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Hydrothermal carbonization for valorization of crop residues: Advances and challenges

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ABSTRACT

Agriculture, particularly modern, high-input farming systems, constitutes the backbone of food and nutritional security globally. The disposal of agricultural waste (the byproducts and leftovers generated from crop farming and food production industries) has emerged as one of the prime challenges recently. Among these, bio-economical disposal methods for crop residues such as straw, husks, and stalks (rice straw, wheat husks, corn stalks), leaves, pruning from fruit trees and vineyards, and spoiled or damaged crops hold pertinence to reduce environmental degradation caused by their open field burning. Chemical engineering approaches such as hydrothermal carbonization (HTC) that entail dehydration and decarboxylation to enhance the carbon content of biomass, thereby increasing its calorific value, have been developed to transform waste into different useful products. This review study critically evaluates the advances in the valorization of agricultural wastes into marketable products using the HTC technique. Moreover, the limitations of the HTC technique, its prospective employment, and future research needs have been objectively highlighted.

Keywords: rice husk, biological waste, corn stover, southern states, pressure conversion, lignocellulosic biomass, pretreatment, conversion.

INTRODUCTION

Louisiana is one of the southeastern states of the U.S. and it occupies a historically and geographically important zone, where the Mississippi-Missouri river system empties into the Gulf of Mexico. The overflowing of its many rivers and bayous, resulting in the deposit of nutrient-rich sediments from all over North America into the delta region, creates very fertile alluvial soil, enabling the production of rice and maize grains. Due to these factors, Louisiana claims the title of the third largest producer of rice in the U.S (Boggs, 2024). While it is common to practice proper waste disposal during the processing of agricultural resources in Louisiana, harvesting residues are found in many of its forests and fields. These residues are a characteristic type of biomass that could be potentially converted into species that are essential in industries, by employing an appropriate treatment technique (Ahmad et al., 2023). Nevertheless, open field burning of biomass, particularly crop residues, contributes to environmental degradation and ozone depletion on a global scale, due to the emission of a substantial volume of greenhouse gases (GHGs) (Aydemir et al., 2023). However, with technological advancements, this biomass has the potential for use in amelioration of soil, providing nutrition for plants (Attia et al., 2021; Choudhary et al., 2021; Tang et al., 2023; Barbosa et al., 2024), and as a clean, renewable energy source (Vassilev and Vassileva, 2015; Álvarez-Murillo et al., 2015), as its energy is stored through photosynthesis (International Energy Agency, n.d.). It is estimated that global crop waste exceeds 170 billion metric tons annually. Among various biomass sources, corn straw, known as corn stover, stands out due to its abundance, given that corn is the most widely cultivated cereal crop across all habitable continents, along with its rapid growth, short life cycle, and low cost. Biomass is primarily the organic material derived from plants and animals in which chemical energy is stored after being sourced from the sun. It can be classified into two categories: non-lignocellulosic and lignocellulosic biomass. Non-lignocellulosic biomass typically includes animal manure and sewage sludge, and is primarily stored in proteins, fatty acids, and small quantities of hemicellulose, cellulose, and lignin (Atta-Obeng et al., 2017). In contrast, lignocellulosic biomass primarily contains cellulose, hemicellulose, and lignin, with a minor concentration of inorganic content and water (Carrasco et al., 2020).

This literature review has synthesized recent advances in the utilization of crop waste into marketable products by employing the hydrothermal carbonization technique. Moreover, the pronounced limitations of this technique have been objectively highlighted along with its prospective applications, while future research needs to improve the HTC technique have been concisely elaborated.

HYDROTHERMAL CARBONIZATION

Hydrothermal carbonization (HTC) entails integration of the dehydration and decarboxylation processes for enhancing biomass's carbon content (Ameen et al., 2022; Maeda and Ueda, 2025), thereby increasing its calorific value (Heidari et al., 2019). This approach effectively converts crop waste and other organic materials (such as rice husk), into hydrochar (carbon-rich solids) (Kambo and Dutta, 2015; Fang et al., 2018) by applying heat and pressure, in aqueous media (Nakason et al., 2018).

Recently, HTC has emerged as one of the most useful thermo-chemical conversion technologies, having the potential for adoption on a large scale (Lee et al., 2022). This technique particularly stands out owing to its ability to process and convert biomass having remarkably higher moisture content. The resultant product of this technique is a carbon-rich product, known as hydrochar (Kang et al., 2012; Li et al., 2021), along with water, which offers a range of valuable and economically pertinent applications across sectors such as energy, materials, agriculture, and chemical synthesis industry. Moreover, hydrochar can be applied to soils directly as an organic soil amendment for improving soil physico-chemical traits and fertility status (Diaz et al., 2019) or as bio-coal. A study investigating different percentages of biochar applied to paddy soil incubated in a soil column for 135 days, at 25 °C showed evidence of the soil organic carbon (SOC) composition shifting to a more stable, aromatic, less polar, higher molecular-weight form. This resulted from alteration of the soil microbiome, causing an increase in decomposers of labile SOC and a decrease in the numbers of aromatic degraders (Sun et al., 2020). In other research, however, it was found that although hydrochar used for soil amendment is beneficial for its yield, its long-term effects can be potentially detrimental to the soil. This is because the hydrochar is an easily degradable carbon source, leading to an increase in microbe population in the soil, disrupted nutrient cycles and heightened greenhouse gas emission, depending on the soil type and hydrochar properties (Kravchenko et al., 2024). Interestingly, further processing of hydrochar enables the synthesis of activated carbon, heat and electricity, as well as doped catalysts, supercapacitors, and electrode materials (Tjeerdsma and Militz, 2002; Congsomjit et al., 2020; Lu et al., 2024). Figure 1 depicts a Parr reactor in which hydrothermal carbonization has taken place, and its components, which assist in maintaining temperature and pressure for biomass conversion into hydrochar.

A pressured reactor is typically used to conduct the HTC process, in which the pressure is monitored throughout the process and observed to rise with time as the reaction proceeds, in accordance with the vapor pressure. The temperature is set using a controller, and the temperature changes are monitored as the reaction proceeds.

TEMPERATURE

One of the reaction conditions the HTC process is highly sensitive to is the temperature of the reaction. The most widely used temperature range for HTC is 180–250 °C, within which the process yields different products according to the temperature reached. Previous research employing the use of HTC corroborate this notion, as demonstrated in experiments carried out on coffee silverskins yielding lower sugar concentrations in the liquid product at higher temperatures, as a result of dehydration and transformation of the monosacharides released, like arabinose and

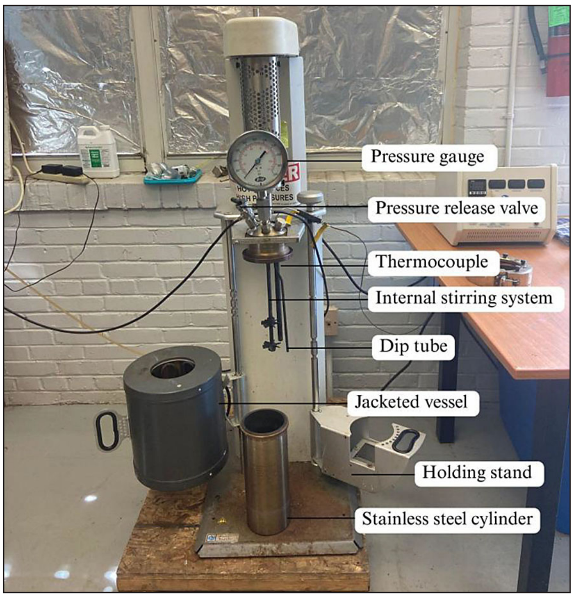

Figure 1. Parr reactor for hydrothermal carbonization (2 L) and its components for maintaining temperature and pressure

galactose, to compounds such as furfural and 5-hydroxymethylfurfural (HMF) (Sagar et al., 2023). HTC conducted on sugarcane bagasse displayed similar trends, due to degradation of sugars such as arabinose present in the liquid product at higher temperatures. With the starting material of rice husks, however, HTC experiments showed greater concentrations of the monosaccharides xylose and glucose in the liquid product at 200 °C compared to 180 °C, attributable to the recalcitrant structure of the rice husks, which only break down at higher temperatures. Replicated experiments ensured the reliability of the data supporting these findings, while the different types of biomass used make it possible to observe the nature of the relationship between product composition and temperature, which is not linear, but strongly interlinked.

Figures 2 and 3 present temperature and time variation for hydrothermal carbonization of rice husk at 250 °C for a nominal 5 min reaction in a 2 L reactor with a 10:1 water to biomass ratio, along with products formed from rice husk after hydrothermal carbonization processing.

The solid products obtained at different temperatures, 180 °C, 200 °C, and 250 °C are distinctly different in terms of appearance and texture, with the solids resembling the starting product for a 180 °C operating temperature, and becoming smaller, finer, and darker in color as the operating temperature increases to 200 °C and 250 °C (Parshetti et al., 2013). The other product of the reaction, a liquid solution is also different for varying temperatures, with not so much variation in appearance, as in intrinsic properties, such as pH. It is observed that the liquid products of HTC of rice husks carried out at 250 °C are much more acidic in comparison to the liquid products obtained from HTC at 180 °C or 200 °C. For HTC of food waste, the hydrochar yield decreased with increasing temperature for the HTC reaction, and with temperatures over 220 °C (up to 260 °C), for increasing temperatures, the higher heating value (HHV) increased as a result of decreasing ratios of H/C and O/C in the hydrochar (Cuevas et al., 2019; Abbas et al., 2024). The aromaticity in the structure of the hydrochar produced was also observed to increase with increasing temperature for HTC conducted on food waste (Abbas et al., 2024). It was observed that the HTC of paper sludge also resulted in lower H/C and O/C ratios in the hydrochar produced, with the increase of HTC temperature (Alemayehu et al., 2020). The overall change in these ratios can are depicted in a Van-Krevelen diagram, in which biomass with high ratios is observed to become hydrochar with lower ratios, closer to coal. It has been reported that the HTC temperature is responsible for the ultimate chemical composition of the products of the reaction, such as the varying forms of nitrogen, NH_4^+-N and amino-N in liquid form to NH_3 found in the gas phase at higher temperatures obtained from conducting HTC on sewage sludge and various rates of conversion achieved for varying reaction temperatures in the case of HTC of pure cellulose (Alemayehu et al., 2020). It could therefore be inferred that the temperature used in HTC is a factor that controls the reaction mechanisms to a great extent, however temperature is coupled with other factors as well.

PRESSURE

The HTC technique mimics the natural coal formation process for the most part, but occurs within a timeframe that is significantly less (Lynam et al., 2015). The process involves multiple steps, beginning with feedstock preparation, where organic waste materials (such as food waste, crop residues, and municipal solid waste) are reduced to shreds and then subjected to grinding to ensure uniform particle size. Next, the hydrothermal carbonization reaction is completed

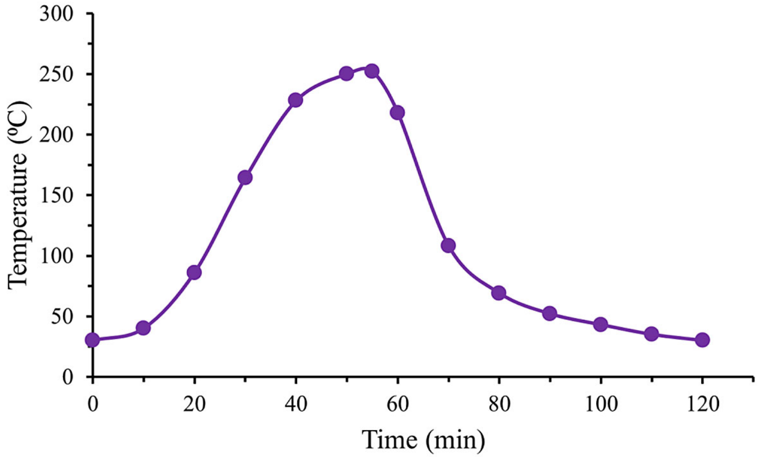

Figure 2. Temperature vs time variation for hydrothermal carbonization of rice husk at 250 °C for a nominal 5 min reaction in a 2 L reactor with a 10:1 water to biomass ratio

Figure 3. Rice husks after HTC treatment at 180 °C (left top), 200 °C (right top), and 250 °C (bottom)

in a high-pressure reactor, where the feedstock, which is unprocessed biomass, is mixed with water and subjected to elevated temperatures. The temperature range at which the reactor operates spans from 180–250 °C while maintaining the pressure sufficiently to keep water in a liquid state (Parr Instrument Company, 2018). It is observed, as the temperature increases, the pressure increases as well, reaching a maximum value, after which it starts to decrease, once the reaction is completed and quenched, eventually reaching a plateau. This trend is depicted in Figure 4.

In the HTC process, the aqueous medium is maintained below its critical point. At lower temperatures, polar ionic or reactions occur, leading to the formation of the liquid phase, while at higher temperatures, free radical reactions take place, resulting in the gas phase. Molecular reactions predominantly happen when the conditions are near the critical region of water. The next and most crucial step is carbonization, which involves chemical reactions that result in the formation of hydrochar and aqueous byproducts (Ghanim et al., 2017). Typically, the complex organic molecules in the biomass are decomposed using deionized (DI) water under high temperature and pressure. Once the chemical reaction is complete, product separation takes place, where hydrochar is extracted from the liquid byproducts (Kazimierski et al., 2021). The liquid fraction is composed of various organic compounds in a dissolved state that can be subjected to further treatment or repurposing, while the solid hydrochar can be directly used as an organic soil ameliorant or undergo additional processing (Sharma et al., 2020). Hydrothermal carbonization (HTC) is a method that successfully converts commonly found biomass – such as rice husks, corn stover, and sweet potato vines – into useful products through the application of heat (Sabzoi et al., 2019; Sabzoi et al., 2017). Despite a huge potential for waste processing, the HTC technique may perform suboptimally; thus, in order to produce products with desired traits, it requires experimentally-tested and validated optimization of key process parameters (e.g., pressure, temperature, residence time, etc.) (Sabzoi et al., 2017).

MECHANISM OF HTC TECHNIQUE

HTC entails a simple process involving submergence of crop biomass in water and then heating it at elevated temperatures ranging from 180 °C to 350 °C for varying durations (5 minutes to 4 hours), while pressure must be maintained during the process to keep the water in a liquid state (Danso-Boateng et al., 2015). Interestingly, this technique aims to reduce the oxygen and hydrogen content in the biomass by employing several chemical reactions, such as decarboxylation, dehydration, aromatization, polymerization and pyrolysis (Álvarez-Murillo et al., 2016). These reactions effectively and efficiently weaken the bonds present within the lignocellulosic structure, which leads to the release of cellulose and hemicellulose ulitmately. Additionally, during HTC reactions, cellulose undergoes hydrolysis, which results in glucose production, while hemicellulose gets broken into several hexoses and pentoses. Unlike

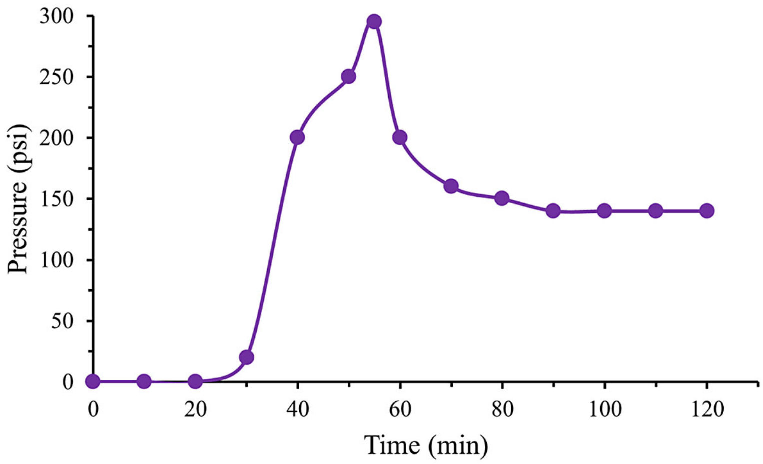

Figure 4. Pressure vs time variation for hydrothermal carbonization of rice husk at 250 °C for a 5 min run of a 2 L Parr reactor

traditional composting, which does not eliminate seeds, the high temperatures and pressures in HTC effectively inactivate all seeds, making it a more efficient biomass processing technique.

A key advantage of converting crop waste through the HTC process is obtaining hydrochar as the product, which is an organic soil amendment. Due to significantly high carbon content, hydrochar tends to enhance the build-up of soil organic matter, improve soil texture and structure, boost population and diversity of microbes and their activity, along with promoting soil restoration, preservation, and fertility (Álvarez-Murillo et al., 2016). Additionally, the process of HTC contributes in mitigating greenhouse gas emissions associated with farming, thereby playing a crucial role in carbons sequestration. Crop residues, weed biomass and agricultural waste can be transformed into hydrochar as well, preventing the harmful practice of open-field biomass burning. Hydrochar further serves as a renewable energy source for agricultural operations, being a solid fuel source (Álvarez-Murillo et al., 2016).

Moreover, conversion of crop waste into hydrochar and other valuable by-products can drastically reduce highly-determinantal open field burning of crop leftovers, thereby halting soil and ecological degradation, water contamination, and environmental pollution. Furthermore, HTC also has a wide range of broader applications, such as processing municipal waste into organics, which can be used alongside crop waste-derived soil conditioners to further enrich agricultural soils.

TIME INFLUENCE ON HTC PROCESS

Once the desired temperature for HTC has been attained, the reactants are kept at that temperature for some time to allow the reaction to take place. This time is commonly known as the "residence time" or "holding time" and can affect the ultimate product formed by the hydrothermal carbonization, to varying extents, depending on the biomass used. For instance, in a study focusing on the hydrothermal carbonization of corn cob residuals, the effect of residence time was explored and it was reported that compared to the raw material, the HHV of the corn cob residuals increased by 40%, but the effect of residence time was negligible beyond a period of 0.5 hour, after which the O/C and H/C ratios of the corn cobs showed no major change and hence, the HHV remained almost constant as well (Álvarez-Murillo et al., 2015). In another case, the effect of varying residence times was noted on hydrochar, the product of the HTC of sewage sludge. It was observed that the hydrochar formed was not significantly different in terms of its physical and chemical properties as a fuel but was obtained at a higher mass yield and increased hydrophobicity with increasing residence times (Álvarez-Murillo et al., 2016). For HTC of corn stover, longer residence times, coupled with higher HTC temperatures as well as acid addition, caused lower O/C and H/C ratios in the hydrochar formed (Aslam et al., 2022).The coupled effect of increasing the residence time and HTC temperature was also observed on rice husks and exhausted black wattle bark in another study, and it was found to have a generally decreased process yield as a result (Álvarez-Murillo et al., 2016). Collectively considering, it could be inferred that residence time is, in general, a parameter that can be optimized to obtain yields as desired; however, it is not independent from other process parameters like HTC temperature or acidity of the medium. It is common practice to do the optimization by conducting multiple experiments over a range of residence times, keeping all other conditions the same, and observing the yield and hydrochar content at the end to determine the residence time that yields a desirable product stream.

SCALABILITY OF THE HTC PROCESS AND INDUSTRIAL APPLICATIONS

The technique of HTC is evolving as of now in industrial scenarios but has a growing demand owing to its ability to resolve waste management problems with a minimum material cost. This is evident in the exponential increase in patents for HTC globally over the last few years, particularly in Germany, the United States and China (Romano, et al., 2023). While the conversion of this technology to a industrial scale system is still undergoing development in many countries, it has already been tested in pilot plants and full-scale facilities in countries such as Europe and China. The collaborative effort between Beijing Aquatic Park Co.(Beijing, China) and Terranova company for instance, which is the largest known HTC plant for sludge treatment, is also compelling evidence of its scability (Romano et al., 2023). In the U.S, HTC and its scalability for various industrial applications is a topic of research in numerous universities and research organizations. The Borough of Phoenixville Wastewater Treatment Plant, in Pennsylvania, is the first of HTC treatment facilities in the United States to have reached commercial-scale actualization, collecting county and state grants to fund its efficient technology for converting sewage sludge and food waste from neighboring facilities into electricity (Taylor et al., 2022). In certain localities in the U.S, such as southeastern Ohio, where the geography and weather conditions make maintenance problematic for home sewage treatment systems (commonly used for sewage sludge disposal), HTC is being considered for its efficacy in utilizing this form of waste. A case study conducted in Athens County, Ohio, a rural Appalachian county, confirmed the economic feasibility of a HTC treatment facility, considering two different prospective models, a break-centralized facility and a decentralized facility, both predicted to break even within 6 years (Saha, et al., 2021). In another study, a techno-economic analysis (TEA) conducted at Honda R&D Americas in Raymond, Ohio, revealed that integrating the technology of HTC to provide low cost nutrients (in the liquid product) and power (from the solid by-product), with an algal bioreactor reduced the cost of operation for the bioreactor system by 17%, costs of carbon capture by 11% and resulted in a significant improvement in its energy return on investment (EROI) (Drabold, 2020). Other than this, the scalability of the HTC treatment process has been explored widely across the globe, leading to pilot scale plants in the United Kingdom and industrial scale factories in Germany, Spain, Finland and Italy.

CHALLENGES AND FUTURE PERSPECTIVES

As with many emerging technologies, one of the primary challenges facing the widespread adoption of the HTC technique is its high capital investment requirements. Additionally, operational costs can be significant, particularly when waste transport over long distances, energy inputs, and manual labor are involved (Bevan and Zheng, 2020). HTC also faces competition from well-established biomass conversion technologies, which further constrain its market penetration. At this early stage of development, the high costs associated with HTC limit its competitiveness as a solid fuel, especially given the decreasing costs of renewable energy generated from advanced solar and wind technologies. To overcome these barriers and foster market expansion, HTC should be strategically implemented at centralized facilities that generate large high-moisture biomass in large volumes, such as tourist cruise ships, waste treatment facilities, and pulp and paper plants. Furthermore, the economic potential of hydrochar needs to be assessed to identify and formulate the most useful, feasible, and value-adding applications. This technique holds potential to ensure efficient utilization of biomass resources, support achievement of the goals of a circular economy, and enhance the viability of this technology in the long run. For instance, recently, hydrochar is being developed as a sustainable electrode material and has been applied successfully as a catalyst. While both applications of hydrochar, acting as an electrode material or as a catalyst have been extensively researched, hydrochar electrodes are closer to commercialization as of now, due to a number of factors. Biomass-based electrodes are synthesized using a substantially easier method compared to traditionally used alternatives (e.g. metals, metal oxides, and other carbon based materials); in addition, they can be made from precursors that are readily available, leading to numerous research studies being conducted to explore their usability in various electrochemical applications (Onfray et al., 2023). While offering the required sensitivity, robustness and rapid responsiveness required for these devices, hydrochar-based electrode materials are also cost-effective, which makes them highly desirable for mass commercialization. On the other hand, the use of hydrochar as a catalyst requires doping with heteroatoms or functionalization, to achieve high performance levels, thus making the commercialization of this application more difficult to achieve, prospectively (Masoumi et al., 2021).

For harnessing biomass utilization in cleaner production, HTC may serve as an innovative approach. Further in-depth research aimed at exploring the chemistry of HTC reactions, their kinetics, mechanisms of heat transfer during different phases, potential impacts of catalysts and operational parameters, strategies for energy and heat recovery, its integration with other technologies, and both economic and technical feasibility, is direly needed to control open field burning of crops left-overs (Nakason et al., 2018). On the whole, the aspects of HTC that are most imperative for consideration of rigorous research are the kinetic models for the reactions, the effect of variable feedstocks and the incorporation of transport phenomena for comprehensive process modeling. Currently, kinetic models studied are linear and oversimplified; with the key components of biomass, cellulose, hemicellulose and lignin studied in isolation, thus neglecting their collective degradation effect and intermediates formed (Dang et al., 2024). Critical heat and mass transfers in the system are also commonly overlooked while analyzing HTC processes (Dang et al., 2024). While all these considerations are integral for scaling the process to industrial operation scale, understanding of HTC in current research remains limited, indicating a significant gap in research that needs to be addressed. Moreover, scaling up HTC for industrial applications also depends on the continuous advancement of reactor design, because batch reactors have been commonly used, while a continuous-type reactor capable of operating under elevated temperatures and pressure may increase the functional efficacy of the HTC technique.

In the wake of several challenges and technical glitches occurring during the transformation of crop waste into organic amendments, the need for future research to focus on determining the economic workability of the process of HTC by performing a detailed economic analysis, grows increasingly. Such an economic analysis must include evaluations of both fixed and variable costs regarding the collection and processing of crop waste, sample preparation, and energy consumption during the HTC process. Additionally, the pricing and market value of carbon-enriched organic amendments are subject only to adjustment depending on market demand, production costs and other production-related localized factors (Heidari et al., 2019).

Further research is also required to optimize key process parameters such as reaction time, pressure, and temperature, to ensure obtaining the maximum possible yield of carbon-rich hydrochar. Achieving optimal production conditions, specific biomass is essential to enhancing the efficiency and effectiveness of the HTC process. Overall, hydrothermal carbonization presents a promising biological approach for transforming crop waste into valuable organic soil conditioners. Furthermore, by repurposing weeds through HTC, the reliance on chemical herbicides could be reduced, addressing environmental concerns related to the persistence of herbicide residues in water, plants and soil, while also addressing challenges related to waste management.

CONCLUSIONS

It may be concluded that hydrothermal carbonization (HTC) is a thermochemical process that is proven to effectively convert biomass into a carbon-rich product (hydrochar). Additionally, this technique primarily also produces a few liquid byproducts and a small amount of gaseous CO_2. One of the key advantages of the HTC technique is its capability to process biomass containing moisture directly, therefore eliminating the need for pre-drying, making it specifically suitable for feedstocks containing significantly higher moisture content (algae, sewage sludge, animal waste, etc.). In addition, this technique has emerged as promising in waste management due to waste's thermochemical treatment at lower temperatures compared to traditional thermal treatments (pyrolysis, gasification, etc.). Despite these advantages, it has become crucial to conduct further in-depth research to address technical challenges by experimentally determining the optimization of the HTC process to achieve maximum quantity and high-quality hydrochar sustainably. In the long run, HTC-based conversion of biomass could be expected to reduce herbicide usage, additionally supporting ecological restoration, providing environmental protection, and enhancing human health by minimizing toxic chemicals retention in agricultural produce. Furthermore, it can improve the economic profitability of farmers by decreasing the cost of production of different field crops while increasing the nutritional value and marketability of agricultural products.

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