Karunanayake *et al. Biochar* (2025) 7:60
https://doi.org/10.1007/s42773-025-00429-3

Biochar

REVIEW Open Access

Role and potential of biochar as a sustainable alternative reinforcing filler to carbon black in rubber composites

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Abstract

Traditionally, carbon black (CB) has been the predominant reinforcing filler in rubber composites. This preference is due to superior mechanical properties of CB, which stem from its unique high carbon-based structure and high purity. However, the production of CB is energy-intensive, poses significant health risks, and is environmentally detrimental, releasing significant amounts of CO_2. Consequently, there is a growing interest in finding sustainable alternatives to CB. Biochar (BC), another carbon-rich solid produced through the pyrolysis of biomass under limited oxygen, continues to be studied as a promising, eco-friendly, and low-cost reinforcement filler for rubber composites. BC exhibits high surface area, stability, and carbon content, making it a contender for enhancing the mechanical properties of rubber. Various biomass materials, including rice husks, corncobs, nutshells, grain husks, and palm kernel shells, have been used to produce BC, with its reinforcement potential influenced by production conditions and feedstock type. However, the variation in the feedstock and production protocol has been found to produce BC with different compositional and morphological features which affect the properties of the final vulcanizate in a detrimental manner. Therefore, it is essential to understand the governing factors that optimize the performance of BC in rubber composites. While BC is increasingly viewed as a promising replacement for CB as a reinforcement filler, a detailed evaluation of its reinforcement capabilities remains lacking. This review explores the viability of BC as an alternative to CB or a partial replacement CB, highlighting its environmental benefits and effectiveness in rubber composite applications. Rheological and mechanical properties of BC-incorporated rubber composites never reached the standards of CB. However, a mixture of BC and CB, replacing CB content partially showed promising results. Therefore, further studies are required to find optimum properties for BC to incorporate into the rubber matrix to replace CB.

Keywords Biochar, Composites, Rubber, Rheological properties, Mechanical properties, Carbon black

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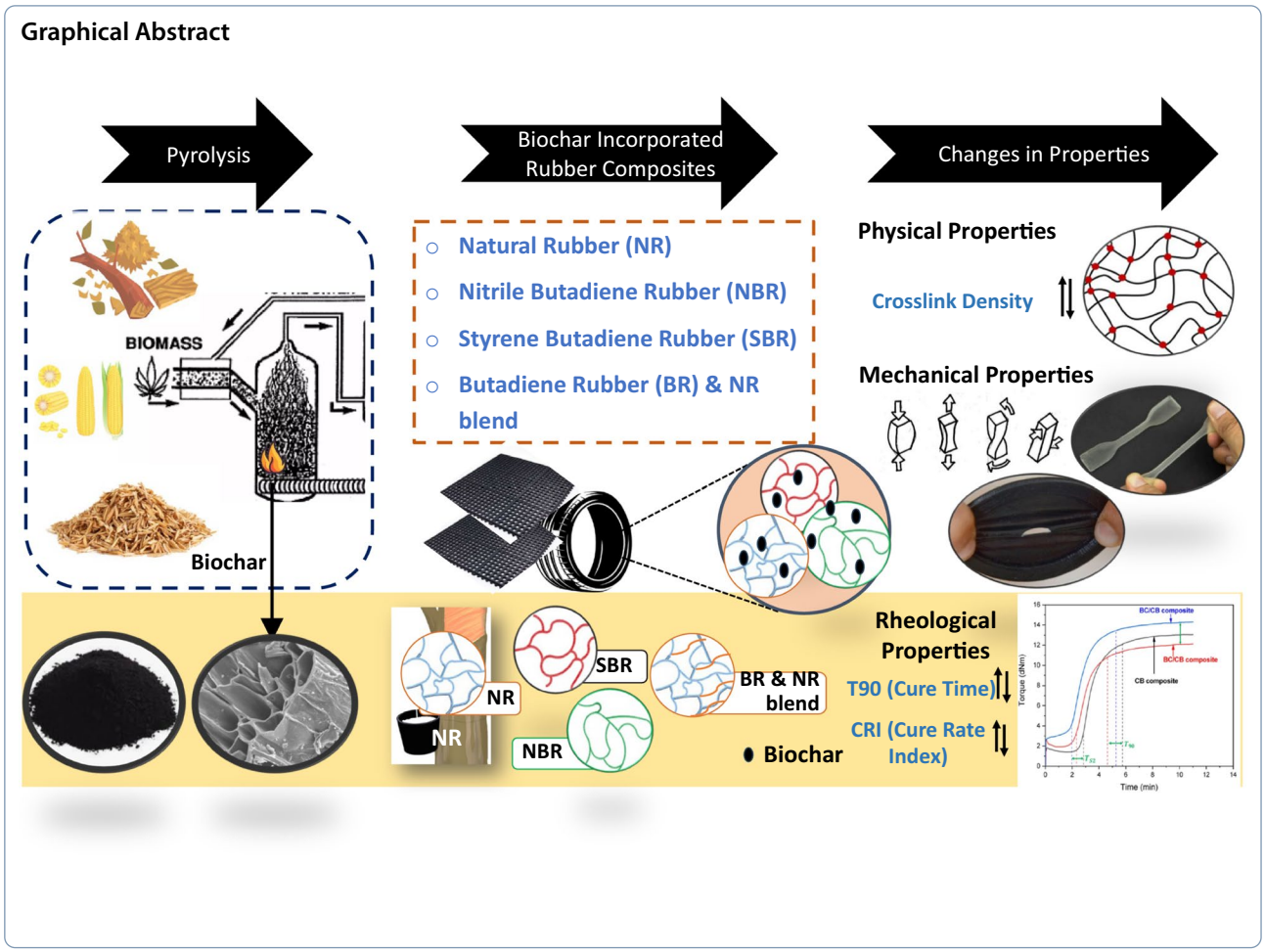

1 Introduction

The significance of rubber to society has progressively increased over the past century. Rubber is ubiquitously used in a wide range of products, including tires, household and other automobile items, as well as in the healthcare sector and medical devices. Additionally, rubber finds applications in various industrial settings, such as in conveyor belts and gaskets. There are different types of rubber, including natural rubber, neoprene, styrene-butadiene rubber (SBR), nitrile butadiene rubber (NBR), butadiene rubber (BR), and silicone, each with its unique properties (Akshay et al. 2021). Fillers are an important part of a rubber product formulation. Reinforcement fillers are additives used in polymers that are essential to improve mechanical properties, wear resistance, thermal stability, and chemical resistance (Yadav et al. 2023). There are two different types of fillers used in rubber product manufacturing: reinforcing fillers and non-reinforcing fillers. While the former is used to improve the properties of the product, the latter is mainly used to reduce the cost (Dieu et al. 2023). An effective reinforcement filler enhances the mechanical properties of rubber (Yadav et al. 2023).

As a reinforcing filler in rubber composites, carbon black (CB) has long been the primary and standard choice due to its surface reactivity, homogeneity, wear resistance, and high purity (Robertson and Hardman 2021). CB shows the multinuclear structure in the form of a spherical shape (graphite-like structure) (Ribeiro et al. 2023). Usually, CB is synthesized above 1600 °C temperature in the decomposition of petroleum fuel in an oxidative atmosphere. CB is mainly produced from tar, coal, and acetylene like non-renewable sources (Koriem et al. 2023). Meanwhile, CO_2 and some carcinogenic polycyclic hydrocarbons are released to the air as byproducts, during this production process. According to the reported results, 2.4 tons of CO_2 are released per 1.0 ton of CB production (Fan et al. 2020). As a result, there is a growing interest in substitute eco-friendly and cost-effective alternative reinforcement fillers in rubber composites for CB due to high carbon footprint.

The effectiveness of fillers obtained from cellulose, chitosan, lignin, and soy protein as replacement fillers during the rubber composite manufacturing process has been evaluated (Jiang et al. 2020; Peterson 2022). Most of these bio-fillers are currently used to prepare the "green tire" with reducing the usage of fossil fuel-based CB. Hence, while looking for substitute material for CB, attention is given to biochar (BC) as an alternative reinforcement filler in rubber composites. BC is a popular carbon-based solid derived from biomass under slow pyrolysis with the presence of limited oxygen (Lubura et al. 2022). The pyrolysis process converts organic compounds into a stable carbon form (Devi et al. 2021; Igliński et al. 2023). BC has obtained precise attention because of its distinctive aspects such as higher surface area, stability, high carbon content, and cation exchange capacity (Wang and Wang 2019). The reinforcement ability of BC mainly depends on the porosity of the surface, which depends on the different production conditions such as feedstock type, pyrolysis temperature, etc. Peterson (2022). BC has shown the potential to be an ideal reinforcing filler; however, it remains a considerable challenge due to its inherent drawbacks such as substantial particle size, limited surface activity compared to activated carbon (Xu et al. 2020), hydrophilicity (Koriem et al. 2023), impurities (Abidin et al. 2023), variable constituents and varying properties depending on the feedstock and production conditions. Due to the variable properties of BC, rubber composites experience weakening, leading to fractures and localized stress, and resulting in a low phase transition enthalpy value in the rubber (Koriem et al. 2023). Milling BC particles with silica-like hard substance material showed 9% improvement in vulcanizate tear strength with unmilled sample (Peterson and Kim 2020). Therefore, a substantial modification of BC is required, both in size and surface properties, to enhance the practical applications. Researchers have tested chemical and physical modifications such as coupling agent addition, milling, and hydrolysis to make BC a better reinforcement material by improving the functional groups, surface area, and pore structure (Bardha et al. 2023; He et al. 2022; Peterson and Thomas 2022). In addition, a higher percentage of carbon is essential for an appropriate filler to be a substitute for CB, which contains more than 95% content of carbon (Long et al. 2013). Relatively low carbon content, low homogeneity and higher ash quantity are major drawbacks of biomass feedstock (Wang et al. 2018).

Although BC can be prepared in different pathways to obtain some properties close to some properties of CB, there found to be gaps between other properties of BC and CB. Additionally, some graphitized BC can be used as a substitute for CB due to similarities in properties (Manyà, 2012). Despite extensive studies on BC as an alternative to CB for rubber reinforcement, a comprehensive review is still lacking. Only a single review has been published on preparation of biochar to use in rubber composites (Bélanger et al. 2023). The review contains data on biochar prepared with various sources such as animal hair, pit, shells, different types of wood sources. Similarly, the properties of composites of the biochar produced with the different sources with different types of rubbers such as NR, SBR and NBR have been discussed. However, there is no analysis to show the importance of BC properties in its capacity to be used as a rubber composite. Additionally, there is a lack of clarity on which BC feedstock and production conditions yield the best outcomes, and the optimal mixing percentages to achieve the best results have not been comprehensively summarized. Hence, this review aims to discuss the literature to ascertain the feasibility and efficacy of BC as a reinforcing filler in rubber, presenting a comprehensive review of its properties, mechanisms, and potential applications. Furthermore, this review will enquire into the experimental approaches used to prepare rubber-BC composites and characterize their mechanical properties. Key parameters such as rheological properties, tensile strength, tear resistance, and abrasion resistance will be evaluated to assess the suitability of BC as a reinforcing filler in rubber formulations. This work aims to fill the knowledge gap and provide a detailed understanding of the optimal conditions and formulations for utilizing BC in rubber reinforcement.

2 Bibliometric analysis on biochar in natural rubber as a reinforcement filler

2.1 Search criteria, data source, and method of analysis

Scopus database was utilized to conduct a search using the terms "biochar" AND "natural rubber". The initial selection of studies was based on their titles, keywords, and abstracts. As of July 2024, this search yielded 20 papers recorded in the Scopus database, which were subsequently employed for the analysis. The analysis of the co-occurrence of countries involved in biochar research related to natural rubber was performed using VOSviewer software (version 1.6.20). This analysis focused on mapping the collaboration networks, with a minimum threshold set at two studies per country to be included in the co-occurrence mapping. Furthermore, to explore the application of biochar in natural rubber (NR) for enhancing reinforcement within the composite, an additional search was conducted in the Scopus database using the terms "Biochar" AND "reinforcement" AND "natural rubber". The resulting data were refined

and categorized by year, country, and author to create detailed maps that illustrate the distribution and concentration of research efforts in this specific area up until July, 2024.

2.2 Actively performed countries

The affiliations of the authors of collected research articles were from 34 countries. When the minimum number of reported studies in a country was 2, there were 26 thresholds for the co-authorship within 34 countries. Figure 1 shows the network among the top 25 productive countries involved in the research on biochar-incorporated rubber composites. The highest number of studies (92) has been performed in China while the United States (46) and India (42) had second and third places, respectively (Fig. 1). Excluding them, only another three countries: Thailand, Malaysia, Germany have been involved in more than 20 studies on the incorporation of biochar into rubber composites.

2.3 Reinforcing fillers in rubber Composites

The analysis of the keywords from the previously mentioned 20 studies reveals a common theme: the incorporation of filler materials into rubber composites to enhance reinforcement. Predominantly, these filler materials include silica, carbon nanotubes, nano clay, and lignin, with the notable exception of carbon black. Beyond reinforcement, these materials are also frequently utilized to modify other mechanical properties and curing characteristics, such as crosslinking density. Upon examining Figure. 2, it becomes evident that, up until 2024, the application of biochar as a reinforcing filler in rubber composites has been minimally explored. However, a slight increase in interest can be observed in the use of lignin-derived biochar as a reinforcing filler within rubber materials. This emerging trend is supported by specific data points: in cluster 9, there are 6 links, a total link strength of 11, with 4 occurrences, and an average publication year of 2021.

2.4 Impact on biochar as a reinforcing filler in rubber composite

Building upon the insights gained from the second search conducted within the Scopus database, six pertinent studies were identified and are detailed in Figure. S1. These studies predominantly focus on the enhancement of reinforcement in rubber composites through the innovative use of various lignin feedstocks as fillers, serving as alternatives to the traditionally used carbon black. This approach reflects a growing interest in exploring sustainable materials for improving the mechanical properties of rubber composites.

The analysis reveals a notable instance of biochar derived from pyrolysis being referenced within the Scopus database, albeit with a singular occurrence and total link strength of 2. This instance highlights the emerging yet still relatively unexplored potential of biochar as a reinforcing filler material in rubber composites. The limited but significant presence of biochar in the academic discourse underscores the necessity for further research into its applications and efficacy as a sustainable reinforcing agent. The data point towards a growing interest in the material, suggesting that biochar, particularly from lignin sources, holds promise as a viable alternative to conventional fillers, justifying deeper exploration and understanding within the field.

3 Biochar utilization in rubber composites

Evaluating BC as a replacement for CB in rubber reinforcement requires addressing several key factors: the type of biochar, the type of rubber used as the polymer matrix, the preparation method of the biochar-rubber composite, and the type of cross-linking system employed (Fig. 3). Covalent bonding involves the formation of strong chemical bonds between the biochar surface and the rubber matrix, enhancing durability and strength. Physical bonding, on the other hand, relies on van der Waals forces and mechanical interlocking, contributing to improved dispersion and compatibility. Crosslinking bonding involves the creation of a three-dimensional network where biochar particles act as crosslinking agents, increasing the elasticity and mechanical properties of composite. These mechanisms collectively enhance the performance of rubber composites reinforced with biochar.

3.1 Biochar vs. carbon black for reinforcement of rubber

As the demand for sustainable materials intensifies, biochar has gained attention as a promising alternative to carbon black in rubber composites. The effectiveness of carbon black as a reinforcing agent is largely tied to its high carbon content, minimal ash impurities, and ultra-fine particle size (Jiang et al. 2020; Lay 2020; Peterson et.al. 2015). However, key factors like its structural arrangement, surface chemistry, and expansive surface area also significantly influence its interactions within the rubber matrix (Balasooriya et.al. 2019; Peterson et.al. 2015). Given that certain critical properties, such as surface chemistry and particle size, of BC can be tailored through controlled processes, the versatility of biochar becomes even more promising. By carefully selecting the feedstock—whether wood, agricultural residues, animal-derived materials, or municipal waste—and optimizing pyrolysis conditions such as temperature, heating rate, and residence time, biochar can potentially be engineered

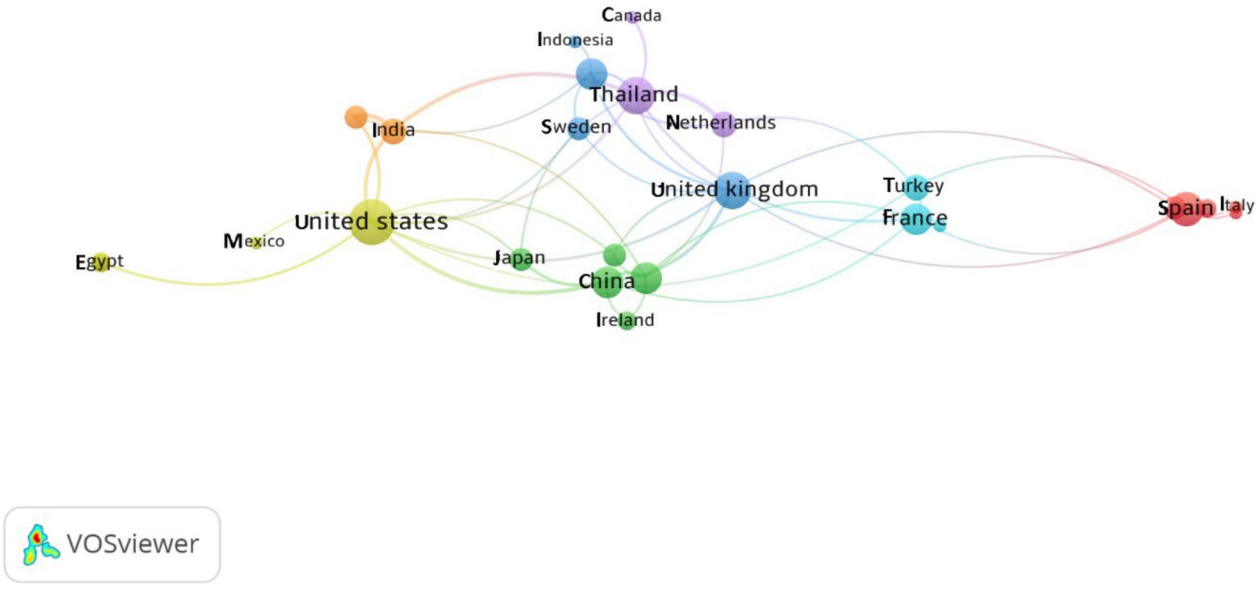

Fig. 1 A network map of the top countries that have published biochar-incorporated rubber composites research up to July, 2024

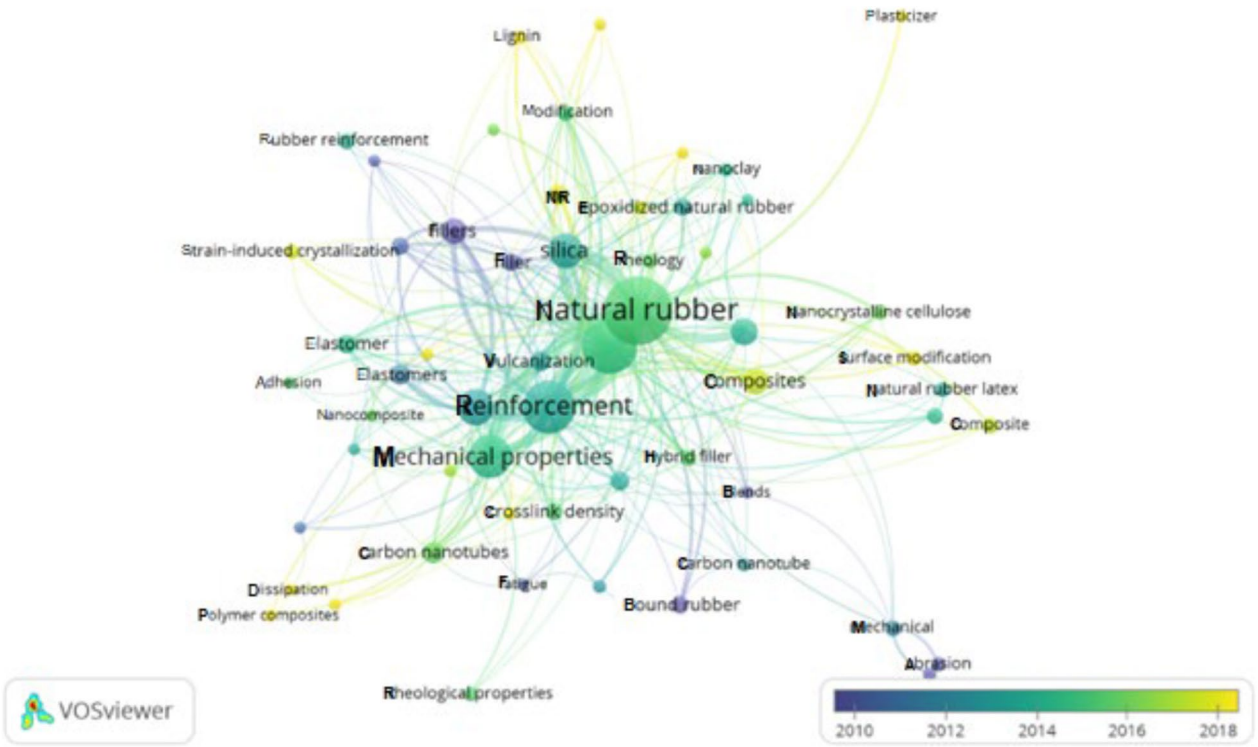

Fig. 2 A map of research areas biochar in rubber composites up to the July 2024

to mimic the desirable physicochemical characteristics of carbon black. This adaptability allows for the creation of biochars with properties that align more closely with carbon black, potentially enhancing their performance as reinforcing agents in rubber composites. With these controlled modifications, biochar could bridge the gap between sustainable material alternatives and the stringent performance requirements of modern engineering

applications, positioning it as a viable substitute for carbon black in diverse industries.

The inherent properties of carbon black can, therefore, be compared to those of biochar to assess its applicability and performance as replacement filler in rubber composites. CB has been the preferred reinforcing filler in the production of dry rubber articles due to its reliable and consistent performance (Robertson 2021). CB is characterized by a complex blend of amorphous and crystalline carbon with turbostratic stacking. The different grades of CB are classified according to the ASTM D1765 system ("Standard Classification System for Carbon Blacks Used in Rubber Product"), where the first digit denotes the particle size range, and the remaining digits indicate specific performance characteristics (Costa et.al. 2022; Fan et.al. 2019; Robertson 2021; Thonglhueng et.al. 2022). A summary of the carbon black grades commonly used for rubber reinforcement is given in Table 1.

The reinforcing mechanism of carbon black in rubber composites is primarily determined by its particle size, surface area, and interaction with the rubber matrix. Smaller particle sizes, like those in the N100 and N200 grades, provide a larger surface area, enhancing interactions between carbon black and rubber polymer chains (Bélanger et.al. 2022). This occurs through physical adsorption, where rubber chains adhere to the carbon black surface via Van der Waals forces (Bélanger et.al. 2022; Kumar 2021). Additionally, physical entanglement

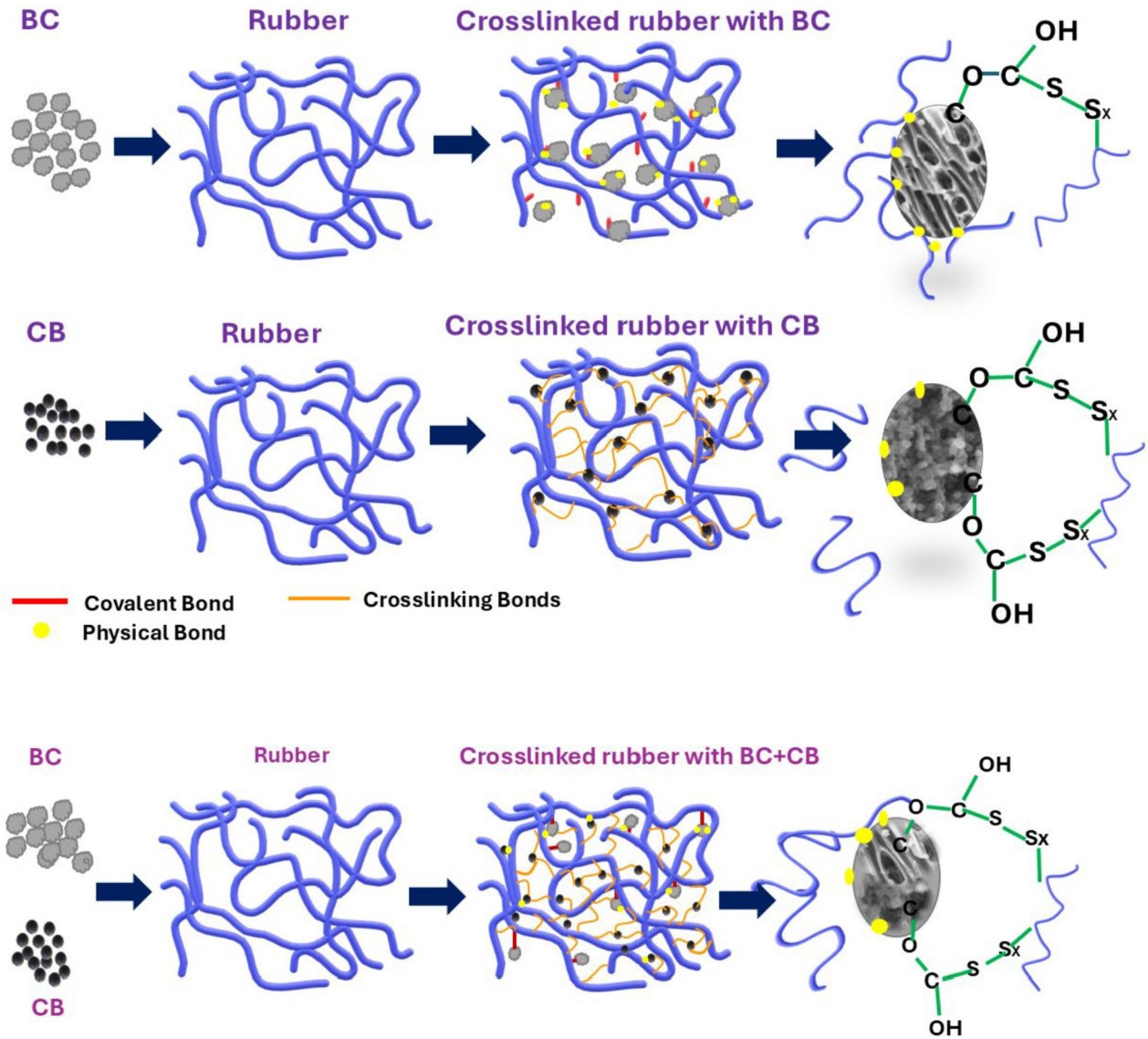

Fig. 3 Mechanisms of interaction between biochar and carbon black to enhance rubber composite properties

forms a network that distributes applied stress across the composite (Bélanger et.al. 2022). In some cases, chemical bonding can occur, where functional groups on the carbon black surface form covalent or hydrogen bonds with the rubber, strengthening the interface (Robertson 2021). These interactions improve stress transfer from the rubber matrix to the filler. The carbon black particles create an interconnected filler network, boosting load-bearing capacity and stiffness, essential for high-performance applications.

The hydrodynamic effect caused by carbon black increases the effective volume of the rubber composite, restricting the mobility of rubber chains and enhancing stiffness (Bélanger et.al. 2022). In natural rubber, carbon black also acts as a nucleating agent for strain-induced crystallization, further improving strength and tear resistance (Utrera-Barrios 2021). Larger grades, like N500 and N600, though less reinforcing, reduce heat build-up due to their lower surface area, improving flexibility and reducing hysteresis (Spahr 2016; Utrera-Barrios 2021). Given that biochar is derived from renewable sources, it presents a promising sustainable alternative to carbon black as a reinforcing filler in rubber composites. However, its effectiveness depends heavily on its preparation, structural properties, and the resulting impact on rubber performance. Unlike carbon black, which is produced through the controlled pyrolysis of hydrocarbons, biochar is created from organic materials such as agricultural residues or wood (Jiang et.al. 2022). This fundamental difference in feedstock and production conditions influences key characteristics like surface area, particle size, and surface chemistry, all of which affect its reinforcing potential in rubber matrices.

Biochar generally has larger particles and a lower surface area compared to carbon black, especially grades like N100 and N200, which are designed to have fine particles with high surface areas that maximize bonding with rubber polymer chains (Bélanger et.al. 2022; Greenough et.al. 2021). The larger, more variable particle size and reduced surface area of biochar limit its ability to reinforce rubber effectively, as there is less physical adsorption and entanglement with the rubber matrix, leading to reduced stress transfer and mechanical reinforcement (Bélanger et.al. 2022; Chang et.al. 2021). The evolving molecular structure of biochar derived from lignocellulosic feedstock, within a temperature range of 100 °C to 700 °C was thoroughly studied by Keiluweit (Kiuluweit et.al. 2009). They identified five distinct structural phases of biochar during thermal treatment: (a) unmodified feedstock; (b) transitional biochar; (c) amorphous biochar; (d) composite biochar; and (e) turbostratic biochar. Their findings revealed that at lower temperatures, the feedstock remains largely unchanged, with its original amorphous lignin, crystalline cellulose, and amorphous hemicellulose structures intact during the early stages of pyrolysis. In terms of surface chemistry, biochar contains various functional groups, including carboxyl, phenolic, and carbonyl groups, due to the incomplete pyrolysis of organic material (He et al. 2022). While these groups can interact with rubber via hydrogen bonding or acid–base interactions, they do not provide the same level of consistent bonding as well-defined surface chemistry of carbon black, affecting the overall reinforcement quality (Bélanger et al. 2022). The porous and irregular structure of biochar also limits its interaction with the rubber matrix (Bélanger et al. 2022; He et al. 2022). This porosity leads to less effective physical entanglement of rubber chains compared to the compact particles of carbon black, and the uneven structure may cause poor dispersion in the rubber, further reducing its reinforcing

Table 1 Characteristics of carbon black grades commonly used for rubber reinforcement

Grade	Particle size (nm)	Surface area (m^2g^{-1})	Reinforcement level	Key properties & applications
N100	20–25	130–150	Super high reinforcement	Excellent tensile strength, abrasion resistance; high hysteresis; used in heavy-duty tires
N200	25–35	110–130	High reinforcement	High tensile strength, improved cut growth; balanced heat dissipation; used in tire treads and conveyor belts
N300	35–50	70–90	Medium reinforcement	Good balance of reinforcement and processability; moderate heat build-up; used in passenger tires and mechanical goods
N500	50–75	40–50	Semi-reinforcing	Moderate stiffness, improved flexibility and heat management; used in sidewalls and hoses
N600	70–90	35–45	General-purpose	Lower reinforcement, better flexibility and processability; used in tire inner components and profiles
N700	90–150	25–30	Non-reinforcing	Minimal reinforcement, excellent elasticity; used where mechanical strength is not essential, such as seals and gaskets

capabilities (Sekar et al. 2023). The larger particle size and lower surface area of biochar result in weaker stress transfer, limiting its ability to enhance tensile strength and abrasion resistance compared to carbon black (Bélanger et al. 2022). Although biochar may improve certain mechanical properties like stiffness, it generally provides lower reinforcement, which may be suitable for applications that do not require high performance.

The lower reinforcement of biochar also affects the dynamic properties of rubber composites, offering reduced resilience and fatigue resistance compared to carbon black (B´elanger et al. 2024). This may limit its use in applications that require high performance under cyclic loading, such as tires. However, the lower surface area and irregular structure of biochar result in less heat build-up, which can reduce hysteresis and improve flexibility (Lubura et.al. 2022). Unlike carbon black, biochar does not promote strain-induced crystallization in rubber as effectively. The irregular, porous structure of biochar prevents the formation of crystalline regions under strain, leading to less improvement in strength and tear resistance.

3.2 Types of biochar used in rubber composites

Several types of feedstocks such as citrus tree stems (Koriem et al. 2023), wood-derived biomass (Lubura et al. 2022), corn cob, and stover (Belanger et al. 2022), pinus (Ribeiro et al. 2023), rice husks (Bardha et al. 2023), silver maple landscape wood waste (Peterson and Kim 2020), palm kernel shell (Abbas et al. 2019), spent coffee ground (Raju et al. 2021), coconut shell (Fan et al. 2017), and pinewood (Naghdi et al. 2017) have been used to prepare BC for rubber composite research. Further, pyrolysis of the feedstocks has been conducted in varying temperature ranges (400 and 900 °C). Biochars derived from lignin-based materials have shown vesicle-like primary nanoparticles which were closely packed to form "high-structure" irregular fragments with a high specific surface area (83.41 m^2g^{-1}) in biochar/styrene-butadiene rubber composites (Jiang et al. 2020). There are studies reported on rubber composites produced from different types of biochar obtained from copied hard wood (Peterson 2012, 2020; Peterson and Kim 2020; Peterson and McMahan 2023; Peterson and Thomas 2022). At a 10% total filler concentration (w/w), composites incorporating 25% or 50% biochar exhibited enhanced tensile strength, elongation, and toughness compared to those filled with CB (Peterson 2012). This highlights the potential of renewable biochar as a partial substitute for CB in flexible, low-filler rubber composite applications. Biochar produced with silver maple landscape wood waste and co-milled with 1wt% nano silica was able to replace 40% of the carbon black filler in a styrene–butadiene rubber composite with virtually no loss in tensile strength. Also, elongation and toughness properties of the optimal biochar substituted composites were improved by over 31% and 24%, respectively (Peterson and Kim 2020). A natural rubber composites with 30% total filler, half of the CB was fully replaced with silica-milled Paulownia-based biochar with slight loss (<6%) of tensile strength, and comparable elongation and toughness compared to the 100% CB-filled control composite (Peterson 2019). Biochars made from coppiced hardwoods, *Paulownia elongata* and *Populus tremuloides* were incorporated to partially replace carbon black in rubber composites made of 50/50 blends of butadiene rubber and natural rubber. Two biochar types were used to replace 30% of the carbon black with virtually no loss in tensile strength, and improved elongation and toughness compared to the sample with carbon black (Peterson 2020). Silver maple, coppiced hardwoods, *Paulownia elongata* and *Populus tremuloides* studies have used biochar from proprietary methods, and it is hard to relate with the production temperature or any biochar properties.

Dry-milled $CaCO_3$ and soy protein, Paulownia hardwood-based biochar, and carbon black were used in various ratios, in NR composites while replacing 40–50% of the carbon black (Peterson 2022). The composites produced had essentially the same tensile strength, with better toughness and elongation properties relative to the carbon black control. The poplar wood biochar that was first treated with carbon dioxide and then coated with lauric acid when incorporated in a SBR composite showed a 19% increase in tensile strength and a 48% increase in toughness (Peterson and Thomas 2022). Guayule composites with up to 60% of the carbon black replaced with two biochar types obtained from *Populus canadensis* (Poplar) and *Paulownia elongata* were reported to have higher tensile strength, elongation, and toughness compared to the 100% CB filled composite (Peterson and McMahan 2023). However, the carbon contents of *Paulownia elongata* (PAUL), *Populus tremuloides* (POP), and *Populus canadensis* (Poplar) materials used have been reported as 87%, 87%, and 95%, respectively. These values are still low compared to 99% in carbon black. Nevertheless, BC produced was reported to contain characteristic graphitic d-spacing peaks inherent to carbon black, which could be the reason for the comparable results obtained at least in partial replacement of CB (Peterson 2019, 2020; Peterson and Thomas 2022). These biochars are likely derived from high-temperature processes, contributing to their desirable properties.

Structure of biochar revealed that it has embedded silica spheres that were originated from natural structure of rice husk. It has been reported that the chemical structure of RH biochar contained various chemical structures

such as C=C, C=O, C–C, and Si–O-C. The presence of Si–O-C bond indicates that not only silica particles were embedded in the biochar, but also they chemically bonded to form cross-link structures. These cross-link structures can improve reinforcing effect of NR composite (Xue et al. 2019). Palm kernel shell biochar has been used in producing carboxylated nitrile butadiene rubber composites (Abidin et al. 2023b). The aromatization, formation of large number of pores, cavities, and cracks on the surface of the biochar has been observed during the biochar formation process of the palm kernel shell (PKSBc) biochar, compared to CB particles that are generally regularly shaped and more rounded. A higher loading ratio of PKSBc in CB/PKSBc was found to reduce T_{90} and increase the CRI of rubber composites. The incorporation of PKSBc helped to provide high abrasion resistance to the XNBR matrix (Abidin et al. 2023b).

BC obtained from hydrothermal carbonization treatment of hardwood waste biomass has been tested in NR composites to replace CB (Lubura et al. 2022). The presence of a macroporous structure in BC has been observed compared to smaller particles of CB fillers. The CB as a filler might potentially be replaced with BC up to 30 phr, while achieving similar rubber vulcanization and mechanical properties (Lubura et al. 2022). Spent ground coffee-based biochar has been incorporated into an epoxidized natural rubber composite to compare properties with carbon black-based similar composites (Raju et al. 2021). Incorporation of 10 phr of spent coffee biochar could improve the composites tensile properties and thermal performance compared to carbon black. Nevertheless, the addition of biochar significantly affects the maximum torque compared to CB and delays the vulcanization time (Raju et al. 2021). Corn cob, corn stover and the highly refined cornstarch feedstocks have been pyrolyzed to obtain three different types of biochar samples for use in comparison with carbon black (Belanger et al. 2022). The biochar particles were found to be agglomerated after ball milling. It is reported that the biochar has a highly porous structure and the micropore areas compose 94.6% and 96.8% of the total BET surface areas for the stover-based and starch-based biochar samples, respectively in SBR composites. Starch biochar SBR composites were highly crosslinked and hence, significantly brittle (Belanger et al. 2022).

Carboxylated styrene–butadiene rubber composites filled with 10 (wt.%) corn starch or a 3:1 blend of corn starch:BC produced better reinforcement, tensile strength, elongation, and toughness compared to the corresponding CB-filled control sample (Peterson 2011). PKSBc in NR composites were produced using three different initial PKS particle sizes as feedstock, i.e., 300, 250 and 100 μm. The PKS size did not show significant relationship to tensile properties and hardness. However, the lowest PKS size produced the lowest value in tear strength suggesting a relationship between the ash content and the tear strength (Abbas et al. 2019). Meanwhile, Pinus BC prepared at 900 °C showed higher volatile matter content and a low ash content appeared to be the optimum temperature to obtain the shortest vulcanization time (Ribeiro et al. 2023).

Biochar produced pyrolyzing pruning stem of citrus trees were used in nitrile butadiene rubber composites (Koriem et al. 2023). Biochar obtained were used as unmodified and stearic acid modified form. The water contact angle values of unmodified and modified biochar (MBi) produced are reported as 121.6° and 132.4°, respectively. This reduced wettability increases the compatibility of modified biochar with the organic polymer. MBi-filled composites did not show increase in t90. Incorporation of MBi did not adversely affect the electrical conductivity while the permittivity was increased due to the interfacial polarization between the MBi and the rubber phase (Koriem et al. 2023). Biochar produced from two different low-ash nutshell feedstocks (hazelnut (HNS) and walnut (WNS)) and two high-silica grain husk feedstocks (oat (OH) and rice (RH)) were used in preparation of SBR rubber composites (Bardha et al. 2023). The nutshell-based BC provides high reinforcement properties compared to that of carbon black. In addition, slurry-based activation of biochar further improves mechanical properties of the produced composite. A low ash content biomass produce BC that improves reinforcement performance (Bardha et al. 2023).

3.3 Method of preparation of biochar used in rubber composites

There is a plethora of studies that examined the effect of different types of biochar (BC) and modified biochar (MBC) preparation techniques on the properties of BC-incorporated rubber composites (Fig. 3). While studying the BC preparation method, researchers have given major attention to reaction conditions such as pyrolysis temperature, residence time, ramping rate, inert gas type, and flow rate (Table 2). Hydrothermal carbonization and slow pyrolysis are the BC preparation methods used for the preparation of biochar used in reinforcement of rubber composites (Lubura et al. 2022). Higher temperatures and increased heating rates in slow pyrolysis enhanced the reactivity of BC in rubber composites (Aboughaly et al. 2023; Greenough et al. 2021a). Flow of N_2 during pyrolysis created micron size pores in BC, improving surface area of stover at 700 °C (Belanger et al. 2022).

The carbon material produced by the pyrolysis of Pinus spp. was used in NR and NBR composites to evaluate

the effect of processing parameters (Ribeiro et al. 2023; Ribeiro et al. 2023a). It was illustrated that the shortest pyrolyzing time (30 min) at high pyrolyzing temperature (900 °C), which is beyond biochar production temperatures, generated turbostratic structure which has similarities to carbon black structurally. It has been reported that the excess heating time at high temperatures removes volatile matter and during the process structure collapses, producing large pores in the structure and reducing the reinforcing ability of carbon produced. As the residence time increased, crystalline structure interfered with the crystallization process of the natural rubber and a decrease in tensile strength and the interaction between the load- elastomer, and an increase in the vulcanization time of the rubber have been observed (Ribeiro et al. 2023). The pH of BC is directly proportional to the pyrolysis temperature and acidic pHs showed a prolonged vulcanization due to the adsorption of basic accelerators whereas the shortest vulcanization time was observed at alkaline pH (Ribeiro et al. 2023).

Hydrothermal carbonization, a pyrolysis process in the presence of subcritical water, is considered as an effective method of converting biomass into carbonaceous materials (hydrochar-HC). This HC has gained attention as a promising filler material in organic polymers due to its specific properties and chemical reactivity (Lubura et al. 2023; Sekar et al. 2023). Co-fillers, HC-CB obtained by hydrothermal carbonization of hardwood sawdust and commercial CB exhibited a strong anti-oxidizing effect and greatly stabilized the rubber composite against oxidative crosslinking (Lubura et al. 2023). A study by Sekar et al. (2023) tested silane-modified lignin HC as a filler for SSBR/BR rubber compounds and identified as a semi reinforcing behavior in comparison with those of the unmodified version. Therefore, the incorporation of HC into rubber fillers would enhance the stability, reduce the rate of oxidative crosslinking and improve the overall durability of the materials.

Both physical and chemical-based modified methods were used to prepare the modified biochar (MBC) for use as an effective filler (Table 3). In some cases, BCs were physically modified by ball and roll milling (Peterson 2012; Peterson and McMahan 2023; Raju et al. 2021). The recorded data showed that ball milling was an efficient physical method to decrease the average particle size from 15.03 µm to 5.81 µm (Xue et al. 2019). Greater than 10 µm particle size can weaken rubber composites due to localized stress, hence milling plays a positive role due to homogeneous particle size (Peterson 2022). The presence of micron-sized pores and the high surface area in biochar (BC) can significantly improve reinforcement in rubber composites. Additionally, ball milling introduced oxygen-containing functional groups on particle surfaces by generating active carbon radicals, which increased the surface energy and enhanced interaction with the rubber matrix (Ramanayaka et al. 2020). Certain studies showed that the round per minute (RPM) parameter of the milling process can influence agglomeration of BC particles which should be avoided (Xue et al. 2019). In some studies, milling was carried out with popular additives such as silica and nano-silica (Jong 2020; Peterson and Kim 2020). Silica-milled BC particles are very much smaller.

Table 2 Production conditions of common BC used in rubber composites

Type of feedstock	Pyrolysis temperature (°C)	Residence time (min)	Inert gas & flow rate (mL min^{-1})	Ramping temperature (°C min^{-1})	Particle size (µm)	References
Stem of citrus trees	450	60	Nitrogen	10	88	(Koriem et al. 2023)
Wood-derived waste	215	165			500–800	(Lubura et al. 2022)
Corn cob and corn stover	700	60	Nitrogen	50	425	(Belanger et al. 2022)
Pinus	900	30, 60, 240	Nitrogen & 200			(Ribeiro et al. 2023)
Pinus	900, 400	30	Nitrogen & 200			(Ribeiro et al. 2023)
Rice husks and nut shells	700	60	Nitrogen & 500	20		(Bardha et al. 2023)
Palm kernal shell (PKS)	500	360			300, 250, 100	(Abbas et al. 2019)
Spent coffee ground	500	30				(Raju et al. 2021)
Woody waste biomass	650	360				(Peterson 2012)
Coconut shell	400 & 800 (under temperature gradient)	0–120	Nitrogen & 100–1000	5–15 with 10 rpm		(Fan et al. 2017)
Pinewood	525 ± 1		Nitrogen			(Naghdi et al. 2017)

More than 10% (wt) of silica mixing in BC was detrimental to the toughness and strength properties of the final yield. About 1% silica content is the optimal mixing quantity to obtain MBC (Peterson 2022; Peterson and Joshee 2018). Meanwhile, nano-silica (NS) as a co-milling agent with BC produced a very small size of BC particles. The co-milling of BC with 1% (wt) NS for 60 min produced a significant alteration in the distribution of sizes in BC particles. The stress-related fracture junctions with poor filler matrix bonding by these NS particles (Peterson and Kim 2020).

Chemical modifications have been common in reinforced rubber composites (Jiang et al. 2020; Koriem et al. 2023; Raju et al. 2021). Inorganic and organic chemicals such as stearic acid (Peterson and Thomas 2022), calcium carbonate (Jong 2020), sodium chloride (Meng et al. 2013), ether compounds (Raju et al. 2021), and zinc oxide (Li et al. 2014) were used to synthesize chemically modified BC (CMBC) for rubber composites. CMBC, prepared with citrus biochar and varying ratios of stearic acid, exhibited a significant increase in hydrogen bonds between CMBC hydroxyl groups and natural rubber carboxyl groups rather than relying solely on reinforcement effect of biochar (Koriem et al. 2023). The lauric acid-saturated BC showed 19% increase of tensile strength and a more hydrophobic surface area indicating a potentially valuable enhancement for reinforced rubber (Peterson and Thomas 2022). Furthermore, the pH of the BC has fluctuated due to the addition of metal oxides and alkali metals (Pongdong et al. 2015). A lignin-based biochar–silica hybrid nanoparticles were incorporated into NR to obtain a composite (He et al. 2022). The nanoparticles with core shell structures were prepared using lignin black liquor extracted from xylose residue, a major component in lignin, and sodium silicate as raw materials. The reinforcement performance of the LBS hybrid nano particles carbonized at 500 °C produced a "high structure" characteristics material comparable to that of commercial carbon black (CB) N550 (He et al. 2022).

The oxidized BC-filled composites exhibited 10% and 20% filler fractions for tensile strength and decreased the crosslinking density of oxidized BC (Jong 2016). Hence, it is essential to limit the oxidation of functional groups, as it introduces polar groups such as carboxyl, hydroxyl, and carbonyl, which decrease the hydrophobicity of biochar and reduce its compatibility with the typically non-polar rubber matrix (Fig. 4). This incompatibility can lead to poorer dispersion and weaker interfacial bonding, ultimately diminishing the mechanical properties and reinforcement effectiveness of the biochar in the rubber composite. Composites incorporating 4% Bael shell BC in an epoxy matrix exhibited a 183% increase in tensile strength compared to neat epoxy, while also significantly enhancing thermal stability (Minugu et al. 2021). High cohesion reinforcement, low-temperature softening, polarity, and weaker interfacial bonding are significant disadvantages found in modified biochar (BC) in rubber composites (Liu et al. 2008). However, these drawbacks are outweighed by the benefits of modified biochar as a reinforcing filler. Therefore, a careful assessment of the best feedstock, production conditions, and modifications in biochar is necessary before incorporating it as a reinforcement filler in rubber.

Conversion of biochar into hybrid materials exhibits synergistic effects with enhanced physio-chemical properties (He et al. 2022; Jiang et al. 2022; Qian et al. 2021). A dual-phase carbon–silica nanohybrid was fabricated from black liquor lignin and sodium silicate through a simple co-gelation/self-assembly and carbonization process and it could achieve tensile strength of 17.92 ± 0.87 MPa and elongation at break of $708 \pm 10\%$. Both properties were superior to those of CB-filled SBR compounds (Jiang et al. 2022). The hydrophobic surface of the hybrid nanoparticles prepared by a combination of two-step acid precipitation and carbonization of lignin had good compatibility with rubber molecules (He et al. 2022). The high specific surface area and large number of pores of these hybrid nanoparticles greatly increase the contact area between the fillers and the rubber matrix enabling rubber chains to be strongly inserted into the filler network (He et al. 2022). Dielectric properties of NR incorporated rice husk BC-silica composites reached 7.62 at 10^3 Hz, which is 2.37 times that of pure NR. Therefore, these hybrid filler materials can act as good reinforcing fillers with enhanced mechanical properties.

3.4 Reinforcing effect of different types of biochar

There are mainly three types of biochar produced from three different biomass sources. That is wood based, straw or grass based, and different types of industrial waste material. The difference in biomass source produces differences in compositions of the biochar produced and hence, the properties of rubber composites produced incorporating biochar obtained from different biomass sources vary from each other significantly. Furthermore, composition also changes with pyrolysis techniques, especially the pyrolysis temperature and the time. In addition, the morphology of the biochar changes with the rate of heating.

The carbon content of wood-based, straw- or grass-based, and different types of industrial waste material are in the ranges of 61–92, 44–90, and 8–57 (%wt.), respectively (Bardha et al. 2023; Greenough et al. 2021b). Although, the fixed carbon content in wood- and plant-based materials are in a similar range, in waste materials

Table 3 Preparation techniques of modified biochar used in rubber composites

Type of modification	Type of feedstock	Involved chemicals/method	Pyrolysis temperature (°C)	Residence time/Time of preparation (min)	Inert gas and Flow rate (mL min^{-1})	Ramping temperature (°C min^{-1})	Particle size (μm)	References
	The pruned stem of citrus trees	Stearic acid dissolved in toluene (2%) as an organic solvent medium	450	60		10		(Koriem et al. 2023)
	Lignin powder	Chemically purified with an alkaline solution (pH > 12.5) and sulfuric acid (5 wt%) for precipitation of lignin	800	120	Nitrogen & 100	10	2–5	(Jiang et al. 2020)
	Spent coffee ground derivative	Epoxide (Cyclic ether compound)	500	30				(Raju et al. 2021)
	Woody waste biomass	Stearic acid						(Peterson 2012)
	Soy protein	Calcium hydroxide at 2.2% (pH = 10), calcium carbonate, Stearic acid, zinc oxide, Silica coated with silane coupling agent, and antioxidant					80–200	(Jong 2020)
Chemical	*Paulownia elongate* and Soy protein (20:80, 40:60, 60:40, and 80:20)	Stearic acid-coated calcium carbonate (SA-CC)						(Peterson 2022)
	Corn flour	Sodium hydroxide (pH ~ 10.5), antioxidant (2,2′-Methylenebis(6-tert-butyl-4-methylphenol)), sulfur, stearic acid, and zinc oxide						(Jong 2016)
	Rice bran	Sulfur 1.5phr, zinc oxide 5phr, stearic acid1.5phr, *n*-cyclohexyl-2-benzothiazole sulfonamide (CBS) 2 phr and di-benzothiazole disulfide (DD) 1phr	900				200	(Li et al. 2014)

Table 3 (continued)

Type of modification	Type of feedstock	Involved chemicals/ method	Pyrolysis temperature (°C)	Residence time/Time of preparation (min)	Inert gas and Flow rate (mL min^{-1})	Ramping temperature (°C min^{-1})	Particle size (µm)	References
	Bamboo	Sodium chloride (10 g) and the flocculating agent (6 mL) were dissolved in distilled water (350 mL), and the pH of the solution was determined at 3–3.5 through the addition of sulfuric acid						(Meng et al. 2013)
	Rice husk ash	Formic acid (94% purity) &hydrogen peroxide (50 wt%)						(Pongdong et al. 2015)
	Lignin	Ball milling		240				(Jiang et al. 2020)
	Silver Maple wood waste	Ball milling		60			≤ 44	(Peterson and Kim 2020)
	Woody waste biomass	Ball milling	650	360				(Peterson 2012)
Physical	*Paulownia elongata* and *Populus tremuloides* (Coppiced BC)	Ball milling		60			10–100	(Peterson and McMahan 2023)
	Paulownia elongate & Soy protein (20:80, 40:60, 60:40, and 80:20)	Ball milling						(Peterson 2022)
	The pruned stem of citrus trees	Ball milling	450	120		10	< 88	(Koriem et al. 2023)
	Spent coffee ground derivative	Roll milling	500	30				(Raju et al. 2021)
	P. *elongata* and P. *tremuloides* biochar with silica	Ball milling		60				(Peterson and Joshee 2018)
	Corn flour	Ball milling	80 (mixing temperature)	20				(Jong 2016)
	Pinewood	Ball milling		180–502.2 (serious of different temperatures)				(Naghdi et al. 2017)

it is quite low except for some materials like lignin liquid (He et al. 2022). The changes in the carbon content can also be observed with the change of heating rate and the time (Almutairi et al. 2023; Domingues et al. 2017; Sheng and Yang 2012). One of the main features of CB is its high carbon content and its structural arrangement that produces the reinforcing effect on the rubber matrix. And hence, it is obvious that BC with low carbon content is not suitable replacement for CB in rubber products. Notably, Pinus spp. pyrolyzed at 900 °C for 240 min has produced a BC with 93 (% wt.) fixed carbon content. Nonetheless, the biochar produced resulted in the graphitic crystallinity which decreases the mechanical properties of the composite and increases in the optimum vulcanization time (Ribeiro et al. 2023a). Lower residence time (30 min @ 900 °C) during the pyrolysis process produced a BC with a lower fixed carbon content and turbostratic structure, resulting in better properties.

However, N, S, and O contents of waste materials-based BC indicate much higher value indicating the increased presence of functional groups and/or defects in the graphite-like layers (Bélanger et al. 2023; Bardha et al. 2023; Greenough et al. 2021b). Increase in functional groups can increase the molecular interactions between the BC and the rubber matrix albeit the defects in graphite-like structure may reduce the strength impart. In fact, this is apparent in many composites where BC is used to completely replace CB in rubber composite (Bélanger et al. 2023).

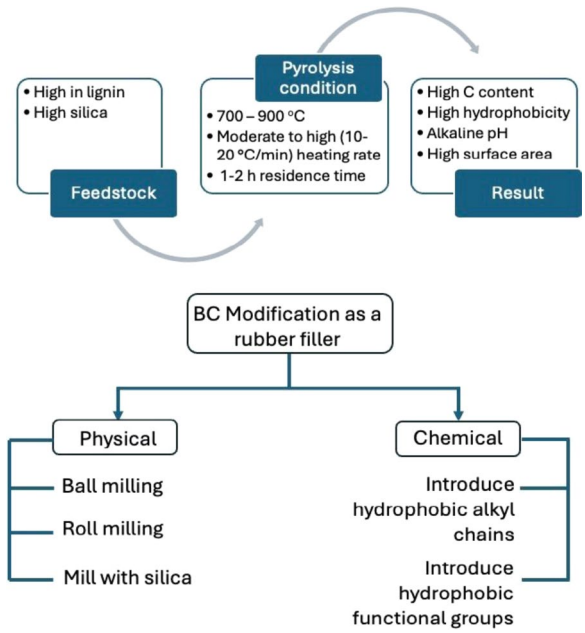

Fig. 4 Summary of best biochar production and modification conditions for its utilization in rubber composites

Further, the surface area of BC produced seems to be mainly dependent on the pyrolysis temperature and the effect of the biomass used is negligible (Bardha et al. 2023; Domingues et al. 2017). The surface area per unit mass increases with the increase of the temperature. Surface area plays a significant role in the filler to rubber interaction. It is a fact that smaller the CB particle size, higher the reinforcement imparts to the composite (Chollakup et al. 2021). However, the increase of surface area per unit mass resulted in formation of cellular morphology while compact graphite-like structures of CB may not produce the expected reinforcement to the rubber composite. CB particles act like stress concentration points in a rubber composite (Liang et al. 2021) whilst porous fragile BC particles with high surface to mass ratio may not be able to behave in the same way (He et al. 2022). Nevertheless, highly porous BC obtained from palm kernel shell imparted high abbreviation resistance and hardness to the carboxylated nitrile butadiene rubber (XNBR) composite (Abidin et al. 2023a) and it could be due to the high polarity of the rubber matrix.

3.5 Method of incorporating BC to rubber matrix and type of rubber.

Surprisingly, studies on the use of biochar in rubber composites have been limited to only four different types of rubber and their derivatives: natural rubber, nitrile butadiene rubber, styrene butadiene rubber, and butadiene rubber. It shows that only a limited attention is given to composites of BC with different rubber types. In addition, there are three additional studies reported related NR. First, an attempt to incorporate BC into epoxidized NR (Raju et al. 2021). Secondly, a comparison of BC based composites made with NR and NBR (Ribeiro et al. 2023). The latter two publications have attempted to check the effect of polarity on the prosperities of rubber composites. Then, a BC composite of NR/BR blend (Peterson 2020) and a BC composite of a polyisoprene obtained from guayule (Peterson and McMahan 2023). Similarly, several reported studies are on BC blends with SBR (B´elanger, et al. 2023; Bardha et al. 2023; Peterson and Kim 2020; Peterson & Thomas 2022). There are two more studies done on incorporating BC to NBR (Koriem et al. 2023) and carboxylated nitrile butadiene rubber (XNBR) (Abidin et al. 2023b). It seems that there is plenty of space for further research in the BC and rubber composites.

Incorporation of BC and MBC has been mainly done in dry rubber stage except in one instance BC was incorporated in latex stage (Peterson 2011). Two main techniques are used in incorporation of BC to dry rubber using two standard techniques with different types of two roll mills and laboratory internal mixtures. Most of the NR-based

compounds were prepared with laboratory type internal mixtures (He et al. 2022; Lubura et al. 2022; Peterson 2019; Ribeiro et al. 2023; Xue et al. 2019). In addition, a BC composite based on the isoprene rubber obtained from Guayule rubber and a NR/BR blend have also been prepared using a laboratory type internal mixture (Peterson 2020; Peterson and McMahan 2023). ENR and BC composite (Raju et al. 2021) has been compounded with a two-roll mill. NR/NBR blend-based BC composites have been prepared with initial mixing in an internal mixture and the second stage in a two-roll mill as two polymers have differences in polarity that need use of different mixing techniques (Ribeiro et al. 2023). Finally, a standard NR form SMR L(standard Malaysian rubber light)-based based BC compound has also been prepared using a two-roll mill for compounding (Abbas et al. 2019). It is observed that both close and open milling techniques could be successfully used in incorporating BC into NR and NR-based composites.

Preparation of SBR-based BC composites has been carried out using both laboratory internal mixtures (Bardha et al. 2023; Peterson 2012; Peterson and Kim 2020) and two-roll mills (Bélanger, et al. 2023; Jiang et al. 2020). However, one of the first SBR-based BC composites reported by Peterson has been produced using a wet blending technique. BC dispersion was mixed with SBR latex and freeze-drying was used to obtain the final dry compound (Peterson 2011). There is one more study where the authors do not specify the method of incorporating BC to SBR rubber although it seems likely that a dry blending technique is used (Peterson & Thomas 2022). Incorporation of BC to both XNBR (Abidin et al. 2023b) and NBR (Koriem et al. 2023) was done using a two-roll mills.

While most of the reported BC composites in literature were investigated to find the suitability of BC as a renewable reinforcing filler to replace CB completely, in some cases it has been used as a partial replacement for CB (Abidin et al. 2023b; Peterson 2019, 2020). In most cases, the BC was added to the rubber first and the vulcanizing ingredients were added to the mixture secondly (Peterson and Kim 2020; Raju et al. 2021; Ribeiro et al. 2023; Ribeiro et al. 2023).

4 Properties of biochar rubber composites

4.1 Physical properties

The potential of BC as a substitute for CB in various rubber matrices has been extensively studied by recent studies (Peterson 2019, 2020, 2022; Peterson and Kim 2020; Peterson and McMahan 2023; Peterson and Thomas 2022). The physical properties, with particular attention to their impact on density and crosslinking density, have been meticulously documented and are presented in Table S1. The biochar-added samples exhibited slightly lower densities compared to the CB control, with Paulownia biochar at 1.76 g cm^{-3} and Populus biochar at 1.66 g cm^{-3}, while maintaining comparable tensile strength and displaying greater elongation and toughness properties (Peterson 2020). Guayule rubber composites with biochar from hybrid poplar and *Paulownia elongata*, were reported to have densities of 1.68 g cm^{-3} and 1.76 g cm^{-3}, respectively, slightly lower than CB-controlled samples (Peterson and McMahan 2023). Porous structure and lightweight nature of BC contribute to the overall reduction in the density when used as a filler material. A decrease in crosslink density occurs with an increase in biochar to carbon black ratio, correlating with trends in tensile strength. However, when biochar (BC) was mixed with nanosilica during milling in styrene–butadiene rubber composites, the composite with silver maple biochar exhibited a density of 1.67 g cm^{-3}, comparable to CB-controlled samples (Peterson and Kim 2020). This reduction in density is attributed to the more amorphous nature of biochar, in contrast to the highly structured carbon black. These findings underscore the importance of considering material properties and structure when selecting fillers for rubber composites, regardless of the type of rubber matrix studied.

4.2 Rheological properties

The diverse array of studies on the rheological properties of composite materials provides invaluable insights into their potential applications and benefits. The impact of biochar produced at two distinct temperatures, 400 °C and 900 °C, on elastomers revealed that biochar produced at 900 °C significantly influenced vulcanization time (Table 3). This high-temperature biochar, due to its alkaline nature, resulted in shorter vulcanization times for both natural rubber (NR) and nitrile butadiene rubber (NBR) compounds (Ribeiro et al. 2023). BC interacted well with elastomers, particularly NBR, due to higher oxygen concentration. Despite low surface area, BC-reinforced blends exhibited enhanced tensile strength. Rheologically, BC-filled compounds had lower minimum torque, indicating a more uniform filler structure and lower viscosity compared to carbon black. BC produced at 400 °C showed higher crosslink density differences than BC produced at 900 °C (Table 4). Compounds with BC had shorter vulcanization times, except for those with acidic BC at 400 °C, attributed to filler pH influencing accelerator adsorption (Ribeiro et al. 2023). This pH variation aligned with BC pyrolysis temperature, corroborating elastomer vulcanization time data. Moreover, the same group investigated the influence of

different residence times on the rheological properties of elastomeric compounds (Ribeiro et al. 2023). They found that shorter residence times (30 min) produced BC with a beneficial graphitic structure, enhancing interaction with elastomers and maintaining mechanical properties. Conversely, longer residence times led to a more crystalline structure, reducing tensile strength and interaction with elastomers while increasing vulcanization time. Rheological analysis revealed that BC-filled blends had lower viscosity and less aggregation compared to carbon black-filled blends, making them easier to process and more cost-effective. Moreover, similar crosslink densities were observed regardless of residence time, and compounds with BC had shorter vulcanization times, indicating improved efficiency in production processes (Ribeiro et al. 2023).

The addition of modified BC (MBi) in NBR composites did not significantly increase the optimum vulcanization time (t_{90}) of the composites, which aligns with the aim of achieving sustainability and environmental friendliness by utilizing BC as filler (Koriem et al. 2023). The observed reduction in ML values for MBi-modified compounds compared to unfilled BC incorporated NBR indicates improved processability, attributed to a less irregular final structure of the filler within the compound. Additionally, similar M_H values between MBi-filled and carbon black (CB)-filled composites suggest a comparable density of crosslinks within the elastomer matrix. Furthermore, MBi-modified elastomers exhibited lower t_{90} values compared to unfilled NBR, enabling shorter processing times. The reduction in curing time was attributed to the pH of the filler, with acidic pH increasing vulcanization time due to adsorption of curing agents, while basic pH shortened it. This explanation is supported by the influence of pyrolysis temperature on the pH of the BC, with higher temperatures yielding alkaline BC and lower temperatures yielding acidic BC. Similar results have been reported in the literature confirming the impact of filler pH on vulcanization time (Downie & Munroe, 2009; Weber and Quicker 2018).

Palm kernel shell biochar (PKSBc) was used as a sustainable filler in rubber composites, and it was observed that an increase in the PKSBc ratio in the hybrid fillers led to a decrease in delta torque (M_H-M_L) values, indicating reduced crosslink density and modulus, and a decrease in scorch time (T_{S2}) and curing time (T_{90}), suggesting accelerated vulcanization (Abidin et al. 2023b). The presence of hydroxyl groups on PKSBc enhanced filler–filler interactions, increasing the curing rate index (CRI) and crosslinking density. Another finding underscored BC as a sustainable substitute for CB in rubber formulations, particularly up to a filler content of 30 phr, where comparable vulcanization behavior was observed (Lubura et al. 2022). Notably, BC-containing formulations exhibited lower torque values during vulcanization, indicative of slower curing kinetics and delayed onset of crosslinking compared to CB-filled counterparts. Furthermore, BC did not significantly affect cure time (T_{90}) or degradation kinetics, although mechanical properties were influenced by the choice of filler. However, mixtures containing BC exhibit lower torque values compared to CB-filled mixtures, suggesting a slower vulcanization process with BC. Moreover, vulcanization starts later in BC-containing mixtures compared to CB-filled ones, indicating a delay in the onset of crosslinking. Further it confirms that BC exhibits a lower curing rate compared to CB, as evidenced by the degradation rate after the peak, which is significantly lower for BC-containing mixtures. The maximum and minimum torque values increase with higher filler content for both BC and CB, but these values are lower for mixtures containing BC particles. Scorch time (T_{S2}) generally decreases with increasing filler content, while the time to reach a certain torque increase is higher for BC-containing mixtures. Interestingly, the study finds that filler content has no significant influence on the cure time (T_{90}) of rubber samples, and the curing rate index (CRI) is only slightly affected by the type of filler used. However, BC has no discernible influence on vulcanization and degradation kinetic parameters, while the choice of filler does affect mechanical properties.

The scorch times of the composites synthesized by various BCs from different feedstocks are comparable across the composites, with minor variations indicating polymer–filler interactions prior to vulcanization (Bardha et al. 2023). During the crosslinking phase, the slope of the vulcanization section reflects the dispersion and cure state of the composites. Activation of BC considerably increases the slope for certain composites, indicating faster vulcanization, better dispersion, and less filler–filler interaction. A general trend is observed where T_{S2} and T_{90} decrease with activation, indicating improved vulcanization kinetics. However, exceptions have been observed, such as minimal change in T_{S2} for certain composites and increased T_{90} for others. The cure rate index (CRI) generally increases with activation, except for certain composites, indicating faster cure times for activated composites compared to their unactivated counterparts. The marching modulus index (MMI) provides insights into filler compatibility and interaction with the rubber matrix. Activation leads to decreased MMI for all BC composites, indicating improved dispersion and reduced filler–filler interaction. The final storage modulus of the cured composites reflects their stiffness and elasticity, with activated BC composites demonstrating comparable values to CB-filled composites, validating the potential of BC as a filler alternative (Bardha et al.

Table 4 Rheological properties of CB and BC added rubber composites

Filler	CB replacement	Tensile strength (MPa)	Elongation at break (%)	Toughness (MPa)	Young's Modulus (MPa)	Tear strength (N mm^{-1})	References
Unfilled NR	0%	11.9 ± 1.2	626 ± 9	19.4 ± 1.6	1.2 ± 0.1		(Peterson 2019)
100% CB	0%	25.7 ± 0.6	476 ± 8	51.7 ± 2.3	3.9 ± 0.4		
100%PB	100%	15.3 ± 1.6	522 ± 21	32.2 ± 4.7	1.9 ± 0.3		
100%PB Silica 0.5%	100%	18.9 ± 0.7	516 ± 10	39.3 ± 2.2	2.2 ± 0.3		
100%PB Silica 1%	100%	19.3 ± 0.9	519 ± 8	40.1 ± 2.3	2.2 ± 0.3		
100%PB Silica 2%	100%	17.3 ± 1.2	469 ± 20	32.4 ± 3.9	2.3 ± 0.4		
75/25% PB/CB Silica 1%	75%	21 ± 0.9	524 ± 11	43.9 ± 2.6	2.2 ± 0.2		
50/50 PB/CB Silica 1	50%	24.3 ± 0.4	527 ± 12	52.3 ± 1.4	2.8 ± 0.3		
UNMILLED / NR	100%	14.4 ± 0.9	1373 ± 48				(Xue et al. 2019)
DM-400/NR	100%	17.6 ± 1.0	1421 ± 41				
DM-500/NR	100%	19.3 ± 1.3	1487 ± 61				
DM-600/NR	100%	18.6 ± 0.5	1440 ± 24				
EM-400/NR	100%	20.7 ± 0.6	1317 ± 50				
EM-500/NR	100%	21.3 ± 0.7	1268 ± 45				
EM-600/NR	100%	19.6 ± 0.6	1383 ± 22				
WM-400/NR	100%	20.4 ± 1.1	1248 ± 68				
WM-500/NR	100%	18.7 ± 1.0	1386 ± 41				
WM-600/NR	100%	17.4 ± 1.3	1253 ± 27				
CB/NR	0%	18.3 ± 0.7	1225 ± 25				
50CB/NR		21.97	923			49.99	He et al. (2022)
$50L_MB_{400}$ S/NR		10.56	1502			15.12	
$50L_MB_{500}$ S/NR		23.01	1415			48.94	
$50L_MB_{600}$ S/NR		21.96	1594			41.77	
$50L_MB_{700}$ S/NR		21.89	1539			36.14	
$50L_LB_{500}$ S/NR		19.24	1462			42.17	
$50L_HB_{500}$ S/NR		14.69	517			33.41	
NR/CB	0%					31.4 ± 3.8	Ribeiro et al. (2023)
NR/BIO400	100%					28.5 ± 3.8	
NR/BIO900	100%					26.1 ± 2.8	
NR/CB	0%	15.8 ± 1.7	1301 ± 66.7			30.6 ± 3.8	Ribeiro et al. (2023)
NR/BIO30 min	100%	12.7 ± 0.9	1537 ± 89.1			25.2 ± 2.8	
NR/BIO60 min	100%	5.4 ± 0.8	534.8 ± 28.9			22.5 ± 2.4	
NR/BIO240min	100%	598 ± 0.2	570.7 ± 11.5			21.01 ± 1.4	

NR Natural rubber, *CB* Carbon Black, *PB* Paulownia Biochar, *DM* Dry milled, *EM* Ethanol medium Milled, *WM* water medium milled, L_M lignin black liquor, L_MS lignin black liquor mixed silica, L_MB lignin black liquor biochar, $L_MB_{400}/L_MB_{500}/L_MB_{600}/L_MB_{700}$: pyrolysis temperature respectively 400 °C/ 500 °C/ 600 °C/ 700 °C, L_L lignin black liquor biochar with 125 g Xylose, L_H Lignin black liquor biochar with 500 g Xylose, BIO 30/BIO 60/BIO 90/BIO 240: Pyrolysis time at 900 °C

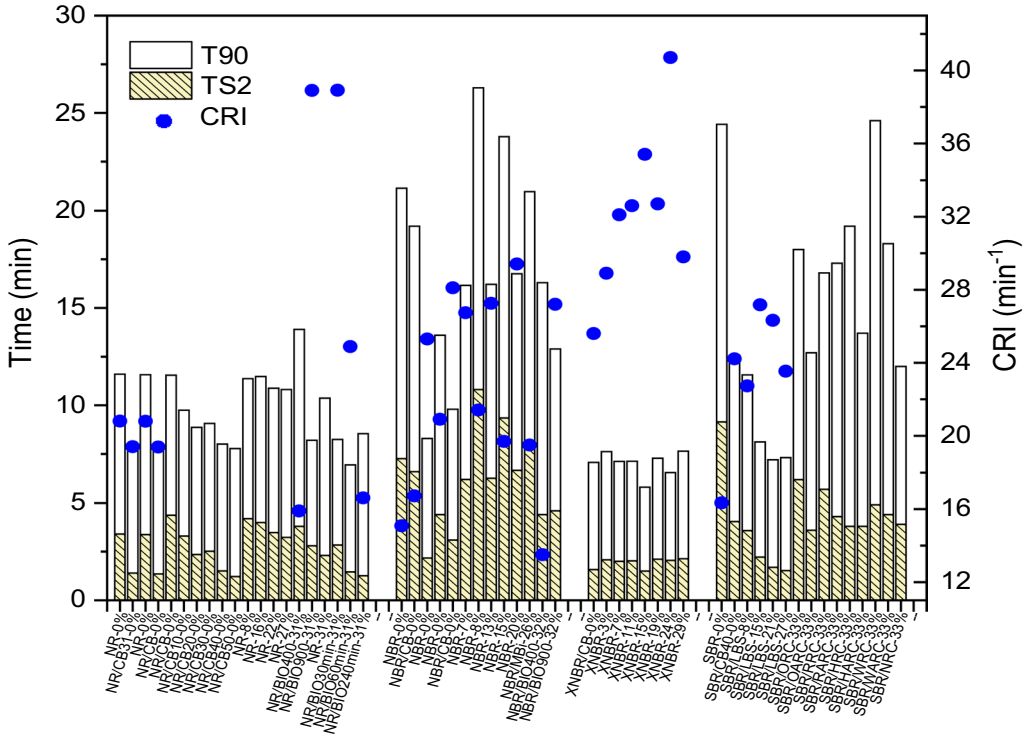

Fig. 5 Comparison of tS2, t90 and cure rate index for the four different elastomer types

2023). Figure 5, shows a comparison of T_{S2}, T_{90} and cure rate index for the four different elastomer types for which BC was incorporated. Sulphur cross-link systems were used in all the composites discussed. The highest CRI range was recorded for XNBR showing that it is a specific to the polymer and the curing system used. It confirms with the fact that XNBR has the lowest range for T_{S2} and T_{90} showing the rapid rate of vulcanization. Whilst other polymers, NR, NBR and SBR, do not show any significant deviations with each other, they have some significant changes within the composites. Furthermore, NBR and SBR compounds have shown high T_{S2} and T_{90} values compared to NR and XNBR compounds.

In NR composites, CRI index is slightly reduced compared to NR compound in the CB incorporated samples. Similar trends can be seen in T_{S2} and T_{90} results. Nevertheless, BC containing composites have reported high CRI values exceeding values reported for CB, except for two samples that contain 31% (w/w) BC. These two samples have been produced with different BC manufacturing processes. However, T_{S2} and T_{90} values do not change significantly, and they are in the range of those of CB based NR composites. In NBR composites, three samples that contain 8%, 15% and 26% (w/w) BC content have shown significantly high T_{S2} and T_{90} values together with low CRI values. This indicates the possibility of using them in large products such as agricultural and industrial vehicle tyres with a slow curing process. T_{S2} and T_{90} values are reduced with the increase of lignin-based BC percentage in SBR composites compared to very high values shown in the control sample. A relative increase in CRI values could also be observed. Relatively high T_{S2} and T_{90} values could be observed for all other SBR composites.

Figure 6 contains ML, M_H and ΔM values for NR, NBR, XNBR and SBR compounds. Like the results seen in the Fig. 6, ML, M_H and ΔM values are very consistent in XNBR compounds compared to other three compounds, except for the composite that has 13% (w/w) BC in it. In the 13% composite, all the values for M_L, M_H and ΔM are quite low. In all other compounds, high M_H and ΔM with a low M_L could be observed. It could be because XNBR has a relatively low inherent viscosity.

Increase in M_L, M_H and ΔM can be observed for two NR/CB composites compared to NR itself owing to increase imparted by viscosity increase resulting from CB addition. It is a steady increase with the increase of percentage CB content. A similar increase could be observed for the incorporation of BC from 8% (w/w) to 27% (w/w). However, remarkable changes in rheological properties can be observed with different types of BC produced with different protocols. The highest values which are comparable to that of BC 31% (w/w) are shown in the sample that was pyrolyzed at 900 °C for 30 min. All three parameters are high in the composite with BC produced

at 400 °C albeit they are lower than the values of BC 31% (w/w) composite produced at 900 °C for 30 min discussed earlier. It could be the fact that the second couples of samples have slightly higher BC percentage, i.e. 32% (w/w). This shows that the fine tuning of pyrolysis protocol is a must.

All NBR and SBR-based compounds have relatively lower M_L, M_H and ΔM values. All five BC composites including the modified BC (MBi) with varying levels of compositions show very low values for M_L, M_H and ΔM. Although, lignin-based BC incorporated SBR composites show lower values, they are comparable to their CB counterpart and LBS 27% (w/w) composite shows properties almost like those of CB SBR composite. Lignin-based BC is a good environmental friendly and sustainable contender for replacing CB in rubber compounds, especially in tire industries where large quantities are used.

By observing the rheological properties of styrene-butadiene rubber (SBR) composites filled with lignin-derived BC (LB), it was found both M_L and M_H increase with the loading of LB, indicating the addition of rigid fillers and the enhancement of the total network structure in the composites (Jiang et al. 2020). The crosslinking density followed a similar trend, suggesting a higher degree of chemical crosslinking in LB-filled composites. The dynamic modulus (DM) of LB-filled composites initially increases with LB loading up to 30 phr, then slightly decreases at 40 phr. This behavior is attributed to the physical adsorption of LB on vulcanizing agents and SBR molecules, hindering the formation of chemical crosslinking networks. The scorch time (T_{S2}) and optimum vulcanization time (T_{90}) decrease significantly with increasing LB loading, indicating accelerated vulcanization behavior due to active groups present in LB. The curing rate index (CRI) is higher for LB-filled composites compared to pure SBR, indicating faster vulcanization facilitated by the active groups in LB. However, at higher LB loadings, the CRI decreases due to the adsorption of vulcanizing agents by LB particles, slowing down the vulcanization rate. Notably, the vulcanization behavior of LB-filled composites, particularly at 40 phr loading, resembles that of CB-filled composites in terms of M_L, M_H, and CRI, but exhibits significantly shorter T_{s2} and T_{90}, implying higher production efficiency.

The collective findings of the above reported studies underscore the transformative potential of BC-based fillers in enhancing the rheological properties of elastomeric composite materials. BC, derived from various feedstocks and produced at different temperatures, demonstrates remarkable versatility and efficacy in improving

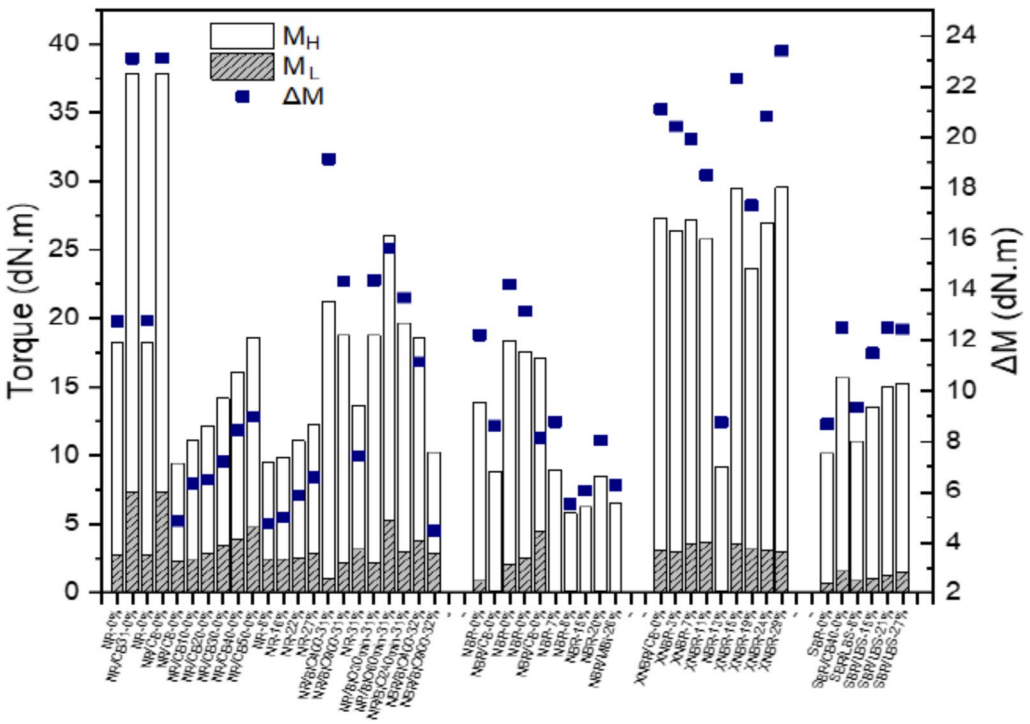

Fig. 6 ML, MH and ΔM values for NR, NBR, XNBR and SBR compounds

processability, reducing vulcanization times, and enhancing mechanical properties. Despite variations in BC production methods and feedstock sources, the studies consistently reveal the ability of BC to interact favorably with elastomers, resulting in more uniform filler structures and lower viscosity compared to traditional carbon black fillers. Furthermore, the investigations into modified BC and hybrid BC fillers highlight the potential for tailored properties to meet specific application needs while advancing sustainability goals.

4.3 Mechanical properties

In the assessment of mechanical properties of BC incorporated rubber composites, tensile strength, elongation at break, toughness, modulus, hardness, and tear strength were the key parameters studied in the literature. These properties provide a comprehensive understanding of the performance and suitability of composites for various applications. Only one study was found incorporating BC into epoxidized natural rubber (ENR). In other studies; (1) BC added to natural rubber (NR) composites, (He et al. 2022; Peterson 2019; Ribeiro et al. 2023; Ribeiro et al. 2023b; Xue et al. 2019) (2) BC added to nitrile rubber (NBR) composites (Koriem et al. 2023; Ribeiro et al., 2023) (3) BC added to styrene-butadiene rubber (SBR) composites, (Peterson 2011) and (4) BC added to blend of butadiene rubber-natural rubber (BR-NR) composites (Peterson 2020). By systematically examining the variations, the effects of biochar incorporation across different rubber matrices are explained, providing valuable insights for material scientists and engineers in optimizing composite formulations for enhanced mechanical performance.

4.3.1 Biochar-incorporated natural rubber composites

The enhancement of mechanical properties of natural rubber composites by incorporating BC from different sources is summarized in the Table 5. While the tensile strength and toughness of biochar-filled composites are generally lower than those of CB-filled controls, certain silica concentrations (0.5% and 1%) can improve elongation behavior (Peterson 2019). Notably, composites replacing 50% of CB with 1% silica-milled biochar nearly match the tensile strength of the CB control and surpass it in terms of elongation and toughness, suggesting the potential of BC and silica blends in applications requiring flexibility in natural rubber composites.

Ball-milled pyrolytic rice husk rubber composite demonstrated a significant enhancement of tensile strength, modulus, and tear strength compared to unmilled PRH (Xue et al. 2019). This study emphasized the importance of particle size, pore structure, and surface properties in optimizing the reinforcement capabilities of biochar-silica hybrids, showcasing rice husk BC as a high-performance bio-filler. The mechanical characteristics of natural rubber (NR) and NBR with BC and CB as fillers highlighted the complex interplay between filler type, surface characteristics, and elastomer properties in determining the mechanical properties such as hardness, tensile strength, elongation at break, modulus at 100% strain, and tear strength of rubber composites (Ribeiro et al. 2023; Ribeiro et al. 2023b). It was reported that NR formulations with biochar exhibited lower hardness compared to those with CB attributed to the interaction between the filler and the elastomer, crosslink density, and the final structure formed by the filler. However, certain biochar blends can approach the hardness levels of CB-filled NR despite a generally lower value. In terms of tensile strength, it was observed that biochar-filled specimens tend to have lower tensile strength compared to CB-filled ones. Similar behavior due to the presence of large biochar particles in natural rubber matrix was observed by (Peterson 2020) and the properties were improved after further optimization of particle characteristics through silica milling. Changes in the stretch crystallization extension of natural rubber are influenced by the crystalline properties, particle size, and agglomeration potential of biochar, contributing to its performance (Ribeiro et al. 2023) In terms of reinforcement capabilities indicate that biochar, despite its lower specific surface area, can enhance certain mechanical properties due to its unique structural characteristics (Ribeiro et al. 2023). However, the presence of BC reduces the Shore A hardness of the rubber composite compared to CB-loaded natural rubber composites.

The mechanical and physicochemical properties of spent coffee BC as a partial replacement for CB in epoxidized natural rubber (ENR) composites exhibited properties influenced by the degree of epoxidation (Raju et al. 2021). Spent coffee BC was able to replace 10–15% of CB, achieving nearly 99% of the tensile strength of CB while improving elongation properties. Furthermore, incorporating BC into ENR composites significantly improved the homogeneity of the material. These findings highlight the potential of using biomass-derived fillers, such as spent coffee biochar, as sustainable alternatives in the rubber industry, promoting eco-friendly advancements in material science.

4.3.2 Biochar-incorporated nitrile rubber (NBR) composites

Regardless of the particle size NBR composites have observed enhanced tensile strength and elongation at break for certain composites with increasing BC loading (Table S2). The behavior was attributed to the reinforcing effect of biochar and the increased crosslinking density resulting from hydrogen bonding between biochar and

Table 5 Mechanical properties of BC incorporated NR composites

Filler	CB replacement	M_L(dN.m)	M_H (dN.m)	ΔM(dN.m)	T_{S1} (min)	T_{S2} (min)	T_{90} (min)	CRI (min^{-1})	References
NR	0%	2.8	15.5	12.7	3.4		8.2	20.8	(Ribeiro et al. 2023)
NR/CB	0%	7.4	30.5	12.7	1.4		6.5	19.4	
NR/BIO400	100%	1.1	20.2	19.1	3.8		10.3	15.9	
NR/BIO900	100%	2.2	16.6	14.3	2.8		5.4	38.9	
NBR	0%	2.6	15.0	13.1	4.4		9.2	20.9	
NBR/CB	0%	4.5	12.6	8.1	3.1		6.7	28.1	
NBR/BIO400	100%	3.8	14.8	11.1	4.4		11.9	13.5	
NBR/BIO900	100%	2.9	7.4	4.5	4.6		8.3	27.2	
NR	0%	2.78	15.52	12.74	3.38		8.19	20.79	(Ribeiro et al. 2023)
NR/CB	0%	7.38	30.50	23.12	1.36		6.52	19.38	
NR/BIO 30 min	100%	2.21	16.55	14.34	2.84		5.41	38.91	
NR/BIO 60 min	100%	5.33	20.79	15.6	1.46		5.48	24.88	
NR/BIO 240 min	100%	3.02	16.66	13.64	1.26		7.28	16.61	
NBR	0 phr	0.85	13.02	12.17		7.26	13.89	15.08	(Koriem et al. 2023)
CB/ Mbi 40/0	0 phr	0.16	8.76	8.60		6.61	12.59	16.72	
CB/Mbi 0/10	10 phr	0.17	5.69	5.52		10.81	15.48	21.41	
CB/Mbi 0/20	20 phr	0.15	6.18	6.03		9.35	14.43	19.69	
CB/Mbi 0/40	40 phr	0.15	6.41	6.26		7.92	13.05	19.49	
CB/Mbi 30/10	10 phr	0.13	8.9	8.77		6.21	9.95	26.74	
CB/Mbi 20/20	20 phr	0.21	8.97	8.76		6.27	9.94	27.75	
CB/Mbi 10/30	30 phr	0.23	8.25	8.02		6.68	10.08	29.41	
NBR	0phr	2.13	16.3	14.2		2.17	6.13	25.3	Abidin et al. (2023)
CB/PKSBc (phr) 35/0	0phr	3.12	24.2	21.1		1.58	5.49	25.6	
CB/PKSBc (phr) 30/5	5 phr	3.00	23.4	20.4		2.08	5.54	28.9	
CB/PKSBc(phr) 25/10	10phr	3.64	23.5	19.9		2.0	5.12	32.1	
CB/PKSBc (phr) 20/15	15phr	3.63	25.9	22.3		1.5	4.31	35.4	
CB/PKSBc (phr) 15/20	20phr	3.68	22.2	18.5		2.03	5.1	32.6	
CB/PKSBc (phr) 10/25	25phr	3.18	20.5	17.3		2.11	5.17	32.7	
CB/PKSBc (phr)5/30	30 phr	3.08	23.9	20.8		2.05	4.51	40.7	
CB/PKSBc (phr)0/35	35phr	3.04	26.5	23.4		2.14	5.5	29.8	
CB/BC 0	0%	2.34	7.19	4.85		4.37	7.18		(Lubura et al. 2022)
CB 10	0%	2.44	8.76	6.32		3.30	6.45		
BC 10	100%	2.46	7.18	4.72		4.19	7.18		
CB 20	0%	2.88	9.33	6.45		2.35	6.52		
BC 20	100%	2.47	7.46	4.99		4.00	7.48		
CB 30	0%	3.53	10.71	7.18		2.52	6.55		
BC 30	100%	2.65	8.52	5.87		3.48	7.40		
CB 40	0%	3.85	12.27	8.42		1.51	6.50		
BC 40	100%	2.87	9.46	6.59		3.23	7.58		
CB 50	0%	4.82	13.77	8.95		1.22	6.56		
BC 50	100%	3.15	10.58	7.43		2.31	8.06		

Table 5 (continued)

Filler	CB replacement	M_L(dN.m)	M_H (dN.m)	ΔM(dN.m)	T_{S1} (min)	T_{S2} (min)	T_{90} (min)	CRI (min^{-1})	References
ORC (Oat husk biochar-filled rubber composite)	100%					6.2	11.8		(Bardha et al. 2023)
OARC (Activated oat husk biochar-filled rubber composite)	100%					3.6	9.1		
RRC (Rice husk biochar-filled rubber composite)	100%					5.7	11.1		
RARC (Activated rice husk biochar-filled rubber composite)	100%					4.3	13		
HRC (Hazelnut shell biochar-filled rubber composite)	100%					3.8	15.4		
HARC (Activated hazelnut shell biochar-filled rubber composite)	100%					3.8	9.9		
WRC (Walnut shell biochar-filled rubber composite)	100%					4.9	19.7		
WARC (Activated walnut shell biochar-filled rubber composite)	100%					4.4	13.9		
NRC (N772-filled rubber composite)	0%					3.9	8.1		
SBR	0%	0.74	9.44	8.70		9.15	15.27	16.34	(Jiang et al. 2020)
LBS-10	100%	0.89	10.22	9.33		3.58	7.98	22.73	
LBS-20	100%	1.07	12.52	11.45		2.22	5.90	27.17	
LBS-30	100%	1.30	13.79	12.49		1.70	5.50	26.32	
LBS-40	100%	1.46	13.86	12.40		1.53	5.78	23.53	
CBS-40	0%	1.63	14.11	12.48		4.05	8.18	24.21	

ML Minimum torque, *MH* Maximum torque, *ΔM* Torque difference (MH—ML) t_{S1}: Scorch time, T_{90} optimum cure time, *CRI* cure rate index, *NR* natural rubber, *NBR* Nitrile Butadiene Rubber, *CB* carbon black, *BIO* Bio-based filler (400 and 900 indicate different processing conditions), *PKSBc* Palm kernel shell biochar, *BC* Biochar, *Mbi* Modified biochar, *SBR* styrene butadiene rubber, *LB* lignin-derived nano-biochar, *LBS* lignin-derived nano-biochar added styrene butadiene rubber composites, *CBS* carbon black added styrene butadiene rubber composites

NBR (Koriem et al. 2023). However, higher micro-sized BC:CB ratios subsequently decrease rubber mobility due to significant filler–rubber interactions. This study provided valuable insights into the potential of micro-sized biochar as sustainable filler for NBR composites, emphasizing its reinforcing capabilities and stable crosslinking performance under various conditions (Koriem et al. 2023). Further, improved tensile strength in biochar–NBR blends compared to unfilled compounds is attributed to the enhanced interaction with elastomer chains, facilitated by the irregular surface of biochar (Ribeiro, Bérti, et al. 2023). However, this reinforcing effect is less pronounced when compared to CB-filled counterparts.

4.3.3 Biochar-incorporated styrene-butadiene rubber (SBR) composites

The mechanical properties of styrene–butadiene rubber composites filled with cornstarch and BC demonstrated good reinforcement properties, and they tend to increase brittleness in elastomers (Peterson and McMahan 2023). To address this, composites of starch-based fillers blended with biochar are formulated, aiming for a balance between reinforcement and reduced brittleness. The observed values are summarized in Table S3. CB-filled composites typically exhibit a good balance of tensile strength and toughness, and neither the starch-based fillers nor the biochar-filled composites match these properties. However, at a 10% filler concentration, both single-filled corn starch and the 3:1 corn starch–biochar blends overtake the carbon black control for all

measured tensile properties, offering potential for applications requiring rubbery material properties. Despite this, the biochar composites, including the blended fillers, still fall short of the toughness properties exhibited by carbon black-filled composites. These findings highlight the trade-offs between reinforcement and brittleness in rubber composites filled with renewable materials and suggest avenues for further research to optimize filler formulations for specific applications.

4.3.4 Biochar-incorporated blend of butadiene rubber–natural rubber (BR-NR) composites

The mechanical properties of BC–incorporated BR-NR rubber lattices are summarized in Table S3. The BR-NR is a soft and a pliable material. For most of the applications the BR-NR is required to be reinforced. The significant enhancement of tensile strength and reduction in elongation were observed in CB–incorporated BR-NR composites. However, substituting CB with BC, without silica milling, resulted in low tensile strengths attributed to the presence of large biochar particles (10–100 μm), inducing local stresses that weaken the composites. This reduction in tensile strength compared to the CB control emphasized the need for further optimization. Silica milling of biochar-filled composites proves to be a promising approach, showing improvements in tensile strength and toughness relative to unmilled biochar samples. Despite these advancements, the tensile strength and toughness of silica-milled biochar composites remain inferior to CB-filled counterparts. However, increasing the filler ratio of CB/biochar demonstrates potential for achieving comparable tensile strength to the CB control, particularly with the substitution of 30% CB with silica-milled biochar.

5 Challenges, limitations and future perspectives

In the preceding sections, we have explored various dimensions of biochar (BC)–rubber composites. Notably, there remain substantial gaps in the current knowledge base. For instance, research on BC composites has been predominantly confined to a limited range of rubber types, primarily focusing on natural rubber (NR). This narrow scope highlights the need for broader investigations encompassing a diverse array of rubber matrices to fully understand the potential and limitations of BC as a reinforcing filler. There are few studies done with polar rubber NBR (Koriem et al. 2023) and carboxylated nitrile butadiene rubber (XNBR) (Abidin et al. 2023). It is evident that the limited number of studies and the variability in experimental parameters across these investigations hinder the ability to draw accurate and definitive conclusions regarding the efficacy of BC in rubber composites. The inconsistent methodologies and differing conditions underscore the necessity for standardized research protocols. Consequently, there are numerous avenues for future research to explore and optimize BC-based rubber composites, including systematic studies across diverse rubber matrices and controlled experimental conditions.

It is proven that BC can be produced with variety of biomass. However, when incorporated to rubber matrixes BC obtained from different biomasses behaves varying manner, producing different properties in the final vulcanizate. Hence, it will be difficult to obtain large quantities of BC with high consistency in properties to replace CB in rubber composites. Furthermore, it is reported that over 15 million metric tons of CB produced annually that emit carbon dioxide in the range of 29–79 million metric tons each year (Rosner et al. 2024) and a large percentage of it goes for the rubber industry. The impact of replacing carbon black (CB) with biochar (BC) will be minimal if only a limited variety of biomass proves suitable for this purpose within the rubber industry. Two primary reasons for the inferior properties of BC composites, compared to those containing CB, are the composition and morphology of BC derived from biomass. These characteristics are influenced by various factors, including the type of biomass used and the pyrolysis techniques employed. Therefore, extensive fine-tuning is required to optimize the composition, purity, and morphology of BC to effectively replace CB in rubber composites. Some of the approaches used to address the issues are reducing BC particle sizes using ball-milling, incorporating some other materials during the ball milling (Peterson 2019; Peterson and Kim 2020), and surface modifications (Peterson and Thomas 2022). This is done in addition to changing the BC manufacturing process (Almutairi, et al. 2023). Furthermore, different biomasses were investigated as feedstock for the manufacture of BC to find the best composition. One of the biomasses shown better properties in the rubber composites is BC produced with lignin-based feedstock (He et al. 2022).

Another concern that can be highlighted is much lower carbon content compared to that of BC (Xiao et al. 2018). Again, the solution could be the fine tuning of the BC production process to suit the biomass feedstock and the postproduction treatments such as milling and surface treatments. These production processes should be tailored to produce a BC that has uniform characteristics suitable for the replacement of CB. There are various studies reported on replacing CB partially with BC (Abidin et al. 2023b; Peterson 2012, 2019, 2020). They have shown that the composites produced with a mixture of BC and CB have better properties compared to composites where BC is used alone. This approach is quite logical as the percentage of annual total BC production is less than 3% that of CB ("Global Biochar Market Report,"

2024; Rosner et al. 2024). Nevertheless, it will reduce carbon dioxide emission by 580,000 to 1.5 million metric tons, if the entire amount of BC produced is to be used in rubber composites replacing 3% of CB used currently. Hence, the partial replacement of CB with fine-tuned BC could be the aim of future research.

6 Conclusion

A significant number of various biomass-based BC has been incorporated to form rubber-based composites to replace CB which is the main reinforcing filler used currently. Biomass-based feedstocks used include wood base materials, agricultural wastes, nut shells, and some food wastes. The resultant BC materials had various morphological, and compositional drawbacks. Carbon contents reported in the BC range from 70 to 90 (wt.%) and the ash content was also high. In addition, BC properties such as difference in carbon structure and pH were also found to change from sample to sample. As the direct use of BC in place of CB in rubber composites did not yield good results, there were subsequent studies reported with post treatment of BC after the initial pyrolysis. These treatments include ball-milling of BC particles with different conditions, secondary materials, and surface treatments. Nevertheless, rheological and mechanical properties obtained using BC alone never reached the standards of CB whilst using BC with CB, replacing CB content partially, produced promising results. This could be the way forward; if research can find optimum properties for BC to incorporate into rubber matrix to replace CB partially, next task would be to produce tailormade BC to achieve the said properties. It need not to be a single protocol, but a series of them finetune to achieve required properties for different types of biomass feedstocks.

To reduce the fluctuation in mechanical properties, the optimal approach involves carefully choosing the type and quantity of BC for a specific polymer. The selection of feedstock and BC preparation protocol must be optimized to obtain the best type of BC for a given polymer. The reinforcement ability of biochar in rubber primarily depends on the porosity and surface area of the biochar, which increases the surface available for interaction with the rubber matrix. Additionally, surface chemistry, including functional groups and polarity, plays a crucial role in improving adhesion and compatibility with the rubber matrix. Particle size and distribution also significantly impact the uniformity and effectiveness of reinforcement. Moreover, the inherent mechanical properties of biochar, such as stiffness and tensile strength, contribute to the overall enhancement of the composite. Effective mixing techniques, appropriate curing conditions, and the thermal and chemical stability of biochar further influence its reinforcement capabilities in rubber.

Abbreviations

CB	Carbon black
BC	Biochar
NR	Natural rubber
SBR	Styrene butadiene rubber
NBR	Nitrile butadiene rubber
XNBR	Carboxylated nitrile butadiene rubber
BR	Butadiene rubber
ENR	Epoxidized natural rubber
PAUL	*Paulownia elongata*
POP	*Populus tremuloidis*
Poplar	*Populus canadensis*
RH	Rice husk
PKSBc	Palm kernel shell biochar
CRI	Curing rate index
BET	Brunauer-Emmett-Teller
MBi	Modified biochar
NS	Nano silica
HNS	Hazelnut nano silica
WNS	Walnut nano silica
OH	Oat Husk
RPM	Rounds per minute
CMBC	Chemically modified biochar
SA-CC	Stearic acid-coated calcium carbonate
SMR L	Standard Malaysian rubber light
M_L	Lowest torque
M_H	Highest torque
T_{90}	Curing time
T_{S2}	Scorch time
MMI	Marching modulus index
BIO	Bio based fillers
LB	Lignin derived nano-biochar
LBS	Lignin derived nano-biochar added SBR composite
CBS	Carbon black added SBR composite
DM	Dynamic modulus
Phr	Parts per hundred
PRH	Pyrolytic rice husk
Dm	Dry milled
E_m	Ethanol medium milled
W_m	Water medium milled
L_M	Lignin black liquor
L_MS	Lignin black liquor mixed silica
L_MB	Lignin black liquor mixed biochar ß
L_H	Lignin black liquor with xylose
CF	Corn flour

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1007/s42773-025-00429-3.

Supplementary material 1.

Acknowledgements
Not applicable.

Author contributions
Laleen Karunanayake: Conceptualization, Formal analysis, Methodology, Writing – original draft. Thusitha Etampawala: Conceptualization, Formal analysis, Methodology, Writing – original draft. Dilusha Jayanidu de Silva: Formal analysis, Methodology, Writing – original draft. Jagath Bandara: Formal analysis, Methodology, Writing – original draft. Anushka Upamali Rajapaksha: Formal analysis, Methodology, Writing – original draft. Meththika Vithanage: Conceptualization, Supervision, Writing – review & editing.

Funding
No financial assistance.

Availability of data and materials
All data will be available at a reasonable request.

Declarations

Ethics approval and consent to participate
No ethical approval needed.

Consent for publication
We confirm that there are no conflicts of interest to disclose related to this study.

Competing interests
Meththika Vithanage is an EBM of the journal Biochar, and she was not involved in the peer-review or handling of the manuscript. The authors have no other competing interests to disclose.

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Received: 30 July 2024 Revised: 19 December 2024 Accepted: 5 January 2025
Published online: 18 March 2025

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