polym13040538.

[21] K. Katueangngan, T. Tulyapitak, A. Saetung, S. Soontaranon, and N. Nithi-uthai, "Renewable interfacial modifier for silica filled natural rubber compound," *Procedia Chemistry*, vol. 19, pp. 447–454, 2016, doi: 10.1016/j.proche.2016.03.037.

[22] P. Nuinu, C. Sirisinha, K. Suchiva, P. Daniel, and P. Phinyocheep, "Improvement of mechanical and dynamic properties of high silica filled epoxide functionalized natural rubber," *Journal of Materials Research and Technology*, vol. 24, pp. 2155–2168, 2023, doi: 10.1016/j.jmrt.2023.03.101.

[23] S. K. Palaniappan, M. K. Singh, S. M. Rangappa, and S. Siengchin, "Eco-friendly biocomposites: A step towards achieving sustainable development goals," *Applied Science and Engineering Progress*, vol. 27, no. 4, 2024, Art. no. 7373, doi: 10.14416/j.asep.2024.02.003.

[24] R. Phiri, S. M. Rangappa, S. Siengchin, and D. Marinkovic, "Agro-waste natural fiber sample preparation techniques for bio-composites development: Methodological insights," *Facta Universitatis, Series: Mechanical Engineering*, vol. 21, no. 4, pp. 631–656, 2023, doi: 10.22190/fume230905046p.

[25] A. Boonmee, P. Sabsiriroht, and K. Jarukumjorn, "Preparation and characterization of rice husk ash for using as a filler in natural rubber," *Materials Today: Proceedings*, vol. 17 pp. 2097–2103, 2019, doi: 10.1016/j.matpr.2019.06.259.

[26] N. Choophun, N. Chaiammart, K. Sukthavon, C. Veranitisagul, A. Laobuthee, A. Watthanphanit, and G. Panomsuwan, "Natural rubber composites reinforced with green silica from Rice husk: Effect of filler loading on mechanical properties," *Journal of Composites Science*, vol. 6, no. 12, p. 369, 2022, doi:10.3390/jcs6120369.

[27] C. Siriwong, S. Boopasiri, V. Jantarapibun, B. Kongsook, S. Pattanawanidchai, and P. Sae-oui, "Properties of natural rubber filled with untreated and treated spent coffee grounds," *Journal of Applied Polymer Science*, vol. 135, no. 13, 2018, Art. no. 46060, doi: 10.1002/app.46060.

[28] S. C. Peterson, "Carbon black replacement in natural rubber composites using dry-milled calcium carbonate, soy protein, and biochar," *Processes*, vol. 10, no. 1, p. 123, 2022, doi: 10.3390/pr10010123.

[29] J. Mago, A. Negi, K. K. Pant, and S. Fatima, "Development of natural rubber-bamboo biochar composites for vibration and noise control applications," *Journal of Cleaner Production*, vol. 373, Nov. 2022, Art. no. 133760, doi: 10.1016/j.jclepro.2022.133760.

[30] R. E. Eyo-Honesty and P. Evudiovo, "Development of carbon fibre reinforced natural rubber composite for vibration isolation," *IOP Conference Series: Earth and Environmental Science*, vol. 730, 2021, Art. no. 012016, doi: 10.1088/1755-1315/730/1/012016.

[31] A. A. Al-Ghamdi, O. A. Al-Hartomy, F. R. Al-Solamy, N. Dishovsky, R. Nickolov, M. Mihaylov, P. Malinova, and K. Ruskova, "Preparation and characterisation of natural rubber composites comprising hybrid fillers of activated carbon / in situ synthesised magnetite." *Journal of Rubber Research*, vol. 21, pp. 94–118, 2018, doi: 10.1007/BF03449164.

[32] H. Tan and A. I. Isayev, "Comparative study of silica-, nanoclay- and carbon black-filled EPDM rubbers," *Journal of Applied Polymer Science*, vol. 109, no. 2, pp. 767–774, 2008, doi: 10.1002/app.28130.

[33] M. H. Al-Maamori, A. A. A. Al-Zubaidi, and A. A. Subeh, "Effect of carbon black on mechanical and physical properties of acrylonitrile butadiene rubber (NBR) composite," *Academic Research International*, vol. 6, no. 2, pp. 28–37, Mar. 2015.

[34] S. Chuayjuljit, A. Imvittaya, N. Na-Ranong, and P. Potiyaraj, "Effects of particle size and amount of carbon black and calcium carbonate on curing characteristics and dynamic mechanical properties of natural rubber," *Journal of Metals, Materials and Minerals*, vol. 12, no. 1, pp. 51–57, 2022.

[35] P. Waramit, B. Krittakom, and R. Luampon, "Experimental investigation to evaluate the effective moisture diffusivity and activation energy of cassava (Manihot Esculenta) under convective drying," *Applied Science and Engineering Progress*, vol. 15, no. 4, 2022, Art. no. 5518, doi: 10.14416/j.asep.2021.10.008.

[36] A. Elmouwahidi, E. Bailón-García, A. F. Pérez-Cadenas, F. J. Maldonado-Hódar, and F. Carrasco-Marín, "Activated carbons from KOH and H_3PO_4-activation of olive residues and its application as supercapacitor electrodes," *Electrochimica Acta*, vol. 229, pp. 219–229, 2017, doi: 10.1016/j.electacta. 2017.01.152.

[37] E. Raymundo-Piñero, P. Azaïs, T. Cacciaguerra, D. Cazorla-Amorós, A. Linares-Solano, and F. Béguin, “KOH and NaOH activation mechanisms of multiwalled carbon nanotubes with different structural organization,” *Carbon*, vol. 43, no. 4, pp. 786–795, 2005, doi: 10.1016/j.carbon.2004.11.005.

[38] S. Li, X. Tan, H. Li, Y. Gao, Q. Wang, G. Li, and M. Guo, “Investigation on pore structure regulation of activated carbon derived from sargassum and its application in supercapacitor,” *Scientific Reports*, vol. 12, no. 1, 2022, Art. no. 10106, doi: 10.1038/s41598-022-14214-w.

[39] P. Yang, L. Rao, W. Zhu, L. Wang, R. Ma, F. Chen. G. Lin, and X. Hu, “Porous carbons derived from sustainable biomass via a facile one-step synthesis strategy as efficient CO_2 adsorbents,” *Industrial & Engineering Chemistry Research*, vol. 59, no. 13, pp. 6194–6201, 2020, doi: 10.1021/acs.iecr.0c00073.

[40] G. Raju, M. Khalid, M. M. Shaban, and B. Azahari, “Preparation and characterization of eco-friendly spent coffee/ENR50 biocomposite in comparison to carbon black,” *Polymers*, vol. 13, no. 16, p. 2796, 2021, doi: 10.3390/polym13162796.

[41] I. Surya and M. Siswarni, “Crosslink density and rheometric behaviour of natural rubber/chloroprene rubber blends,” *IOP Conference Series: Materials Science and Engineering*, vol. 505, 2019, Art. no. 012113, doi: 10.1088/1757-899X/505/1/012113.

[42] J. Lubura, P. Kojić, B. Ikonić, J. Pavličević, D. Govedarica, and O. Bera, “Influence of biochar and carbon black on natural rubber mixture properties,” *Polymer International*, vol. 71, no. 11, pp. 1347–1353, Nov. 2022, doi: 10.1002/pi.6439.

[43] S. Phattarateera and C. Pattamaprom, “The viscosity effect of masticated natural vs. synthetic isoprene rubber on toughening of polylactic acid,” *International Journal of Polymer Science*, vol. 2019, 2019, Art. no. 5679871, doi: 10.1155/2019/5679871.

[44] P. Mekbuntoon, S. Kongpet, W. Kaeochana, P. Luechar, P. Thongbai, A. Chingsungnoen, K. Chinnarat, S. Kaewnisai, and V. Harnchana, “The modification of activated carbon for the performance enhancement of a natural-rubber-based triboelectric nanogenerator,” *Polymers*, vol. 15, no. 23, p. 4562, 2023, doi: 10.3390/polym15234562.

[45] I. Sittitanadol, N. Srakeaw, P. Somdee, P. Chumsamrong, S. Noyming, W. Singsang, and N. Prasoetsopha, “Utilizing coconut biochar as a bio-reinforcing agent in natural rubber composites,” *Starch – Stärke*, 2024, Art. no. 2400168, doi: 10.1002/star.202400168.

[46] Ş. Bülbül and H. Ergün, “Investigation of the usability of activated carbon as a filling material in nitrile butadiene rubber/natural rubber components and modeling by regression analysis,” *Journal of Elastomers & Plastics*, vol. 56, no. 1, pp. 53–73, 2024, doi: 10.1177/00952443231215469.

[47] D. M. Manohar, S. Chandramohan, R. Natarajan, and V. Muralidharan, “Investigations on the mechanical and damping properties of styrene-butadiene rubber with graphene and carbon black,” *FME Transactions*, vol. 51, no. 3, pp. 386–395, 2023, doi: 10.5937/fme2303386C.

[48] W. B. Ribeiro, G. B. Bérti, M. Faccio, M. Godinho, and R. N. Brandalise, “Evaluation of biochar production temperature in interaction with elastomers of different polarities,” *Materials Research*, vol. 26, 2023, doi: 10.1590/1980-5373-MR-2022-0341.

[49] T. A. T. Yasim-Anuar, L. N. Yee-Foong, A. A. Lawal, M. A. A. Farid, M. Z. M. Yusuf, M. A. Hassan, and H. Arifin, “Emerging application of biochar as a renewable and superior filler in polymer composites,” *RSC Advances*, vol. 12, pp. 13938–13949, 2022, doi: 10.1039/D2RA01897G.

[50] M. Qian, B. Zou, Z. Chen, W. Huang, X. Wang, B. Tang, Q. Liu, and Y. Zhu, “The influence of filler size and crosslinking degree of polymers on mullins effect in filled NR/BR composites,” *Polymers*, vol. 13, no. 14, p. 2284, 2021, doi: 10.3390/polym13142284.

[51] D. Y. Kim, J. W. Park, D. Y. Lee, and K. H. Seo, “Correlation between the crosslink characteristics and mechanical properties of natural rubber compound via accelerators and reinforcement,” *Polymers*, vol. 12, no. 9, p. 2020, 2020, doi: 10.3390/polym12092020.