

Review

Hydrochar Production by Hydrothermal Carbonization: Microwave versus Supercritical Water Treatment

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Abstract: Hydrochar, a carbonaceous material produced through hydrothermal carbonization of lignocellulosic biomass, has gained significant attention due to its versatile applications in agriculture, energy, and environmental protection. This review extensively explores hydrochar production by hydrothermal carbonization, specifically microwave and supercritical water treatment. These innovative approaches hold substantial promises in enhancing the efficiency and sustainability of hydrochar synthesis. The review commences with an in-depth analysis of the fundamental principles governing hydrochar production, emphasizing the distinct mechanisms of microwave and supercritical water treatment. Insightful discussions on the influence of critical process parameters, such as temperature, pressure, and residence time, underscore these factors' pivotal role in tailoring hydrochar characteristics. Drawing on a wide array of research findings, the review evaluates the impact of different lignocellulosic biomass feedstocks on hydrochar properties, which is crucial for optimizing hydrochar production. The comparative assessment of microwave and supercritical water treatment sheds light on their unique advantages and challenges, guiding researchers toward informed decision-making in selection of methods. Furthermore, the review delves into the myriad applications of hydrochar, spanning soil amendment, carbon sequestration, and renewable energy. Environmental considerations and life cycle assessments associated with microwave and supercritical water treatment are also explored, providing a holistic perspective on the sustainability of hydrochar production. In conclusion, this comprehensive review synthesizes current knowledge on hydrochar production from diverse lignocellulosic biomass sources, emphasizing the efficacy of microwave and supercritical water methods.

Keywords: hydrochar; carbonization; lignocellulosic; biomass; microwave; supercritical water



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1. Introduction

Humanity's most significant concern is environmental pollution, which is the primary impediment to human health [1]. The world's main causes of environmental pollution are human activities such as mining, exploration, industrialization, urbanization, etc. Traditional treatment of waste involves either disposal in landfills or open burning as an unwanted material, resulting in negative environmental impacts such as greenhouse gas (GHG) emissions and air pollution [1,2]. Environmental pollution generated by solid waste has become a massive concern worldwide as the amount of waste being released has become overwhelming [3]. The increase in the disposal of municipal solid waste and many miscellaneous wastes from industry in different countries has posed environmental problems [4,5]. Environmental pollution may not directly affect the economy but adversely affects the human population, especially in the health sector. Environmental conservation and limiting the production of waste materials by recycling them into useful products can never be over-emphasized [4].

Although awareness and tougher legislation in developed countries have helped to safeguard their environment to a greater extent, both developed and developing countries

share the burden of GHG emissions and air pollution, which have a significant influence globally because of their serious long-term effects, even with increased attention from around the world [5,6]. Alternatively, technologies are available to convert waste to renewable resources. From the environmental sustainability perspective, it becomes clear that waste conversion to renewable resources could improve the quality of life [4,7].

CO₂ is a greenhouse gas responsible for global warming and climate change. It results from combustion processes, deforestation, and industrial activities. PM includes tiny particles suspended in the air. These particles can penetrate deep into the respiratory system, causing health issues. Sources include vehicle emissions, industrial processes, and natural dust. NH₂ is a precursor to fine particulate matter (PM₂₋₅) and contributes to air pollution. It mainly comes from agricultural activities, livestock waste, and industrial processes. CO is a colorless, odorless gas produced during incomplete combustion [7]. It poses health risks by reducing oxygen-carrying capacity in the blood. PM₂₋₅ comprises ultrafine particles that penetrate the lungs deep. It is associated with respiratory diseases, cardiovascular problems, and premature death. TC represents the total carbon content, while EC refers to the black carbon component. Both impact air quality and climate change. NO_x includes nitrogen dioxide (NO₂) and nitric oxide (NO). They result from combustion processes and contribute to smog formation and respiratory issues. VOCs are emitted from various sources (e.g., vehicles, industrial processes, solvents). They contribute to ground-level ozone formation and air pollution. CH₄ is a potent greenhouse gas primarily associated with livestock, agriculture, and fossil fuel extraction. SO₂ results from burning fossil fuels (coal, oil). It causes acid rain, respiratory issues, and damages ecosystems. BC (soot) absorbs sunlight, contributing to warming. OC includes organic compounds from combustion and biomass burning [3].

The recycling of biomass as high-value-added products, such as biofuels, bioproducts, and carbonaceous materials [8–12], has received increasing attention for biomass valorization [13,14]. For instance, the elevation in agricultural and kitchen waste has endangered the lives of livestock and plants. Researchers have worked on the biodegradation of food waste, such as citrus peel and kitchen waste, to produce important products such as biogas and ethanol [15]. Several research studies have been carried out in various ways to reduce the pollutant impacts and guarantee a healthier environment, such as bioconversion of waste to glucose and waste citrus and sweet potato peel to biodiesel [15,16].

The transformation of biomass to carbonaceous materials has recently attracted increasing attention owing to the renewable nature and associated carbon sequestration. Various applications of biomass-derived carbonaceous materials are also currently sought because of their potential cost-effectiveness, large-scale implementation, and quality-controllable production in an eco-friendly manner. The transformation of biomass to carbon nanomaterials, such as hydrochars, biochars, activated carbon, carbon fibers, etc., exhibits attractive potential for biomass valorization [16].

In recent years, the production of hydrochar, a carbon-rich material derived from the hydrothermal carbonization of lignocellulosic biomass, has emerged as a key area of interest in sustainable energy and waste management research [17]. Hydrochar exhibits versatile applications, ranging from soil amendment to carbon sequestration, making it an environmentally significant product [18]. The conventional methods of hydrothermal carbonization have been extended and refined through the application of microwave and supercritical water treatment technologies, each offering distinct advantages in reaction kinetics, product yield, and energy efficiency [19]. By exploring the literature on hydrochar production, this review seeks to elucidate the fundamental principles underlying these methods and their impact on the properties of the resulting hydrochar. Understanding the interplay of critical parameters, such as temperature, pressure, and residence time, is imperative for optimizing the production process and tailoring hydrochar characteristics [20].

This review is structured to encompass the current knowledge on hydrochar production from various lignocellulosic biomass feedstocks, including agricultural residues, forestry waste, and energy crops [21]. Incorporating microwave and supercritical water

treatment into the discussion aims to highlight the advancements and challenges associated with these innovative approaches. This review aims to provide a valuable resource for researchers and guide future endeavors toward sustainable and innovative methodologies. The synthesis of theoretical insights, practical considerations, and references to key studies in the field makes this review a comprehensive reference for those studying hydrochar production from lignocellulosic biomass.

2. Biomass and Biowaste: Sources and Composition

Biomass is the organic matter from living plants that can be naturally replenished or renewed [22]. Biowaste can be converted into valuable resources with high efficiency at low costs, saving money and conserving natural resources [23], which results in usable forms of energy and high-value products [24]. Biomass is an organic, renewable, and sustainable carbon-rich material, generally composed of plants with complex natural polymers such as hemicellulose (15–30 wt%), lignin (16–33 wt%), cellulose (40–50 wt%), and other extractive materials (1–10 wt%), which function as structural organizing ingredients [25,26]. Cellulose is a major biomass component and can be derived from biomass in sustainable and renewable ways. The extracted cellulose is biodegradable, carbon neutral, and environmentally safe [27–29]. Cellulose is the primary component of plant cell walls and comprises long chains of glucose linked by (b-1,4)-glucosidic bonds. These chains are arranged in bundles associated with hydrogen bonds, forming a primary component of paper products. Another molecule interlinks with cellulose molecules, hemicellulose [30–32], primarily composed of xylose. The structural strength of the plant and paper material is caused by lignin [33,34], which exhibits a very complex molecular structure. Newspapers contain almost 61% cellulose and 16% hemicellulose, making these materials good renewable biomass sources [35].

Concerns regarding the future availability of fossil fuels and the environmental impact of their use have spurred research for producing biofuels from biomass, considered an important renewable and sustainable energy source [36,37]. Recently, there has been renewed interest worldwide in producing biofuels from a range of biomass feedstocks [35]. Modern biorefineries and bioenergy greatly benefit from the valorization of biomass to produce various value-added biochemicals and biofuels, which helps move the industry toward climate neutrality. Hydrothermal and biological treatments are among the developed biorefining valorization procedures that have been shown to produce reasonably acceptable product yields by upgrading biorefinery intermediate products or valorizing raw biomass materials [38].

The presence of landfills gives rise to environmental concerns, primarily because they can lead to surface and groundwater contamination through leachate [39,40]. Additionally, landfills generate GHG emissions during the decomposition of organic waste, which poses risks to air quality and human well-being and plays a role in exacerbating climate change. Nevertheless, biomass has served as a fuel source for centuries. While its combustion can result in the emissions of pollutants, notably GHGs, with CO₂ being the predominant component (as illustrated in Figure 1), the potential exists for its transformation into biofuels via thermochemical methods. These processes present a more environmentally sustainable substitute for fossil fuels [41–43].

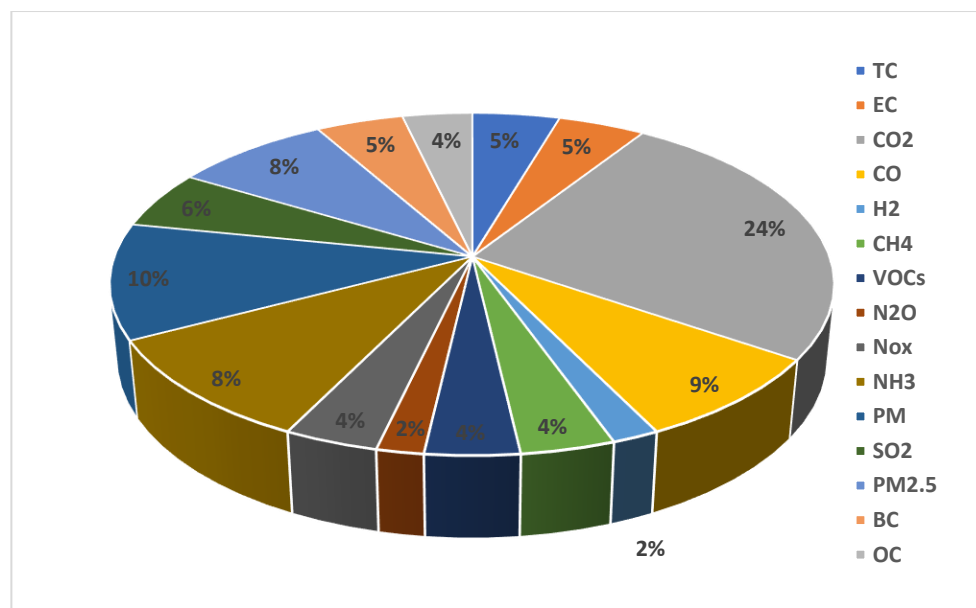


Figure 1. Contributions of biomass burning emissions [13,21].

Legend:

- CO₂—Carbon Dioxide
- PM—Particulate Matter
- NH₃—Ammonia
- CO—Carbon Monoxide
- PM₂—Fine Particulate Matter
- TC—Total Carbon
- EC—Elemental Carbon
- Nox—Nitrogen Oxides
- VOCs—Volatile Organic Compounds
- CH₄—Methane
- SO₂—Sulfur Dioxide
- BC—Black Carbon
- OC—Organic Carbon [7].

From the perspective of both green chemistry and circular economy, whether the interdisciplinary approach could achieve complete biomass valorization with balanced energy and extra profits remains questionable [44]. Moreover, managing biomass waste sustainably is crucial due to its significant environmental and economic impacts. The burning or disposing waste in fields or landfills is inefficient and causes severe environmental pollution. Thus, finding sustainable ways to manage biowaste is gaining interest [24,41].

2.1. Lignocellulose

Lignocellulose is a complex structural component in the cell walls of plants, particularly in woody tissues. It comprises three main polymers: cellulose, hemicellulose, and lignin. These polymers provide structural support to the plant cell walls and play crucial roles in plant tissues' mechanical strength and rigidity [45–47].

Cellulose: Cellulose is a complex carbohydrate and the most abundant organic compound on Earth, primarily found in the cell walls of plants. Structurally, it consists of long chains of glucose molecules linked together by β -1,4 glycosidic bonds [48]. This linear polymer arrangement's robust, fibrous structures support plant cells and provide stiffness. Cellulose serves crucial roles in the plant kingdom, including maintaining cell shape, facilitating water uptake, and contributing to plant growth [49]. Cellulose is renowned for its remarkable physical properties, such as high tensile strength, insolubility in water,

and resistance to degradation by most enzymes. These properties make cellulose essential in various industries, including paper and textile manufacturing, food production, pharmaceuticals, and biofuel production. Furthermore, cellulose holds significant promise in sustainable biotechnology and renewable energy sectors due to its abundance, biodegradability, and potential for conversion into biofuels and other valuable products through enzymatic hydrolysis and fermentation processes [50,51].

Hemicellulose: Hemicellulose is a heterogeneous polysaccharide comprising various sugar monomers such as xylose, glucose, mannose, and arabinose [52,53]. Unlike cellulose, hemicellulose has a branched structure and exhibits greater variability in composition among different plant species. It interacts with cellulose and lignin to form a cross-linked network within the cell wall, contributing to its mechanical strength and flexibility [54].

Lignin: Plant cells are supported and given stiffness by this linear polymer arrangement's robust, fibrous structures. It comprises three main monolignols: coniferyl alcohol, sinapyl alcohol, and p-coumaryl alcohol, which undergo oxidative coupling reactions to form a complex three-dimensional polymer network. Lignin acts as a binder between cellulose and hemicellulose fibers, conferring structural integrity and resistance to degradation by microbial enzymes [55].

Lignocellulosic biomass, derived from various plant sources such as wood, agricultural residues, and dedicated energy crops, is a promising renewable feedstock for producing biofuels, bioproducts, and bio-based materials. The efficient utilization of lignocellulosic biomass relies on depolymerizing cellulose, hemicellulose, and lignin into fermentable sugars or platform chemicals through biochemical or thermochemical conversion processes [56].

Alga microbial lipids extracted from algae have become an alternative source for biodiesel and oleochemical industries. These lipids are particularly attractive due to their high quality, shorter cultivation time, independence from geographical or climatological conditions, lower space requirements, and non-competitiveness with food supply [57]. Algae can accumulate lipids, which can be converted into biodiesel through processes like transesterification. These lipids serve as a renewable energy source and reduce dependence on fossil fuels. However, large-scale lipid production remains limited due to high processing costs. Researchers are exploring strategies to enhance lipid synthesis and reduce production expenses [58].

Protein from Food Waste: Microorganisms can utilize inexpensive feedstock and wastes (such as food waste) as carbon and energy sources for growth. They produce biomass, protein concentrate, or amino acids. Microbial protein contains high protein content (about 60–82% of dry cell weight) and fats, carbohydrates, nucleic acids, vitamins, and minerals like potassium and phosphorus. When these components are combined, they can contribute to various processes, including biofuel production, waste reduction, and sustainable energy generation. Researchers continue to explore ways to optimize these processes and minimize environmental impact [58,59].

2.2. Biomass Sources

Various biomass feedstocks have been explored for hydrochar production [60], including lignocellulosic, algal, and waste. Specifically, wood chips, sawdust, municipal solid waste, and agricultural residues are commonly used lignocellulosic biomass. Their composition affects hydrochar properties, such as surface area and porosity [61]. Algae-based hydrochar shows promise as a renewable energy source, while algal species, growth conditions, and harvesting methods impact hydrochar quality [62]. Organic waste (e.g., food waste, sewage sludge) can be converted into hydrochar [63]. The waste-to-energy strategies benefit waste management and hydrochar production [63].

2.3. Biomass Types and Conversion

Biomass, a sustainable energy source, can be classified as:

- Wet Biomass: Contains more than 30 wt. % moisture. Examples include algae, sewage sludge, cattle manure, and industrial effluents.
- Dry Biomass: Contains less than 30 wt % moisture. Examples include woody, herbaceous, and agricultural biomass [63].

Hydrochar Yield and Physicochemical Characteristics

The amount of hydrochar produced depends on specific production conditions such as temperature, time, pressure, and the type of biomass used [64].

Physicochemical Properties:

- Surface Area: Hydrochar's surface area affects its reactivity and adsorption capacity.
- Porosity: Porous hydrochar provides sites for adsorption and catalysis.
- Fixed Carbon Content: Indicates the carbonaceous nature of hydrochar.
- Ash Load: The inorganic content in hydrochar.
- Nutrients and Minerals: Hydrochar may retain essential nutrients and minerals from the original biomass.
- Chemical Characteristics: Reaction temperature significantly influences hydrochar's chemical properties [65].

3. Hydrochar

Hydrochar is produced through the process of hydrothermal carbonization (HTC) or hydrothermal treatment (HTT) of biomass in the presence of water under elevated temperature and pressure conditions [66]. This process mimics the natural coal formation process but occurs at significantly shorter timescales, typically from hours to days. Hydrochar possesses a porous structure and contains a significant amount of carbon, making it a promising material for various applications, including soil amendment, energy generation, and environmental remediation [49]. During hydrothermal carbonization, biomass undergoes complex chemical reactions involving dehydration, decarboxylation, and polymerization (Figure 2). The specific mechanisms of hydrochar formation vary depending on the biomass feedstock and process conditions [67]. Biomass (i.e., agricultural residues, algae, or organic waste) is mixed with water and heated to supercritical conditions. Under these extreme conditions, the biomass is converted into hydrochar, water, and other volatile components. Hydrochar is a solid material rich in carbon, similar to biochar [67]. The general steps involved in hydrochar formation are summarized in Figure 2 [67]:

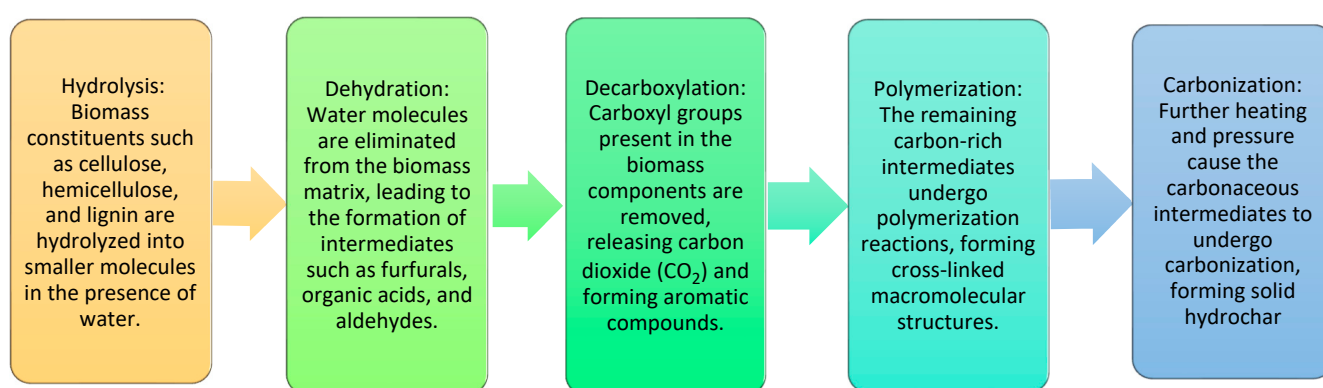


Figure 2. Production of hydrochar [53].

The properties of hydrochar, including its carbon content, surface area, porosity, and chemical composition, can be tailored by adjusting the process parameters such as temperature, pressure, reaction time, and biomass feedstock [54,66].

3.1. Properties and Applications of Hydrochar

Hydrochar properties depend on the chosen method and biomass source [55]. Typically, hydrochar contains 50–90% carbon with a porous structure that enhances its adsorption capacity. Hydrochar is also rich in oxygen-containing functional groups that promote its reactivity [1–3]. Hydrochar is used in soil amendment, energy storage, and water purification. Hydrochar offers several advantages over other carbonaceous materials such as [41]. Since hydrochar is derived from biomass, hydrochar is a renewable and sustainable carbon material [68]. Unlike fossil fuels or mined coal, hydrochar production does not deplete finite resources. Hydrochar possesses a highly porous structure with a large surface area. This porosity allows for efficient adsorption of pollutants, making it suitable for water treatment and soil improvement [68]. The properties of hydrochar can be tuned by adjusting reaction conditions during its production. Surface functional groups, pore size distribution, and thermal stability can be customized [60]. Hydrochar production occurs under mild conditions (e.g., hydrothermal carbonization) [46]. It requires less energy compared to traditional carbonization methods.

Hydrochar finds applications in various fields. For soil amendment, hydrochar improves soil structure, nutrient retention, and water-holding capacity [66]. It can also be used as a precursor for activated carbon electrodes. For water filtration, hydrochar can remove organic pollutants and heavy metals. Hydrochar can blend well with polymers to form biocomposites for sustainable materials [58]. Hydrochar has lower toxicity than other carbon materials (e.g., activated carbon derived from coal) [60]. It is less likely to leach harmful substances into the environment. Hydrochar also promotes carbon sequestration by converting biomass into a stable form [61]. Thus, hydrochar can be used for carbon capture and storage (CCS) to mitigate GHG emissions [61].

3.2. Biochar

Biochar is a highly porous carbonaceous material derived from the thermal decomposition of biomass under oxygen-limited conditions, a process known as pyrolysis. This ancient technology has gained renewed interest recently due to its potential benefits in agriculture, environmental remediation, and carbon sequestration [69]. In agriculture, biochar is a soil amendment, enhancing soil fertility, water retention, and nutrient availability [63]. Its porous structure provides a habitat for beneficial microorganisms and improves soil structure, promoting plant growth and resilience to environmental stressors. Additionally, biochar has shown promise in environmental remediation by adsorbing pollutants, such as heavy metals and organic contaminants, from soil and water. Its high surface area and strong adsorption capacity make it an effective tool for mitigating soil and water pollution, contributing to environmental sustainability [64].

Biochar has the potential to mitigate climate change by sequestering carbon in soils for centuries to millennia, thereby reducing GHG emissions and enhancing soil carbon stocks [69]. Biochar presents a multifaceted solution to various environmental challenges, offering opportunities for sustainable agriculture, pollution remediation, and climate change mitigation [70]. As research in this field continues to evolve, biochar holds promise as a versatile and eco-friendly resource for addressing pressing environmental concerns. The difference between biochar and hydrochar is summarized in Table 1.

Table 1. Differences between hydrochar and biochar.

Aspect	Biochar	Hydrochar	Reference
Definition	Biochar is a high-carbon, fine-grained residue produced via pyrolysis—the thermal decomposition of biomass without oxygen. It results in a mixture of solids (biochar), liquid (bio-oil), and gas (syngas) products.	Hydrochar is produced through hydrothermal carbonization (HTC) or liquefaction (HTL). HTC and HTL yield hydrochar, which is a distinct category of biochar. These processes involve subjecting wet biomass to high temperatures and pressure in the presence of water.	[65,66,71]
Stability and Carbon Sequestration	Biochar is a stable solid rich in pyrogenic carbon. It can endure in soil for thousands of years, making it suitable for carbon sequestration (PyCCS) and climate change mitigation.	Hydrochar also exhibits stability and can persist in soil. Its refractory nature contributes to carbon storage, although its longevity may not match biochar.	[67,72]
Composition and Properties	Biochar typically contains a mix of carbon, ash, and other elements. Its properties depend on the feedstock and pyrolysis conditions.	Hydrochar has a similar carbonaceous composition but may differ in alkali, alkaline earth, and heavy metal content. It often possesses a larger surface area and higher heating value than biochar produced at the same operating temperature.	[69]
Applications	– Used primarily for soil amendment to improve fertility, nutrient availability, and water filtration. – Applied in agriculture, forestry, and environmental remediation.	– Also used for soil improvement, similar to biochar. – Being explored for applications in water treatment, energy storage, and pollutant removal.	[70]
Environmental Considerations:	Biochar can alter soil pH and introduce chemical characteristics that impact microorganisms.	Hydrochar research continues to explore its benefits and drawbacks.	[71,72]

4. Biomass Conversion

4.1. Methods

Addressing the need for a sustainable resolution to manage the increasing volume of waste generated by the agri-food industry has been a key research focus for scholars over recent decades [73]. Waste management and conversion are becoming increasingly popular due to biowaste’s negative impact on the economy, the environment, and human health. However, recent research has shown that biomass waste can be converted into valuable resources with high efficiency and low cost, saving money and conserving natural resources [74].

Biomass can be converted into several useful forms of energy and biochemicals using different processes and technologies [10–18]. Broadly, biomass conversion technologies fall into biochemical and thermochemical categories [74]. The selection of a specific conversion technology is influenced by several variables, including the type of feedstock and its moisture content, as well as the quality and quantity of biomass feedstock, its availability, the desired end products, economic considerations such as profitability and market accessibility, and environmental considerations [75]. Water content is a very important criterion for technology selection when used for energy [76]. Therefore, high-moisture biomass would not be appropriate for technologies that require prior drying but would be suitable for technologies that benefit from water content.

4.2. Hydrothermal Technology

The word “hydrothermal” was first introduced by a British geologist, Sir Roderick Murchison, in the 1840s [76]. Hydrothermal techniques have been found in many branches

and applications. Based on the fundamental concept, hydrothermal conversion is the process that transforms waste biomass into valuable products (solids, liquids, or gases) in the presence of water, solvent, or catalyst at a medium temperature of above 100 °C [77]. HTC mimics the natural process of coal formation (known as “Inkohlung”) but occurs within hours rather than geological periods [78].

Hydrothermal technologies generally refer to physical and chemical transformations in high-pressure (5–40 MPa), high-temperature (200–600 °C) liquid, or supercritical water [79]. Since there is no phase change to steam when heating water to high pressures, there are no significant enthalpic energy penalties when using this thermochemical method of reforming biomass. Numerous processes, such as those involving dehydration and decarboxylation, occur in biological compounds, which are influenced by the temperature, pressure, concentration, and presence of homogeneous or heterogeneous catalysts. Liquefaction processes are generally lower temperature (200–400 °C) reactions that produce liquid products, often called “bio-oil” or “bio-crude”. Gasification operations can yield large amounts of hydrogen or methane gasses, typically between 400 and 700 °C [80].

With the benefits of both approaches combined, the combination of hydrothermal and biological techniques can, therefore, be promising for biomass valorization, helping to overcome the diverse and resistant nature of biomass for particular platform biochemical/biofuel production [81].

4.3. Hydrothermal Carbonization (HTC)

HTC is a thermochemical conversion process that transforms biomass into hydrochar in the presence of water in a sealed reactor at elevated temperature and pressure. The temperature typically ranges from 356 °F to 482 °F (180 °C to 250 °C) [82]. The process occurs under autogenous pressure, automatically generating the system’s pressure. The residence time of the feedstock in the reactor varies from 0.5 to 8 h [83]. The process involves decomposing and recombining biomass components to form a carbonaceous material known as hydrochar. HTC offers several advantages, including utilizing a wide range of biomass feedstocks, producing a carbon-rich material, and potential applications in waste management, soil improvement, and energy production. Reactions occur in an isolated air environment, forming hydrochar [84] (Figure 3).

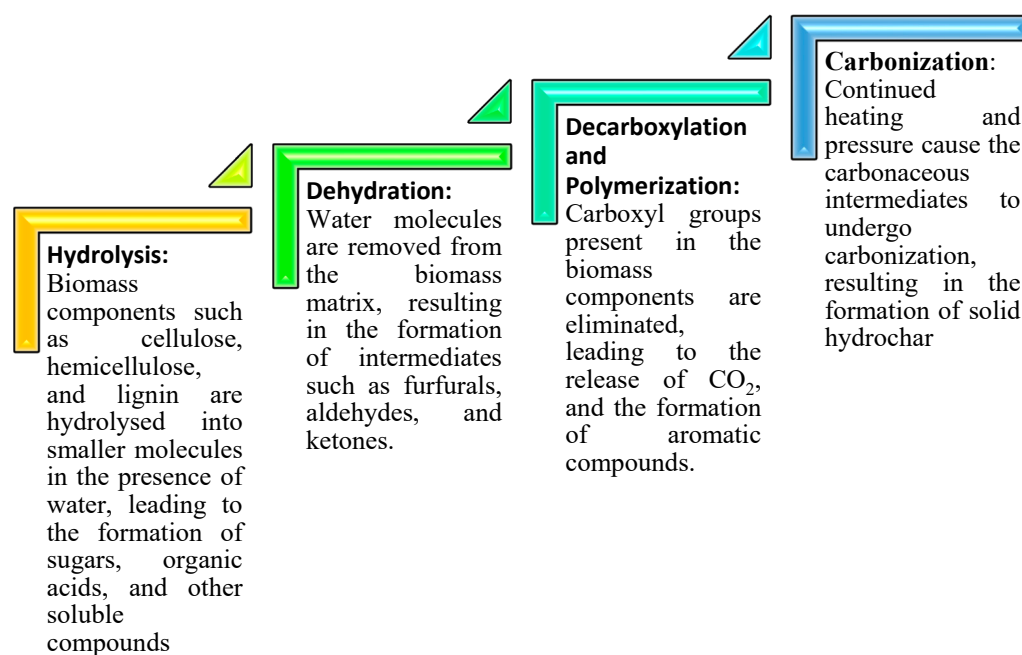


Figure 3. Flow diagram for HTC method [84,85].

4.4. Microwave-Assisted Hydrothermal Carbonization (MHTC)

MHTC is an innovative approach to the hydrothermal carbonization (HTC) process, which involves the conversion of biomass into carbonaceous materials under high temperature and pressure conditions in the presence of water. This method utilizes microwave irradiation to enhance the HTC process, leading to faster reaction rates, higher yields, and potentially improved product properties compared to conventional HTC methods. Key factors affecting MHTC include biomass type, moisture content, reaction time, temperature, and microwave power (irradiation) [86]. The MHTC process typically involves subjecting biomass feedstock to microwave irradiation in a pressurized water reactor. The combination of microwave energy and hydrothermal conditions accelerates biomass's decomposition and carbonization, producing hydrochar or biochar, a carbon-rich material [87]. Several studies have investigated the MHTC process and its potential applications in biomass conversion and waste management. For instance, a study evaluated the MHTC of sewage sludge and found that microwave irradiation significantly enhanced the production of hydrochar compared to conventional HTC, with improved carbon yield and properties of the resulting hydrochar [88–90]. This is particularly relevant as the demand for sustainable and efficient waste management solutions grows. The MHTC process offers a promising avenue for converting organic waste into valuable products like hydrochar, which can be used as a soil amendment, energy storage, or water treatment adsorbent. Moreover, the process contributes to the circular economy by transforming waste biomass into bioenergy and other byproducts, reducing reliance on fossil fuels, and mitigating environmental pollution. The versatility of the MHTC process is further highlighted by its ability to process a variety of feedstocks, including agricultural residues, municipal solid waste, and industrial byproducts. As such, the MHTC process addresses waste management challenges and offers a pathway to renewable energy and sustainable agricultural practices [91].

Furthermore, research by [92] investigated the MHTC of lignocellulosic biomass for biofuel production. It demonstrated that microwave irradiation effectively promoted biomass conversion into biochar and bio-oil, leading to higher energy yields and shorter reaction time than conventional HTC methods. Another study explored the MHTC of agricultural waste for environmental remediation applications and found that microwave irradiation facilitated the production of hydrochar with enhanced adsorption properties for removing heavy metals from aqueous solutions [93,94]. These studies collectively highlight the potential of microwave-assisted hydrothermal carbonization as a promising technology for biomass conversion, waste valorization, and environmental remediation. By leveraging microwave energy, this process offers advantages such as faster reaction kinetics, higher product yields, and tailored properties of the resulting carbonaceous materials, thus contributing to sustainable resource utilization and waste management strategies.

4.5. Supercritical Water Treatment (SCWT)

SCWT is an advanced thermochemical conversion method that involves biomass treatment in supercritical water (above its critical point), i.e., 374 °C (705 °F) and 22.1 MPa (3200 psi), where it exhibits unique properties such as high density, low viscosity, and enhanced solubility for organic compounds. The use of supercritical water offers several advantages for hydrochar production, including rapid reaction rates, efficient biomass conversion, and the potential for high-quality hydrochar products. High temperatures and pressure lead to efficient gasification and hydrochar formation. Catalysts are crucial in SCWT, influencing product distribution since they accelerate chemical reactions, promoting feedstock conversion into desired products while influencing the type and quantity of products formed [95,96]. It is a fascinating thermochemical process that transforms wet biomass into a valuable carbonaceous material known as hydrochar. Supercritical water refers to water at conditions beyond its critical point (critical temperature and pressure). When exposed to supercritical water, biomass undergoes rapid decomposition and carbonization (Figure 4) [97–100]. Table 2 summarizes the challenges that supercritical water hydrochar production faces, which researchers are actively addressing.

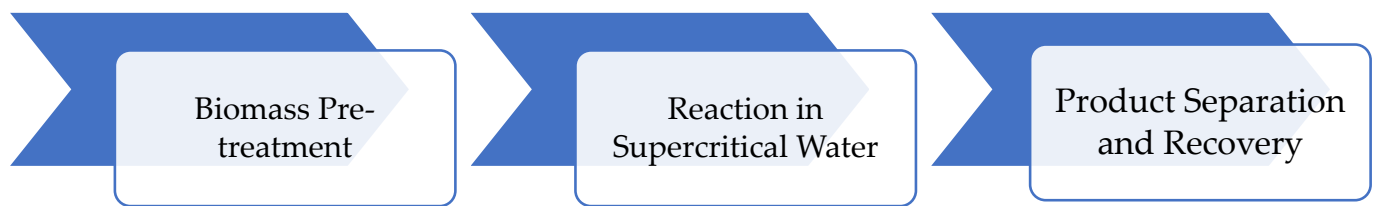


Figure 4. Supercritical water treatment for hydrochar production [101–104].

Table 2. Challenges faced by supercritical water hydrochar production.

S/N	Challenges	Issue	Impact	Reference
1	Low Yield	The yield of hydrochar obtained from supercritical water conditions is relatively low.	This affects the overall efficiency of the process and limits the effective utilization of valuable biomass resources.	[105–107]
2	Gaseous Product Yield	The gaseous products generated during supercritical water hydrochar production often have low selectivity for hydrogen gas.	Inefficient utilization of the biomass feedstock and suboptimal conversion efficiencies.	[108]
3	Energy Consumption	The process requires high-energy input due to the extreme conditions (temperature and pressure) needed for supercritical water.	Increased operational costs and environmental footprint.	[109]
4	Ash Content and Composition	Hydrochar may contain ash from the biomass feedstock.	Ash content affects the quality of hydrochar and suitability for various applications, such as agricultural applications, energy production, and carbon sequestration.	[110]
5	Reaction Mechanisms and Kinetics	Understanding the complex reactions during supercritical water hydrochar production is challenging.	Lack of detailed knowledge hinders process optimization and control.	[111]
6	Resource Recycling and Valorization	Efficiently reusing and valorizing post-processing water and byproducts.	Resource management can enhance economic viability and environmental sustainability.	[112]
7.	Scaling Up	Transitioning from lab-scale experiments to large-scale production.	Impact: Ensuring consistent product quality, safety, and cost-effectiveness.	[112]
8.	Functionalization and Applications	Tailoring hydrochar properties for specific applications.	Unlocking its potential in agriculture, pollutant adsorption, catalyst support, bioenergy, and carbon sequestration.	[4,113]

MHTC and SCWT are promising for hydrochar production [114–117]. The best method varies based on factors such as feedstock availability, yield, quality, energy constraints, and desired hydrochar properties. Researchers will continue to explore and optimize these methods for sustainable hydrochar production. The similarities between the two methods are that they both produce hydrochar from biomass such as agricultural waste, algae, or organic matter. Both hydrochars find applications in soil improvement, energy storage, and pollutant adsorption. While MHTC offers speed and energy efficiency advantages, supercritical water hydrochar production remains a reliable and widely used process for sustainable hydrochar production. Table 3 compares the differences between MHTC and SCWT for hydrochar production. The advantages and disadvantages of MHTC and SCWT for hydrochar production from biomass are summarized in Table 4.

Table 3. The differences between supercritical water (SCWT) and microwave-assisted hydrothermal carbonization (MHTC) for hydrochar production.

S/N	Aspect	Supercritical Water Treatment	Microwave-Assisted Hydrothermal Carbonization	References
1	Process	Hydrothermal conversion using supercritical water.	Biomass conversion using microwave heating.	[118–120]
2	Temperature	Requires supercritical water conditions (high temperature and pressure).	Higher temperatures due to microwave energy absorption.	[121–124]
3	Reaction Rate	Slower due to hydrothermal process (due to conventional heating).	Faster due to selective, fast, and homogeneous heating.	[123,125]
4	Product Characteristics	– More aliphatic structures. – Lower thermal stability.	– Varies based on biomass type—may yield bio-oil or valuable chemicals.	[124–127]
5	Energy Efficiency	– Less energy efficient – Requires high energy input due to extreme conditions.	– Energy efficient due to microwave heating, reduced energy consumption.	[126–129]
6	Uniform Heating	– May have temperature gradients affecting product distribution.	– Provides uniform heating throughout the reactor. – Minimizes temperature gradients.	[130]
7	Scale-Up and Industrial Applications:	Also, scalable but may require more space and time.	– Scalable to larger reactors. – Suitable for industrial-level production.	[130]
8.	Applications	Agriculture, pollutant adsorption, bioenergy.	Energy, pharmaceuticals, and chemistry sectors.	[131,132]

Table 4. The advantages and disadvantages of supercritical water hydrochar production and the microwave-assisted method for biomass conversion.

Aspect	Supercritical Water Treatment Method	Microwave-Assisted Hydrothermal Carbonization	Reference
Advantages	– Utilizes wet biomass without pre-drying. – Produces hydrochar with lesser heavy metals. – Combines microwave heating benefits with sustainable hydrothermal valorization.	– Energy efficient: Selective, fast, and homogeneous heating. – Yields bio-oil and valuable chemicals. – Shorter processing time and reduced costs.	[133,134]
Disadvantages	– Lower thermal stability of hydrochar compared to pyrolytic biochar. – Slower reaction rate due to hydrothermal process.	– Higher temperatures due to microwave energy absorption. – Varied product characteristics based on biomass type	[134–138]

Prior publications contribute significantly to understanding hydrochar production processes, highlighting the advancements, challenges, and potential research directions in the field (Table 5). However, there remain gaps in terms of optimization, scalability, environmental impact assessment, and integration with other biorefinery processes, which warrant further investigation to facilitate the widespread adoption of hydrochar as a sustainable carbonaceous material. Future research should address these gaps to unlock the full potential of hydrothermal carbonization as a sustainable biomass conversion technology.

Table 5. Hydrochar production using HTC process.

Biomass Source	Temperature (°C)/Reaction Time (Hour)	High Heat Value (HHV) (MJ/kg)	Hydrochar Yield %	Reference
Rice husk	300/6	16–18	65–67	[135,137]
Algae	190–210/2	20–25	25–46	[136,139]
Sewage sludge	200–250/5	20–23	60–65	[137]
Corn cob residue	245–250/0.5	20–25	45–50	[138]
Orange peel	180–200/20	-----	37	[139]
Water hyacinth	220–240/0.5	17–20	61	[140]
Palm shell	180–200/0.5	26–27	45–65	[141]
Bamboo shoot shell	200–210/0.5	16–17	56	[142]
Switchgrass	300–400/1–2	18–22	32–82	[142]
Mix wood	215–295/1	20–232	70–50	[143]
Sawdust	240–250/2	20–23	40	[143]
Cattle manure compost	400–900/3	17–20	50	[144]
Food waste	40–250/3	20–23	47	[145]
Paper	240–250/20	30–32	29–30	[146]
Mixed municipal solid waste	240–250/20	23–25	74	[146]

Table 5 highlights the versatility of the HTC process in handling various types of biomass, each with its unique set of process conditions and outcomes in terms of energy content and hydrochar yield. The data underscore the potential of HTC as a sustainable waste management and biomass conversion technology.

- **Temperature and Reaction Time:** The HTC process is conducted at a range of temperatures from as low as 40 °C for food waste to as high as 900 °C for cattle manure compost. The reaction times vary from 0.5 h for materials like corn cob residue to 20 h for orange peel and mixed municipal solid waste.
- **High Heat Value (HHV):** The HHV, which indicates the energy content, varies widely. For instance, algae and corn cob residue have a higher HHV of 20–25 MJ/kg, suggesting a higher energy potential, while bamboo shoot shell has a lower HHV of 16–17 MJ/kg.
- **Hydrochar Yield:** The yield percentage indicates the efficiency of the conversion process. Switchgrass shows a wide yield range of 32–82%, possibly due to variations in process conditions or biomass properties. In contrast, rice husk and sewage sludge have more consistent yields of 65–67% and 60–65%, respectively [144,146].

Table 6 provides a concise overview of previously published manuscripts on the production process of hydrochar, highlighting their perspectives and identifying existing gaps.

Table 6. Previously published manuscripts on the production process of hydrochar, highlighting their perspectives and gaps.

Article	Perspectives	Gaps	Summary	Reference
1 Hydrochar: A Review on Its Production Technologies and Applications. <i>Catalysts</i> , 11(8), 939.	Comprehensive Overview: The review offers a comprehensive overview of hydrochar, covering its characteristics, production mechanisms, and activation methods. – Multidisciplinary Approach: By discussing applications in agriculture, pollutant adsorption, catalyst support, bioenergy, carbon sequestration, and electrochemistry, the paper highlights the multidisciplinary nature of hydrochar research. – Emerging Trends: It likely identifies emerging trends and areas where hydrochar can make significant contributions.	– Economic Viability: While the review covers applications, it does not delve deeply into the economic feasibility of large-scale hydrochar production. – Environmental Effects: The environmental impact of hydrochar use (e.g., soil effects, greenhouse gas emissions) could be explored further. – Standardization: Standardization of hydrochar production methods and quality assessment protocols remains an area for improvement.	This paper provides a valuable foundation for understanding hydrochar, but further research is needed to address gaps and optimize its practical implementation.	[147]
2. A bibliographic study reviewing the last decade of hydrochar in environmental application: history, status quo, and trending research paths. <i>Biochar</i> 5, 12 (2023).	– Holistic Assessment: The study provides a comprehensive assessment of hydrochar research over the last decade, covering various environmental applications. – Identification of Key Topics: By analyzing research trends, the study identifies critical topics related to soil quality, plant growth, carbon capture, greenhouse gas emissions, organic pollutant removal, and heavy metal adsorption. – Awareness of Research Trajectories: Researchers and policymakers can gain insights into the trajectory of hydrochar research, allowing them to align efforts with emerging trends.	– Quantitative Synthesis: While the study identifies research topics, it may lack a quantitative synthesis (e.g., meta-analysis) of findings across studies. – Long-Term Effects: Further investigation into the long-term effects of hydrochar application on soil health, plant growth, and ecosystem dynamics is needed. – Standardization and Guidelines: The absence of standardized protocols and guidelines for hydrochar application hinders its widespread adoption. – Economic Viability: The study could explore economic feasibility and cost-effectiveness of hydrochar-based solutions.	This study provides valuable insights into hydrochar research trends, but addressing gaps related to long-term effects, standardization, and economic viability remains crucial for practical implementation.	[148]

Table 6. Cont.

Article	Perspectives	Gaps	Summary	Reference
3. Hydrochar: A Promising Step Towards Achieving a Circular Economy and Sustainable Development Goals. <i>Frontiers in Chemical Engineering</i> , 4, 867228.	– Circular Economy Potential: The article highlights hydrochar as a valuable resource within the circular economy framework. It emphasizes its role in closing material loops and reducing waste. – Sustainable Biomass Utilization: By discussing hydrochar production from various biomass sources, the article promotes sustainable utilization of organic materials. – Environmental Benefits: Hydrochar can contribute to carbon sequestration, soil improvement, and pollutant removal, aligning with circular economy principles.	– Water Remediation Potential: While the article identifies water remediation as a gap, it could delve deeper into hydrochar's effectiveness in removing contaminants from water bodies. – Soil Effects: Further research is needed to understand hydrochar's long-term impact on soil health, nutrient cycling, and microbial communities. – Greenhouse Gas Emissions: The article explores the net effect of hydrochar production on greenhouse gas emissions, considering both carbon sequestration and energy-intensive processes. – Economic Viability: Economic feasibility and cost-effectiveness of large-scale hydrochar production require more investigation.	While the article recognizes hydrochar's promise, addressing gaps related to water, soil, emissions, and economics will enhance its practical implementation.	[149]
4 Hydrochar-based soil amendments for agriculture: a review of recent progress. <i>Arabian Journal of Geoscience</i> 14, 102 (2021).	– Soil Fertility Enhancement: The review highlights hydrochar's role in improving soil fertility by providing slow-release nutrients. – Carbon Sequestration: Hydrochar contributes to carbon sequestration in soils, promoting sustainable agriculture. – Positive Effects on Soil Microbes: Hydrochar positively influences soil microbial communities, impacting nutrient cycling and plant health.	– Water-Holding Capacity: Further research is needed to understand how hydrochar affects soil water-holding capacity, considering factors like reaction temperature and particle size. – Economic Feasibility: The article could explore the economic feasibility of large-scale hydrochar production and application. – Long-Term Effects: Investigating the long-term impact of hydrochar on soil properties and crop productivity remains essential.	While hydrochar shows promise as a soil amendment, addressing gaps related to water dynamics, economics, and long-term effects will enhance its practical adoption in agriculture.	[150]

Table 6. Cont.

Article	Perspectives	Gaps	Summary	Reference
5. A Comprehensive Review on Hydrothermal Carbonization of Biomass and its Applications. <i>Chemistry Africa</i> 3, 1–19 (2020).	– Energy-Efficient Process: HTC is gaining interest due to its energy efficiency. It allows hydrochar production at lower temperatures without specific pressure requirements. – Comparative Analysis: The review differentiates between hydrochar and biochar from various sources. It evaluates maximum efficiency for hydrochar production. – Applications: The study highlights hydrochar's applications in wastewater treatment, carbon sequestration, gas adsorption, and soil amendment.	– Economic Feasibility: While the review discusses applications, it could explore the economic feasibility and cost-effectiveness of large-scale hydrochar production. – Environmental Effects: Further research is needed to understand hydrochar's impact on soil, air, and water quality. – Optimal Parameters: Investigating optimal parameters (pH, temperature, exposure duration) for hydrochar yield would enhance practical implementation.	This review sheds light on HTC's environmental remediation potential and industrial applications, but addressing gaps related to economics and optimal conditions is essential.	[151]
6. Microwave-assisted hydrothermal treatments for biomass valorization: a critical review. <i>Green Chemistry</i> , 23(10), 3502–3525.	– Synergy of Microwave and Hydrothermal Conditions: The review focuses on the unique combination of microwave energy and hydrothermal conditions for biomass conversion. – Energy-Efficient Approach: Microwave-assisted hydrothermal treatments offer energy efficiency compared to conventional methods. – Versatile Applications: The study likely discusses various applications of microwave-assisted biomass valorization, such as bio-crude production and chemical synthesis.	– Combined Effects: Further research is needed to understand the synergistic effects of microwaves and water during biomass conversion. – Optimization: Investigating optimal process parameters (e.g., microwave power, exposure time) is crucial for efficient biomass valorization. – Comparisons: The review could compare microwave-assisted hydrothermal treatments with other biomass conversion techniques.	While the review highlights the potential of microwave-assisted hydrothermal treatments, addressing gaps related to process optimization and understanding combined effects will enhance its practical application.	[152]

Table 6. Cont.

Article	Perspectives	Gaps	Summary	Reference
7. A comprehensive review on hydrothermal carbonization of biomass and its applications. <i>Chemistry Africa</i> , 3, 1–19.	This study investigates the hydrothermal carbonization of biomass in supercritical water conditions. It explores the effects of temperature, pressure, and residence time on hydrochar yield and properties. The research provides insights into the potential of supercritical water treatment for hydrochar production.	While the study offers valuable insights, gaps exist in understanding the mechanisms underlying hydrochar formation under supercritical conditions. Further research could elucidate reaction pathways and optimize process parameters to enhance hydrochar yield and quality.	This review provides an extensive overview of hydrothermal carbonization (HTC) of biomass. It covers HTC process mechanisms, hydrochar properties, and applications. The study emphasizes the potential of hydrochar in wastewater treatment, carbon sequestration, and soil improvement. However, gaps related to economic feasibility and environmental impact require further investigation. In essence, the review highlights the promise of HTC-derived hydrochar while identifying areas for future research.	[153]

5. Challenges and Future Perspectives

Producing hydrochar using the supercritical water method and microwave-assisted hydrothermal carbonization presents several challenges and holds promising future perspectives. Both supercritical water treatment and microwave-assisted methods require precise control of process parameters such as temperature, pressure, residence time, and biomass-to-water ratio. Optimizing these parameters to maximize hydrochar yield, quality, and desired properties remains a challenge, particularly on a large scale [154]. Scaling up these methods from laboratory-scale to industrial-scale production poses significant challenges. Designing and engineering reactors capable of operating under supercritical conditions or efficiently utilizing microwave energy on a large scale while maintaining process efficiency and safety is complex and requires careful consideration.

While microwave-assisted hydrothermal carbonization offers rapid heating and potentially lower energy consumption compared to conventional heating methods, optimizing energy efficiency remains crucial. Balancing energy input with desired reaction kinetics and product quality is essential for sustainable and cost-effective hydrochar production. Lignocellulosic biomass feedstocks vary in composition, structure, and properties, which can significantly impact hydrochar production processes and product characteristics. Addressing the variability in feedstock properties and understanding their influence on hydrochar quality is essential for process optimization and application development. Establishing standardized methods for characterizing hydrochar properties is critical for comparing results across studies and ensuring consistency in product quality. Developing comprehensive characterization techniques that capture key physical, chemical, and structural properties of hydrochar will facilitate its widespread application and commercialization.

Further research is needed to elucidate the underlying mechanisms of hydrochar formation and evolution during supercritical water treatment and microwave-assisted hydrothermal carbonization. Understanding reaction pathways, intermediate species, and kinetics will enable more precise process control and optimization. Advances in reactor design and engineering are essential for improving process efficiency, scalability, and safety. Developing innovative reactor configurations, materials, and heating methods tailored to the specific requirements of supercritical water treatment and microwave-assisted carbonization will drive technological advancements in hydrochar production.

Integrating hydrochar production processes with biorefinery concepts offers opportunities for valorizing byproducts, minimizing waste, and enhancing overall process efficiency. Exploring synergies between hydrochar production and other biomass conversion pathways, such as biofuels, biochemicals, and biogas, can create more sustainable and economically viable biorefinery systems. Tailoring hydrochar properties to specific application requirements, such as soil amendment, water treatment, energy storage, and carbon sequestration, will unlock new market opportunities and enhance the value proposition of hydrochar-based products. Understanding the relationships between hydrochar properties and performance in different applications is crucial for expanding its use across various sectors.

Conducting comprehensive environmental and economic assessments of hydrochar production processes is essential for evaluating their sustainability and competitiveness. Integrating life cycle assessment (LCA) and techno-economic analysis (TEA) will provide insights into the environmental impacts, resource efficiency, and cost-effectiveness of different production pathways. By addressing these challenges and exploring future perspectives, research and innovation in producing hydrochar using supercritical water treatment and microwave-assisted hydrothermal carbonization can contribute to sustainable biomass utilization, environmental stewardship, and the transition toward a circular bioeconomy.

6. Conclusions

In conclusion, this review provides valuable insights into the production processes of hydrochar, highlighting the utilization of microwave and supercritical water methods for converting lignocellulosic biomass into a carbonaceous material with diverse applications.

Through a thorough examination of existing literature, the manuscript elucidates the key factors influencing hydrochar production, including process parameters and biomass feedstock properties. The review underscores the significance of microwave-assisted and supercritical water methods in enhancing the efficiency and sustainability of hydrochar production. Microwave heating offers rapid and selective heating of biomass, facilitating the decomposition and carbonization processes. At the same time, supercritical water treatment enables efficient biomass conversion under elevated temperatures and pressures, leading to high yields of hydrochar. The comprehensive nature of this review provides researchers and practitioners in the field with a valuable resource for further exploration and development of hydrochar production technologies. However, gaps and challenges persist, including the need for a more detailed mechanistic understanding of microwave and supercritical water-assisted carbonization processes, optimization of reactor designs for scalability, assessment of economic feasibility, and exploration of novel biomass feedstocks. In light of these considerations, future research efforts should focus on addressing these gaps to advance the field of hydrochar production and utilization. By leveraging innovative approaches, interdisciplinary collaborations, and sustainable practices, the potential of hydrochar as a versatile carbonaceous material for addressing environmental challenges and supporting a circular bioeconomy can be fully realized. Overall, this reviewed serves as a foundation for further research and innovation in the field of hydrochar production from lignocellulosic biomass sources using microwave and supercritical water methods.

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