WOODHEAD PUBLISHING IN MATERIALS

ADVANCED NANOCARBON POLYMER BIOCOMPOSITES

SUSTAINABILITY TOWARDS ZERO BIOWASTE

Edited by
MD REZAUR RAHMAN
MUHAMMAD KHUSAIRY BIN BAKRI

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WOODHEAD
PUBLISHING
An imprint of Elsevier

Woodhead Publishing is an imprint of Elsevier
50 Hampshire Street, 5th Floor, Cambridge, MA 02139, United States
125 London Wall, London EC2Y 5AS, United Kingdom

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ISBN: 978-0-443-13981-9 (print)

ISBN: 978-0-443-13982-6 (online)

Publisher: Matthew Deans
Acquisitions Editor: Gwen Jones
Editorial Project Manager: Tessa Kathryn
Production Project Manager: Maria Bernard
Cover Designer: Greg Harris

Typeset by MPS Limited, Chennai, India

Dedication

This work is dedicated to my amazing wife and daughters—Shirin Akther, Fahriah Rahman, and Faizah Rahman, who are very special to me and made it possible for me to complete this work.

—Ts. Dr. Md Rezaur Rahman

First, I would like to thank the Almighty God for the guidance, strength, power of mind, protection, and for giving us a healthy life. All of these we offer to you. Every difficult task needs self-effort as well as the guidance of elders, particularly those who are near to our hearts. I offer my humble dedications to my beautiful and loving father, mother, wife, and brothers, whose devotion, love, support, and nightly prayers have enabled me to work toward this significant achievement, along with all the dedicated, well-liked, and well-respected teachers and supervisors.

—Ts. Dr. Hj. Muhammad Khusairy Bin Bakri

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Ts. Dr. Hj. Muhammad Khusairy Bin Capt. Hj. Bakri obtained his doctor of philosophy, PhD (2018), master of engineering (by research), MEng (2016), and bachelor of engineering (mechanical engineer), BEng (2014) from Swinburne University of Technology, Australia (SUT). Currently, he is working with Washington State University (WSU) as a Postdoctoral Research Associate in Composite Materials and Engineering Center (CMEC), focusing on materials science, wood composites, polymer composites, biomaterials, and biocarbons. Previously, he was a Research Fellow at Universiti Malaysia Sarawak (UNIMAS) from 2019 to 2021, prioritizing materials science, membrane, concrete, polymer composites, biomaterials, and education. He was also a Higher Degree Researcher/Teaching Assistant from 2014 to 2018 in Swinburne University of Technology Sarawak Campus (SUTS). During that time,

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Preface

Integrating nanotechnology and polymer composites has emerged as a transformative paradigm in the rapidly evolving landscape of materials science and engineering, offering unprecedented opportunities to develop advanced materials with tailored properties and multifunctional applications. This book, *Advanced Nanocarbon Polymer Biocomposites*, represents a comprehensive exploration of the synergistic possibilities of the fusion of nanocarbons, polymers, and biocompatible elements.

Nanocarbon materials extracted from wood (pine and aspen) biomass (natural fiber, etc.) exhibit exceptional mechanical, thermal, and electrical properties. Harnessing the unique characteristics of these nanoscale entities and combining them with polymers, which provide flexibility, processability, and a wide range of functionalities, opens new frontiers in material design. Moreover, incorporating biocompatible components facilitates the development of materials that excel in mechanical, morphological, and chemical performance and demonstrate compatibility with living systems, paving the way for applications in biomedicine, construction and building, packaging, and sustainable technologies.

This book is crafted to provide a comprehensive overview of the fundamental and state-of-the-art research and developments in nanocarbon polymer biocomposites. Each chapter is meticulously crafted by experts in the respective areas, covering fundamental principles, synthesis methods, characterization techniques, and diverse applications. The chapters are organized to guide readers through the intricate landscape of nanocarbon polymer biocomposites, from theoretical foundations to practical applications, fostering a holistic understanding of this burgeoning field.

The multidisciplinary nature of this book makes it an invaluable resource for researchers, academics, and practitioners working at the intersection of nanotechnology, polymer science, and biocompatible materials. Whether delving into the fundamental science behind nanocarbon interactions with polymers or seeking insights into the practical applications of these advanced materials, this book serves as a roadmap to navigate the complexities and potentials of nanocarbon polymer biocomposites.

As editors, we would like to express our gratitude to the contributing authors for their scholarly contributions and dedication to advancing the knowledge in this field. We believe this compilation will inspire further exploration, foster collaboration, and contribute to the evolution of nanocarbon polymer biocomposites as a transformative technology.

Md Rezaur Rahman
Muhammad Khusairy Bin Bakri

CHAPTER TWO

Nanocarbon from pine wood sawdust and its biocomposites applications

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2.1 Introduction

The development and utilization of nanocarbon in innovative biocomposite products are receiving special attention from researchers. Most researchers' study covers the development of carbon based materials which range from activated carbon, carbon fiber, carbon nanotubes, nanocarbon powders, and graphene. However, the studies conducted on the source of the nanocarbon and utilization of the nanocarbon for further biocomposite development and applying to their respective applications are not a popular research topic. Therefore, in this book chapter, the topic regarding the development of nanocarbon from Pine wood sawdust biomass and the possible applications from the biocomposites developed are specifically selected. The compilation of studies compiled showcases the nanocarbon potential application in various fields, methods for developing nanocarbon from pine wood sawdust, characterization of the developed nanocarbon, and comparison with other types of nanocarbon. It also showcases the synthesis and characterization of properties of pine wood sawdust nanocarbon biocomposites, applications of the developed nanocarbon biocomposites, comparison over traditionally known composite materials, and future direction for improvements in this field of study.

Advanced Nanocarbon Polymer Biocomposites.
DOI: https://doi.org/10.1016/B978-0-443-13981-9.00002-8

There is significant waste produced by the wood industry, especially from pine wood sawdust. The disposal of this waste is not only expensive but also unsustainable for the ecosystem. As a result, there is an increasing need for environmentally friendly waste management techniques that may turn pine wood sawdust into products that are useful. Pine wood sawdust can be used as source material to synthesis nanocarbon, this offers a sustainable option for the industry regarding managing wood waste. The production process still needs to be made more effective and scalable, the end product, safety and environmental sustainability must be guaranteed, and regulatory difficulties must be resolved.

2.2 Pine wood sawdust

Pine wood is well known for its excellent quality joinery wood, which is used for furniture, windows, doors, shutters, paneling, siding, moldings, and other architectural millwork and joinery items. Wood processing generally produces lignocellulosic biomass in the form of sawdust and uneven chips, which can account for up to 20% of the total input mass (Foo & Hameed, 2012; Mao et al., 2014).

Wood properties vary primarily as a result of its growth pattern (Downes et al., 2000) and biological origin (Dias et al., 2020; Zobel & Buijtenen, 1989). From pith to bark, and from earlywood to latewood, there is variance throughout the tree (Zobel & Sprague, 1998). This high variability complicates the forecast of wood performance and, as a result, the efficiency of its processing and usage (Koga & Zhang, 2004). Wood quality is defined by its properties for specific end use, with density being the most important feature, followed by chemical composition (the content of cellulose, hemicellulose, and lignin) and mechanical properties (modulus of elasticity and rupture) (Zobel & Buijtenen, 1989). Wood density is the primary factor influencing timber strength, pulp yield, drying ease, machining, and hardness (Brazier & Howell, 1979; Elliot, 1970; Panshin & Zeeuw, 1980). This wood feature is determined by the cell size/wall thickness ratio, the proportion of earlywood/latewood, the number of ray cells, vessel components, and chemical composition (Cave & Walker, 1994; Zobel & Buijtenen, 1989). The chemical composition of wood (cellulose, hemicellulose, lignin, and extractive components) and

its variation is critical for a segment of the forestry sector, such as the pulp business. In this scenario, high cellulose, low extractive content, and lignin content are required to obtain high pulp output and brightness of the bleached paper (Campbell & Sederoff, 1996; Uner et al., 2009). Furthermore, the chemical composition of particle boards determines their strength (Uner et al., 2009). The mechanical properties of wood are of relevance in numerous fields of engineering, particularly in the selection and application of wood for certain end uses. The modulus of elasticity and rupture are two of the most often measured parameters for evaluating wood quality for structural components (Forest Products Laboratory, 1999). P. nigra is widely utilized in the forestry industry due to its ability to thrive in a variety of conditions and produce adequate products (Dias et al., 2020; Uner et al., 2009).

Wood factories can be found in almost every country in the world, and sawing is a constant daily operation that generates a lot of waste. In an open region, sawdust is routinely dumped, burned, or landfilled (Adu, 2014; Ogundipe & Jimoh, 2012) Sawdust is difficult to dispose of and, when burned, contributes to greenhouse gas emissions, despite the pollution and accompanying threats to public health, open fire is the most feasible method for saw millers to dispose of sawdust (Mwango & Kambole, 2019; Okedere et al., 2017; Olaiya et al., 2023). For many years, sawdust has been utilized in construction applications. This material could be found in large volumes, as well as being portable and light. Different physical and chemical properties of sawdust may vary from tree to tree (Olaiya et al., 2023). Construction applications have been using sawdust composite materials, such as sawdust low cost concrete, which has been around for more than 40 years (Kumar et al., 2014; Olaiya et al., 2023). In addition to concrete, the literature indicates that different sawdust composites have been used in a variety of building applications, including particle boards, bricks, floor slabs, paneling, partitioning and attic insulation.

In regards to specifically pine wood sawdust for polymer composite development, a few studies have shown improved mechanical properties such as withstanding impact forces (Martins et al., 2022) and flexural forces (Narlıoğlu et al., 2021). The improved impact strength properties of the polymer composite made from polyethylene (PE) matrix and pine wood sawdust as filler contributed to the lower weightage content of 10% and finer particle size which enables a compact structure to be developed in the composite material (Martins et al., 2022). The improved flexural strength properties of the polymer composite made from polylactic acid

matrix and pine wood sawdust as filler at 5% weightage content came to a similar conclusion of the improved mechanical properties (Narlıoğlu et al., 2021). Incorporating nanotechnology in polymer composite development may open up many possibilities for research. Simply reducing the particle size to smaller from macro, micro to nanosize will increase surface area. Some specific examples of size reduction of carbon based materials could be observed as the resulting effects of milling processes, for example, conventional ball milling and planetary ball milling (Garg & Das, 2018; Peterson et al., 2012; Zhang et al., 2019).

2.3 Development of nanocarbon from sawdust (pine wood)

This section discusses the development of pine wood sawdust into a nanosize carbonized material. Generally, the view on pine wood sawdust in this section is described as a basic sawdust material from lignocellulosic biomass. The main focus is to overview the development processes or techniques of achieving a nanosize carbonized material.

2.3.1 Biochar (carbon sawdust) synthesis via pyrolysis

Biochar has drawn a lot of interest from researchers to investigate practical uses in several fields of study, including energy production, soil amendment, water treatment, nutrient retention capacity, waste management, greenhouse gas reduction, and environmental restoration (Liu & Balasubramanian, 2014; Qian et al., 2015). The materials used as feedstock for synthesizing biochar include animal manure, agricultural waste or crop residue, and wood waste (Rajapaksha et al., 2016; Vijayaraghavan & Balasubramanian, 2021)

In terms of pine wood sawdust, it will be within the wood waste category for feedstock used. The synthesis of biochar generally involves thermal decomposition as the standard process. Pyrolysis, hydrothermal carbonization, gasification, and torrefaction, are examples of different thermochemical methodologies for synthesizing biochar (Amalina et al., 2022a; Tang et al., 2019; Wang & Wang, 2019).

Pyrolysis is a nonoxidative thermal breakdown process, as a result, 3 distinct product fractions are created: a solid residue called biochar, a

condensable liquid called bio oil, and a noncondensable gas called syngas (Amalina et al., 2022b; Rangabhashiyam and Balasubramanian, 2019; Senthil & Lee, 2021). Combustion will not occur when oxygen is not present in the environment; hence pyrolysis occurs instead. Pyrolysis commonly occurs at temperatures between 300°C and 700°C. The most advantageous method for synthesizing biochar and bio oil from biomass appears to be pyrolysis. This is due to the greater yields of biochar obtained with lower pyrolysis temperatures and longer residence durations. Longer reaction times are known to encourage polymerization, which increases the synthesis of biochar. More liquids often come from moderate temperatures and brief reaction times (Yaashikaa et al., 2019). These thermochemical reactions operate under a variety of conditions, such as residence periods between 1 second and hours, heating rates between 11°C and 1000°C/second, and temperatures between 300°C and 700°C or higher (Rangabhashiyam and Balasubramanian, 2019). It is essential to choose the pyrolysis procedure carefully in order to get a desirable outcome because each pyrolysis produces a different proportion of the byproducts. The direct conversion of biomass during the pyrolysis process can be characterized along three different pathways: fragmentation, depolymerization, and char formation (Karimi et al., 2018; Rangabhashiyam and Balasubramanian, 2019; Yaashikaa et al., 2020). The synthesis of biochar is frequently encouraged via intramolecular and intermolecular rearrangement processes, resulting in a more thermally stable residue. This path is established by the formation of benzene rings, which are then combined to form an aromatic polycyclic structure (Amalina et al.a, 2022; Karimi et al., 2018).

Slow pyrolysis increases biochar production by activating secondary reactions through extended vapor residence times, the slow pyrolysis generates both primary and secondary char. The lower temperature range of 300°C to 550°C, slow heating rates of 0.1°C to 0.8°C/s, and a longer contact period of 5−30 minutes or 25−35 hours characterize slow pyrolysis (Das et al., 2021; El-Naggar et al., 2019). Furthermore, the moderate heating rate mixed with the medium pyrolysis heat promotes the creation of biochar. The biochar yield is determined by the properties of the materials as well as the pyrolysis processes, specifically the temperature, heating rate, and pyrolysis reactor (Sonu et al., 2020). The yield of biochar formed from mineral rich biomass is lower. Due to the methodical development of secondary reactions, slow pyrolysis may be an exothermic reaction. Particles with sizes ranging from 5 to 50 mm can be absorbed by slow

pyrolysis (Sakhiya et al., 2020). The reaction in intermediate pyrolysis is faster than slow but slower than fast pyrolysis. It occurs between 450°C and 550°C, is faster than slow pyrolysis, lasts 10−30 seconds, and produces less biochar than slow pyrolysis (Ge et al., 2021; Sakhiya et al., 2020). At appropriate temperatures, intermediate pyrolysis chemosynthesis inhibits the formation of high molecular weight tars and produces a variety of product qualities, that is; biochar, bio oil, and syngases. In intermediate pyrolysis, the size and form of the biomass particles are less important than in fast pyrolysis. It can handle a wider variety of biomass, from larger particles to pellets and chips, as well as material containing over 40% biomass (Mbarki et al., 2019).

Fast pyrolysis is distinguished by the higher temperature settings, rapid heating rates (10°C to 1000°C/second), and short residence durations (0.5 to 2 seconds). By utilizing short vapor residence periods and maintaining high biomass heating rates, fast pyrolysis eliminates secondary reactions. It increases bio oil yield (Mutsengerere et al., 2019; Tomczyk et al., 2020).

Byproduct distribution is influenced by biomass composition, heating rate, and temperature. If bio oil is the desired result, the ideal pyrolysis temperature range is 425°C−600°C with a maximum heat of less than 650°C. However, if gas generation is the primary goal, the peak temperature can approach 1000°C (Ge et al., 2021). A finely powdered biomass feedstock, frequently less than 1 mm in size, is required to provide extremely high heat transfer rates and, as a result, extremely high heating rates, easing mass and heat transfer restrictions (Ravindran et al., 2018). Although only primary carbon is produced in fast pyrolysis, biochar yields are frequently insufficient. The entire fast pyrolysis process is endothermic due to the lack of secondary reactions. To decrease water in the final bio oil, fast pyrolysis suggests biomass with less than 10% moisture content by weight. Furthermore, a low moisture content allows the feed to be ground into fine enough particles to allow for rapid heating and pyrolysis (Amalina et al., 2022a; Lee et al., 2020).

Flash pyrolysis yields mainly the same products as rapid pyrolysis. It occurs between 800 and 1000°C, requiring excellent biomass feed particles (0.2 mm). The goal of flash pyrolysis is to optimize bio to oil production. It is distinguished by high temperatures, rapid heating (> 1000°C/second), and short contact times (0.5 seconds) (Amalina et al., 2022a; Gaurav et al., 2020).

One of the more intriguing methods for accelerating and optimizing chemical processes is microwave assisted pyrolysis. Due to the superior

heat transfer profile, chemical reactions occur faster and more efficiently in comparison to other thermochemical processes (Yin et al., 2018). Microwave assisted pyrolysis has several advantages over conventional pyrolysis, including consistent heating, rapid heating rate, volumetric and selective heating. It is also known that microwave assisted pyrolysis has a quick on/off control while increasing production and product output quality (Amalina et al., 2022a; Xiang et al., 2020).

Studies have shown that increasing microwave power results in a decrease in biochar yield (Arafat Hossain et al., 2017; Hossain et al., 2016; Nizamuddin et al., 2016; Safarian, 2023; Sahoo & Remya, 2022; Wallace et al., 2019)

A study on the optimization of process parameters for microwave pyrolysis of oil palm fiber for hydrogen and biochar production demonstrates that microwave power influences biochar and syngas yields, with lower microwave power favoring biochar yield and limiting gaseous yield. It was also reported that the biochar yield at 400 W microwave power is 48.2 wt.%, which drops to 31.2 wt.% at 900 W microwave power (Arafat Hossain et al., 2017). A study conducted by the same researcher explains that higher microwave power leads to higher heating rates, and higher heating rates cause an increase in thermal cracking, resulting in an increase in syngas yield and a decrease in biochar yield (Hossain et al., 2016).

The key variable affecting the distribution of the products during the microwave pyrolysis of the feedstock biomass is the temperature. By simultaneously adjusting the temperature and microwave power, it is possible to change both the yield and quality of the biochar product. It was determined that yielding more biochar with high carbon content occurs at lower temperatures, whilst producing less biochar with a nearly unchanged quality at higher temperatures. In regards to feedstocks such as softwood chips, changing the temperature and microwave power from 348.4°C to 459.8°C and 2100 to 2700 W, respectively, resulted in a reduction in biochar yield from 40% to 24%. They discovered that as the temperature was raised from 400°C to 700°C, the production of biochar decreased, but there was no change after that point (Safarian, 2023; Wallace et al., 2019).

Table 2.1 shows examples of different studies regarding activated char synthesis from sawdust as raw material. The majority of the studies mentioned involve slow pyrolysis, which many attribute to high carbon content and high activated carbon/biochar/char yield.

Table 2.1 Examples of activated carbon-char synthesis from sawdust as raw material.

Type of process	Raw material	Catalyst	Temperature	References
Brief carbonization via slow pyrolysis, activated with microwave pyrolysis	Chengal wood sawdust	Potassium carbonate (K_2CO_3)	700°C	Foo and Hameed (2012)
Carbonization via slow pyrolysis	Pine wood sawdust pellets	Carbon dioxide (CO_2)	800°C	Nowicki and Pietrzak (2010)
Carbonization via slow pyrolysis	Oak wood sawdust	Nickel (II) acetate tetrahydrate ($Ni(CH_3COO)_2 \cdot 4H_2O$), nitric acid ($HNO_3$) solution, and potassium carbonate (K_2CO_3)	400°C	Zhang et al. (2012)
Carbonization via slow pyrolysis	*Havea braziliansis* (rubberwood) sawdust	Potassium carbonate (K_2CO_3)	600°C	Krishnan et al. (2010)
Carbonization via slow pyrolysis	Rubberwood sawdust	Potassium hydroxide (KOH)	700°C–900°C	Phainuphong et al. (2022)
Carbonization via slow pyrolysis	Cedar deodar sawdust	-	350°C–650°C	Varma et al. (2019)
Carbonization via fast pyrolysis	Mixed wood waste sawdust	-	400°C–700°C	Salehi et al. (2009)
Carbonization via fast pyrolysis	Mixed wood waste sawdust	-	400°C–600°C	Duanguppama et al. (2016)
Carbonization via slow pyrolysis	Pine wood (*Pinus radiata*) sawdust	Zinc chloride ($ZnCl_2$)	600°C	Pimentel et al. (2023)
Carbonization via slow pyrolysis	Pine wood (*Pinus strobus*) sawdust	Sodium hydroxide (NaOH), phosphoric acid (H3PO4), oxalic acid ($C_2H_2O_4$)	600°C	Yakout et al. (2019)
Carbonization via slow pyrolysis	Durian wood (*Durio zibethinus*) Sawdust	-	350°C–550°C	Chowdhury et al. (2016)

2.3.2 Biochar (carbon sawdust) size reduction and activation techniques

2.3.2.1 Physical activation processes via ball milling

Nano particles synthesized mechanically through the process of milling utilizes equipment intended to apply mechanical forces to the materials to be processed, such as ball mills, planetary mills, or vibrating mills. Ball milling is an effective nonequilibrium processing technique which utilizes physical work done mechanically to reduce the solid particles feedstocks from a macro size scale into the micro and/or nanosize scale (Lyu et al., 2017; Soares et al., 2015; Ullah et al., 2014).

A study on the impact of ball milling on the physicochemical and sorptive properties of biochar exploring the experimental observations and governing mechanisms, shows that utilizing ball milling processes on activated carbon for modifying carbon based nanocomposites produces improved properties for real life applications. Due to activated carbon and biochar having shared characteristics and properties, ball milling could be utilized as a physical modification technique to create a biochar tailored from different targeted applications (Gao et al., 2015; Lyu et al., 2018; Ramanujan et al., 2007). It could be linked, that the biochar can be an activated carbon, from simply using ball milling processes, to achieve increased surface area, porosity, pore size, and sorption capabilities. These improved properties/characteristics are in line with what is considered to be an activated carbon. The processing time of ball milling to achieve micro to nanosize range samples may vary according to the volume of feedstock, type and number of balls used. Some studies have shown processing time ranging from 1 to 24 hours of ball milling (Amusat et al., 2021; Lyu et al., 2018).

According to a review study conducted on ball milling synthesis of biochar and biochar—based nanocomposites and prospects for removal of emerging contaminants, there is a scarcity of research specifically on the ball milling methods to modify biochar to develop composite materials. However, the majority of the research stated by the author, do discuss the improved properties of biochar after ball milling, in which leads to improved application performances in usage in filtration of contaminants, such as dyes, heavy metals, and other organic and inorganic contaminants. In terms of wet and dry ball milling procedures, one comparative study found that ball milled biochar improved its specific surface area by 200 times that of pristine biochar, but no significant difference was found between the two processes (Amusat et al., 2021; Yuan et al., 2020). According to one study, it is hypothesized that in

ball milling biochar enhances both its internal surface area and external surface area by opening the inner pore networks. Biochar that had been ball milled had pores that were 6.4 to 48 times larger than biochar samples that have not undergone ball milling (Lyu et al., 2018).

2.3.2.2 Chemical activation processes of biochar

According to a study on the recent advancements and challenges in emerging applications of biochar based catalytic agents, chemical activation of biochar is normally performed at a moderate temperature with or without the thermal processes to assist further biochar activation (Kumar et al., 2020; Yuan et al., 2022). Acid and alkaline chemical activation, and impregnation of metals, salts, and oxides are the most common activation processes. Various chemicals, such as potassium hydroxide (KOH), phosphoric acid (H_3PO_4), sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), and zinc chloride ($ZnCl_2$), are employed for chemical activation of biochar (Patel et al., 2022; Yuan et al., 2018, 2023.

During the soaking stage, chemicals dilate and hydrolyzed the biochar, retains porosity during the carbonization stage, and inhibits the generation of undesirable compounds (Kumar et al., 2020) Minerals are removed in an acidic alteration to improve the hydrophobicity of biochar (Zhang et al., 2015). Alkaline treated biochar produces greater positive surface charges (Li et al., 2014). As a result, chemical changes have a greater impact on increasing biochar surface functioning than physical activation (Yuan et al., 2021). Alkaline treated biochar, in particular, has the highest surface functionality (Ahmed et al., 2016; Yuan et al., 2021). Acidic treatments, on the other hand, increase the oxygenation functional groups of biochar (Ahmed et al., 2016; Yuan et al., 2021; Zhang et al., 2015). According to one study on synthesizing engineered biochar for CO_2 capture for sustainable food waste management, alkaline chemical activation of biochar using Potassium hydroxide (KOH) at 600°C for 1 hour has increased in surface area of 807 (m^2/g). The researcher has also compiled surface area data of similar chemical activation parameters from different studies has shown biochar surface area to be ranging 947 to 1479 (m^2/g) (Chen et al., 2015; Huang et al., 2015, 2019; Yang et al., 2018, 2022).

2.3.2.3 Surface functionalization via hydrothermal processes for biochar activation

The incorporation of surface functional groups to the carbon surface of the biochar through surface functionalization via hydrothermal process has

been proposed as an additional method to enhance biochar adsorption performance. Although hydroxyl and carbonyl groups are among the functional groups that biochar naturally possesses, their presence is minimal in comparison to the material's overall surface (Ibrahim et al., 2021; Wang & Liu, 2017).

Numerous research demonstrate the benefits of increasing surface functional groups for biochar and other carbon materials' adsorption capabilities, particularly for dyes and heavy metals. The acidic oxygen functional groups increase electrostatic interaction between the functional groups and the adsorbate molecule, particularly negatively charged molecules like heavy metals and cationic dyes, and the functional groups increase its hydrophilicity, increasing efficiency in wastewater application (Ibrahim et al., 2021; Liu et al., 2015; Park et al., 2010; Sophia A. & Lima, 2018; Wang & Wang, 2019). To increase the oxygen surface functional groups on biochar, oxidizing agents like nitric acid could be used to functionalize the surface. While successful, surface functionalization of biochar employing nitric acid via a chemical soaking treatment technique often takes an extended period of time with a higher solution concentration of nitric acid (Tan et al., 2017; Ibrahim et al., 2021). Furthermore, the functionalization process has a tendency to change the morphology and pore structure of the changed carbon material, which reduces its surface area. Most industries find it difficult to adopt the functionalization process due to the risk and expense involved, the reduction in surface area, the use of very caustic chemicals, and the excessively lengthy time required. With this issue in mind, an autoclave functionalization procedure is recommended to speed up biochar functionalization and improve the surface functionalities of biochar, which will then improve its adsorption performance.

The combination of increased temperature and pressure during treatment distinguishes the autoclave from other documented methods of modification. According to the researcher, this will accelerate nitric acid's conversion to nitrogen containing species like nitrous acid, which can also oxidize organic molecules (Catherine and Housecroft, 2008; Ibrahim et al., 2021). The process efficiency can be considerably improved when there are several oxidation paths accessible.

According to Ibrahim et al. (2021), the production of biochar as an adsorbent from oil palm empty fruit bunch biomass utilizing a simple functionalization procedure in an autoclave. The surface of the biochar was functionalized in this system by using nitric acid as an oxidation agent

to increase the number of functional oxygen groups on the surface. The functionalization process was sped up and made more effective using an appliance called an autoclave. In other words, compared to other surface functionalization processes previously reported, the hydrothermal functionalization process could increase the quantity of oxygen surface functional groups on the surface of the biochar using a lower concentration of nitric acid, a shorter period of time, and a simpler process. The resulting biochar has more surface functional groups, which is crucial for the adsorption of contaminants from aqueous solutions like dyes and heavy metals. By comparing treated and untreated biochar, the hydrothermally treated biochar performed better during application, with a sevenfold increase in adsorption capacity from 8.70 ± 0.09 to 62.52 ± 0.48 mg/g (Ibrahim et al., 2021).

Table 2.2 shows examples of size reduction and modifying techniques from different studies for activated carbon. On average, the majority of the studies are able to achieve good sample size and specific surface area properties for their activated carbon samples. Most studies may suggest the main effects of these properties are the process used and pretreatments.

2.4 Synthesis of nanocarbon (biochar) biocomposites

Nanocarbon materials categorized as carbon nanotubes single walled (SWCNTs), multiwalled (MWCNTs), graphene (G) or graphene oxide (GO), activated carbon (e.g., biochar), carbon nanoparticles (CNPs) have gained popularity due to their unique structural regularity, high surface area, electrical conductivity, chemical inertness, biocompatibility, mechanical and thermal stability (Allen et al., 2010; Gopiraman & Soo Kim, 2019; Rahman et al., 2011; Vairavapandian et al., 2008). One way to utilize or incorporate their unique properties for different real life applications is to develop nanocomposites. Generally, the methodology of developing nanocomposites with nanocarbon materials depends on the desired properties, application, and cost. This section will focus on the standard techniques for incorporating nanocarbon with polymer matrices, as there is a wider field of real-life applications which benefit both polymer and nanocarbon properties.

According to the handbook of carbon nanotubes (CNTs) polymer nanocomposites, techniques for the preparation of CNT reinforced

Table 2.2 Examples of size reduction and modifying techniques for activated carbon.

Type of process	Pretreatments and catalyst	Average size	Specific surface area	References
Planetary ball milling	-	75 μm	>3000 m^2/g	Eguchi et al. (2020)
Planetary ball milling	-	521 nm	432 m^2/g	Baheti et al. (2015)
Chemical activation followed by thermal annealing	Potassium hydroxide (KOH)	-	1303–2004 m^2/g	Nowicki (2016)
Chemical activation, followed by thermal annealing	(Oxidation) nitric acid (HNO_3), (oxidation) hydrogen peroxide (H_2O_2)	-	582–657 m^2/g	Gil et al. (2019)
Catalytic chemical vapor deposition	Nickel (II) nitrate hexahydrate ($Ni(NO_3)_2$)	100–200 nm	837 m^2/g	Ahmed et al. (2016)
Catalytic chemical vapor deposition	Nickel (Ni) impregnation, and calcination in N_2 atmosphere	15.2–39.8 nm	792.3 m^2/g	Rezvani et al. (2019)
Hydrothermal processes	Nitric acid (HNO_3)	50–120 nm	79 m^2/g	Sedira and Mendaci (2020)
Hydrothermal processes	Potassium hydroxide (KOH)	4.48–11.74 μm	3026 m^2/g	Hao et al. (2016)

polymers include: solution processing, bulk mixing, melt mixing, and in situ polymerization (Nasir Mahmood, 2014). The stated CNT polymer nanocomposites preparation techniques could be utilized with the developed pine wood sawdust nanocarbon/biochar/activated carbon to synthesize similar nanocomposites. According to a study on nanobiochar and biochar based nanocomposites advances and applications, as biochar is characteristically a carbon nanomaterial and has been explored for its numerous potential applications, biochar is a cheaper and sustainable precursor for synthesizing CNT. As the biochar is synthesized from lignocellulosic plant biomass (e.g., pine wood sawdust), the biochar could be identified as a possible agent for phytoremediation of various contaminants such as organic and inorganic pollutants, as well as heavy metals in wastewater (Chausali et al., 2021; Noreen & Abd-Elsalam, 2020). Hence, techniques for synthesizing CNT polymer composite by the researcher may be applied to other nanocarbon forms, for example, pine wood sawdust biochar, activated carbon, and nanocarbon particles.

A technique for synthesizing CNT polymer composites called solution processing involves combining the two materials with a specific solvent. The solvent is then evaporated under a vacuum or at a higher temperature to synthesize the composite. The vacuum assists in removing the solvent from the mixture as well as any tiny air bubbles that may otherwise negatively affect the properties. The high temperature helps the solvent evaporate and starts the curing reaction of the resin. In simple terms, the solution processing techniques follow a general approach that entails dispersing CNT powder in a liquid medium using stirring and/or sonication, combining the CNT dispersion with a polymer solution, and carefully evaporating the solvent (Nasir Mahmood, 2014; Ma et al., 2010). Synthesizing the composites with a uniform distribution of CNTs depends on both mechanical stirring and ultrasonication. The ultrasonic, abrupt collapse of cavitation bubbles that are produced as ultrasonic waves pass through a liquid media is linked to the chemical effects of ultrasounds. According to sonochemical theory and the accompanying research, ultrasonic cavitation can produce a harsh local environment with temperatures and pressures as high as 5000 K and 500 atm. MWCNTs were mechanically combined with epoxy resin and hardener after being dispersed in a solvent using a similar technique for thermosetting resin in our investigation. After the solvent had evaporated, the CNT-resin mixture was cast into capsular mold for the finishing curing process (Nasir Mahmood, 2014).

Bulk mixing could be characterized as a fabrication technique as a result of high pressure localized from the grinding media and high energy impacts during the milling processes. Carbon nanostructures are reduced in length or size via milling processes. According to the researcher, CNT/polypropylene composite powder has been reported to be prepared via pan milling (a solid state mechano chemical pulverization method). To create a homogenous composite, the powder was melt mixed via a twin roll masticator. The length of the CNT is reduced from the microsize range to within the 500 nm range (Nasir Mahmood, 2014).

In addition, the melt mixing technique involves the use of thermoplastics, which softens and flows in a molten state when heated above or at the melting point. Therefore, when utilizing thermoplastic, the melt mixing technique is an important technique for the synthesis of CNT polymer composites. Melt mixing could be the standard technique used for thermoplastic polymers, in which are insoluble in common solvents (solution processing).

Melt mixing generally involves melting and blending the thermoplastic polymer with CNT material via high shearing forces. Mold casting is also utilized, depending on the final morphology/shape of the composites, the bulk samples could be extruded into a mold using an extruder (Nasir Mahmood, 2014; Sahoo et al., 2010).

In situ polymerization involves the incorporation/dispersion of CNTs in a monomer or thermoset polymer (resin or epoxy) which immediately proceeds to the polymerization process. This technique has the added advantage due to the higher volume fraction of the dispersion of CNTs, hence attaining good uniform dispersion and developing a stronger CNT matrix bonding. This technique is effective for synthesizing nanocomposites utilizing polymers that could not be processed via solution processing or melt mixing, such as insoluble and thermally unstable polymers (Sahoo et al., 2010). In situ polymerization of vinyl monomers in the presence of CNT material has piqued the interest of researchers working on functional composites (Nasir Mahmood, 2014; Spitalsky et al., 2010).

Table 2.3 shows examples of synthesis processes for nanocarbon polymer composites with their respective mechanical properties, processes and polymer types. The majority of the studies mentioned yield different mechanical properties, attributed to the polymer and carbon base used. The process used may be optimized for achieving a better sample quality, poor handling during the processes may affect the composite properties.

Table 2.3 Examples of synthesis process and mechanical properties of nanocarbon polymer composites.

Type of process	Type of polymer	Type of carbon base	Mechanical properties	References
Melt mixing	Polypropylene	Multi walled carbon nanotube	– Tensile strength 35.23–38.23 MPa – Young modulus 1843.08–2251.56 GPa	Stanciu et al. (2021)
Melt spinning	Polypropylene	Single walled carbon nanotubes fibers	– Average yield strength 380 MPa – Average Young modulus 6 GPa	Acierno et al. (2017)
Shear mixing and melt mixing	Isotactic polypropylene	Single walled carbon nanotubes	– Yield Strength 24.7–31 MPa – Young modulus 868–1187 MPa	Manchado et al. (2005)
In situ polymerization	Epoxy resin	Nanocarbon fiber and E glass fiber	– Tensile strength 355–390 MPa – Hardness 55–75 HRC	Vinayaka et al. (2023)
Melt mixing	High density polyethylene	Activated carbon, carbon briquettes, carbon black	– Ultimate tensile strength >31–34 MPa – Flexural modulus >800–840 MPa – Izod impact strength >30–35 MPa	Choudhury et al. (2021)
In situ polymerization	Polyethylene	Multi walled carbon nanotube	– Tensile strength 30.1 MPa – Young modulus 132 MPa	Al-Harthi and Bahuleyan (2018)
In situ polymerization via heating & compression molding	Epoxy resin	Activated carbon	– Tensile Strength 19–20 MPa – Young modulus >7000–9000 MPa – Hardness 95–96 (Digital shore scale "D")	Mohmad et al. (2018)
Electrospinning	Cellulose acetate	Activated carbon	– No mechanical testing – Oil sorption capacity 8.2 g/g	Narlıoğlu et al. (2021)
Shear mixing, in situ polymerization	Cellulose, chitosan	Activated carbon	– Compressive resistance 53.6–98.5 KPa – Compressive modulus 214–394 KPa	Ergun (2023)
Shear mixing, in situ polymerization (phase inversion)	Cellulose acetate	Activated carbon	– No mechanical testing – Dyes adsorption equilibria 58.23–58.69 mg/g	Zhao et al. (2019)

2.5 Applications of nanocarbon (pine wood sawdust) biocomposites

The applications of nanocarbon and nanocarbon composites for this section of the chapter may be directed towards the nanocarbon derived from activated carbon or biochar, as the theoretical source material is sawdust (pine wood). Hence, according to a study on nanobiochar and biochar based nanocomposites advances and applications (Chausali et al., 2021), there are several categories of potential applications for nanobiochar and biochar based nanocomposites. For example:

Environmental, Energy and Material Science applications:

- Energy production;
- Organic and inorganic pollutant removal;
- Water and wastewater treatment;
- Carbon sequestration (mitigating climate change);
- Agricultural application (fertilizer and soil amendment).

Among the applications involving nanobiochar polymer composites are limited to improving polymer or plastic based products. Hence, the potential applications are regarding improving packaging, films, coatings, various thermoplastic extruded product parts, membranes for water and wastewater treatment and recently, electroconductive composite materials (Rahman et al., 2011). However, for this section of the chapter regard applications of nanocarbon biocomposites, energy production, organic and inorganic pollutant removal, water and wastewater treatment, carbon sequestration (mitigating climate change), and agricultural application (fertilizer and soil amendment) are discussed.

Researchers extensively studied and demonstrated the use of macro biochar in environmental applications. However, nanobiochar is currently being researched for a number of environmental applications, including waste management, pollutant removal, wastewater treatment, and carbon sequestration (Chausali et al., 2021). However, it has been established that carbon dioxide created by the combustion of biomass, which includes plant matter or wastes, is ultimately consumed by plants, making this a carbon neutral source of energy. As biochar is applied to agricultural areas, the carbon is highly resistant to further deterioration and is sequestered for an exceptionally long period of time. As a result, biochar has been recognized as a carbon negative energy resource, providing energy while sequestering carbon. Biochar has emerged as a potential solution in relation to

numerous environmental challenges such as climate change (Timmons et al., 2017). Furthermore, it is now being researched for the creation of sustainable energy and electrode applications, as well as its increased features via various pre and postmodification approaches (Ramanayaka et al., 2020). Nanobiochar has acquired attention as an excellent adsorbent due to its greater adsorption capacity, among other responsibilities. Pharmaceuticals, steroid hormones, pesticides, hazardous metals, and personal care products have all been successfully removed using nanobiochar and its sorption capabilities (Taheran et al., 2018) These micropollutants spread easily in the atmosphere as a result of numerous human activities, posing a hazard to human health and the environment. Contaminants enter the environment through improper wastewater discharge, trash disposal, and pesticide use (Ramanayaka et al., 2020). When compared to ordinary biochar, nanobiochar and biochar enhanced with nanominerals showed exceptional adsorption capability for the remediation of different pollutants (Ma et al., 2019; Samsudin et al., 2019). Furthermore, by adsorbing hazardous substances such as pesticides and immobilizing metals, biochar can remediate pollution, which can create serious environmental and health issues (Cernansky, 2015). As a result, nanobiochar has been regarded as a promising agent for bioremediation of a wide range of pollutants.

Heavy metal contamination in water is a major environmental concern due to its buildup and toxicity to humans, land and aquatic life. Heavy metals, unlike other organic and inorganic pollutants, cannot be degraded (Ramadan et al., 2020), therefore their removal is a worry. Adsorption, ion exchange, chemical precipitation, and other strategies for heavy metal removal have been cited (Zhu et al., 2012), but adsorption from aqueous solution/effluent has become the most popular due to its economic viability (Guo et al., 2018). Adsorbents such as activated carbon, flat iron oxide, silica gel, zeolite, and attapulgite, as well as carbon based nanofibers, have a few drawbacks, including limited adsorbent capacity, oxidation and assimilation ability, cost ineffectiveness, and lower selectivity (Gan et al., 2015; Ramadan et al., 2020). Biochar is currently being investigated as a promising adsorption agent for eliminating pollutants, which may also be useful in water pollution prevention (Tan et al., 2015). Advanced technologies that incorporate nanoparticles into biochar improved its characteristics and function for a variety of applications, including wastewater remediation and carbon sequestration (Ramadan et al., 2020; Zhang et al., 2013). Notable in particular was the simultaneous adsorption and catalytic degradative action of catalytic material coated biochar for the removal of

organic pollutants (Tan et al., 2016). However, conventional biochar showed a limited ability to absorb ionic contaminants (Yao et al., 2013). Current research has demonstrated numerous techniques for synthesizing various types of nanobiochar to increase its adsorption ability for aqueous and ionic contaminants (Ramadan et al., 2020).

Industrialization, deforestation, overgrazing, and various soil tillage activities have dramatically lowered soil nitrogen levels. Nutrient depletion reduced production and plant development, threatening food security (Ramadan et al., 2020). Chemical fertilizers have been used extensively to maintain soil nutrients. Nutrient leaching and runoff, on the other hand, reduced bioavailability in soil and promoted chemical based fertilizers. This was the cause of soil pollution and a negative impact on soil biota in several regions (Bah et al., 2014). Biochar had a favorable impact on soil properties because it not only increased water retention, permeability, and soil fertility, but its high charge density also carried large amounts of nutrients, enhancing crop output(Braghiroli et al., 2019; Liu et al., 2016).

Biochar made from lignocellulosic biomass contains not only carbon rich products, but also higher concentrations of other macronutrients such as nitrogen (N), phosphorous (P), magnesium (Mg), potassium (K), calcium (Ca), and sulfur (S), as well as micronutrients such as copper (Cu), manganese (Mn), iron (Fe), zinc (Zn), and ash (Hossain et al., 2011), making it a high performance organic fertilizer. Total P and N were found to be higher in biochar produced from animal feedstock such as grill litter and sewage sludge than in biochar produced from plant biomass such as wood and green garbage (Bopp et al., 2016). Combining biochar with other fertilizers, such as urea, revealed a significant increase in yield while reducing N input (Joseph et al., 2013; Qian et al., 2014).

In addition, biochar composites which contain minerals and organic components could enhance fertilizer utilization. Biochar derived nanocarbons have piqued the interest of researchers due to their ability to store water and nutrients (Joseph et al., 2013; Manyà, 2012) and can be used as nanofertilizers (Manyà, 2012; Tiwari et al., 2014). Furthermore, according to Zhang et al. 2020, the researchers demonstrated that the use of biochar carbon nanoparticles significantly boosted wheat crop development and productivity (Zhang et al., 2020). Lateef et al. investigated if a biochar nanocomposite derived from maize cob helped to safeguard the environment by transforming trash into a valuable product while also addressing traditional fertilizer leaching issues (Lateef et al., 2019). However, the efficiency of biochar based fertilizers in immobilizing heavy metals while also supplying nitrogen requires additional investigation (Chausali et al., 2021; Ramadan et al., 2020).

2.6 Conclusion

In conclusion, the incorporation of nanocarbon materials derived/synthesized from pine wood sawdust offers great potential for the production of sophisticated polymer composites with several applications. Converting sawdust into nanocarbons such as activated carbon, carbon nanotubes, or graphene is an environmentally beneficial and sustainable method of obtaining these important materials. Overall, the use of nanocarbon compounds produced from sawdust in polymer composites holds great promise for a variety of sectors. These composites are very appealing for improving technologies in the waste and wastewater treatment, energy storage, electronics, agriculture and environmental sectors due to their sustainability and advantageous properties. More research and development in this subject will definitely lead to even more inventive applications, paving the way for a more sustainable and technologically advanced future.

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About the Editors

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ISBN 978-0-443-13981-9
9 780443 139819