

A Review on the Effect of Hydrothermal Carbonization of Agricultural Waste as a Sustainable Energy from Phosphorus and Nitrogen Recovery

**N. Jafar¹, N.A. Khan¹, S. Afzal², A. A. Khan³,
A . Ali⁴, H. M. B. Munir¹, M. Siddique¹ and M. S. Aamir¹**

¹*Department of Chemical Engineering, BUIITEMS, Quetta, Pakistan*

²*Department of Environmental Science, Faculty of Life Sciences and Informatics, BUIITEMS*

³*Department of Electrical Engineering, BUIITEMS*

⁴*Department of Mechanical Engineering, BUIITEMS*

Corresponding author: siddiqnasar786@gmail.com

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Abstract

Bio refineries are gaining popularity as population increase drives up energy and resource demands. This approach aimed to employ biomass to generate energy and recover valuable chemicals. Nitrogen (N) and Phosphorous (P) are essential nutrients for manufacturing fertilizer, which contributes significantly to global food production. Hydrothermal carbonization (HTC) is an effective energy recovery process that can also be used before treatment to improve nutrient recovery. During HTC management, some P and N dissolve in water, while the remainder stays in hydrochars (HC). HCs are commonly utilized as soil additions due to their high fertilizer and phosphate content. However, water retains significant levels of these chemicals, making it a viable source of recovery. HTC may improve the removal of nutrients by extraction (water process) or concentration (HC) of biomass, especially when combined with other processes. This paper provides a summary of both N and P pathways throughout the HTC process in terms of nutrient recovery, including current methods and future trends.

Keywords: biomass; hydrothermal carbonization; Nitrogen; nutrients; phosphorus.

Introduction*

Although nutrients in nature mostly come from animal dung, wastewater, and organic residues from human activity [1], their excessive discharge on soil and water constitutes a significant environmental concern. Nitrogen (N) and phosphorus (P) are essential components of contemporary agricultural waste and fertilizer

* The abbreviations list is in page 591.

manufacture, and their use is linked to population increase. Traditionally, manure waste is applied to farmlands, but its uneven P and N content exceeds the demands of crops and pasture fertilizers, leading to soil nutrient saturation [2]. Excess nutrients in soils are washed into water bodies by rainwater, leading to *algae* development, which reduces oxygen levels among aquatic organisms, harming the natural ecosystem by destroying plants and animals [3]. P is a nonrenewable mineral derived from rock through phosphate mining. The phosphate recovery method may enhance P and N-rich wastes, turning them into a valuable agricultural resource [4]. Researchers have proposed several scenarios for producing P and N-rich goods from a range of wastes, particularly manure. The solid fraction may be used to produce elemental P or N for commercial markets, including food and detergents, as well as a P/N-rich fertilizer for agriculture [5]. The field of investigation is under intense pressure to find technology capable of recycling nutrients, such as P, from sources of waste. These methods will reduce the environmental impact and reliance on scarce resources such as fertilizers.

Nitrogen and Phosphorus Recovery Techniques

Due to their high levels of nutrients and enormous volumes of waste, sewage sludge, animal feces, and digested estates have been the main targets for both N and P separation and recovery methods [6]. Alternative high-concentration P and N sources, comprising food debris, *algae* growth, and waste from industries, should be covered in these strategies. Table 1 describes procedures for removing and reinstating P and N.

Table 1: Strategies for removing and recovering P and N.

Method	Description	Focus	Recovery/removal	Reference
Washing method with extract	Consists of extracting P from the solid sample with an extraction solution (i.e., CaCl ₂ , Olsen solution, LiCl, etc.), considering variables like pH, solid-to-solution ratio, and extraction time	P	Not reported as a percentage	[21]
Co-combustion	Thermochemical conversion of biomass into ashes at high temperatures (850 and 1250 °C). Most of the organic P from biomass is retained in ashes in the form of P-metal consortia. P is recovered through mineral or acid extraction.	P	Up to 100%	[8]
HTC	Solubilization of N and P from biomass through high temperatures (180 and 250 °C) and pressure (5e45 bar) in the presence of water. The solid product also retains some P and N, making it suitable as a potential fertilizer.	P and N	Up to 49% P and up to 41% N solubilization	[22]

Biological denitrification and combined precipitation with chemicals are some of the main and most prevalent methods for removing P and N. However, these approaches do not support retrieval. Existing P and N recovery techniques include building materials precipitation, air stripping, and acid cleaning [7]. P recovery systems typically include precipitating phosphoric crystals from liquid wastes, resulting in struvite, hydroxyapatites, or phosphates of calcium [8]. This technique yields high-quality phosphoric minerals with high recovery rates (99-100%), suitable for direct use in agricultural land [9]. The material precipitate compounds are environmentally friendly since they contain little toxic metals [10]. Low absorption of concrete can be advantageous, since it reduces the potential of P penetration into deep water and enhances eutrophication when an adequate fertiliser is applied to the subsurface. Struvite-based P precipitation approaches only remove 12.5% N [11]. One typical technique is to recover P from biomass ashes by co-combustion. Biomass is thermostatically transformed (incinerated) at extremely hot temperatures (850-1250 °C), leaving most naturally occurring P in the dust as P-metal associations. Separating P from material or organic chemicals (such as water in the form of HNO₃, HCl, H₃PO₄, and acidic substances such as oxidizing acids) can be costly [12]. Extraction using certain acids, such as H₂SO₄, can lead to the leaching of small heavy metals. Recovery rates for this technique have been reported to range from 80 to 100%. *Algae* are a well-studied and frequently employed technique for recovering P. To treat high P and N concentrations in liquid waste, *algae* are grown in the medium to absorb these nutrients. *Algae* harvesting methods include coagulation/flocculation, centrifugation, buoyancy, and others [13]. Growing *algae* requires certain circumstances, such as installation space, temperature, and light, which can be costly and difficult to accomplish.

HTC

Despite certain limits, biomass energy is a viable alternative to fossil fuels, alongside wind, solar, and wave power. Biomass transformation involves several processes, including thermochemical, biological, and mechanical methods [14]. Thermochemical conversion of biomass, including combustion, pyrolysis, torrefaction, and hydrothermal treatments (HT- carbonisation, gasification, and liquefaction), has been extensively researched and developed globally. Table 2 shows several heating methods for biofuel conversion. Combustion processes used to generate energy from biomass may have limits that affect system performance [15], which include increased expenses for storage, transport, and production inefficiencies. Drawbacks are related to high humidity content and rapid biological breakdown. Biomass offers a low mass density, weak power requirements, a large amount of chemical-related real estate, and milling issues such as clogging and chemical degradation [16]. HT has advantages over other thermochemical therapies

(burning), because they eliminate the need for preceding drying treatment, lowering energy costs [17]. HT seeks to boost energy density by removing oxygen and carbon from materials. Characteristics of HT by-products vary depending on the process intensity (pressure, temperature, and reaction time), yielding hard HC, bio-crude, and syngas, among others. However, one of the major drawbacks is that the equipment has high setup demands (energy and installation costs) [18].

Table 2: Types of thermal processes used in biomass transformation [33].

HT	Observations	Process conditions for biomass	Pressure	Main product	References
		Temperature range			
HTC	In the presence of water. Produces mainly HC.	200 – 250 °C	1 atm	Char	[33]
HTL	In the presence of water. Produces mainly bio-crude.	280-370 °C	10-25 MPa	Bio crude	[34]
Torrefaction	Heating is low, and limited oxygen content.	270-290 °C	1 atm	Char	[34]

HTC, or wet torrefaction, occurs at temperatures from 200 to 250 °C. HTC is the process of turning high-moisture wood into carbon-based biofuel, which is petrol (mainly CO₂), and process water that contains both inorganic and organic substances [19]. It includes physicochemical processes such as hydrolysis, decarboxylation, and dehydration [20]. Fig. 1 shows that features are influenced by the extent of the process, including residence duration, temperature, and feedstock used for treatment [21].

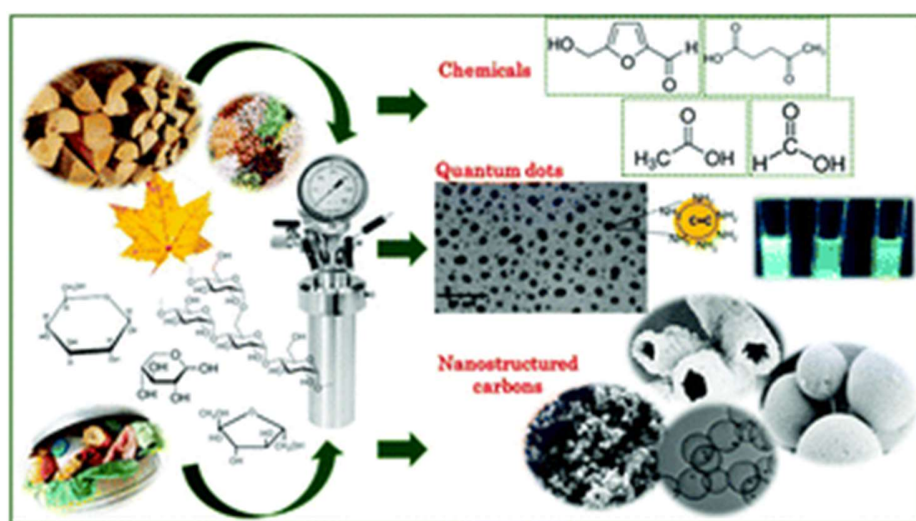


Figure 1: New developments in HT processes.

HTC process produces HC, a solid product with higher energy and mass density compared to raw biomass. Furthermore, the HTC method improves dewatering along with combustion efficiency of solid-fuel combustion [22].

Significance of HTC integration for N and P recovery

HTC by-products pose environmental challenges, including NO_x emissions from combustion and eutrophication caused by high N and P concentrations in liquid or solid wastes, which can permanently harm underground water. To solve these difficulties, new tactics must be created that combine or alter existing methods to achieve larger recuperation. The HTC procedure is one such approach. Advantages of including HTC into a nutritional recovery strategy involve: treating a wide range of waste products; obtaining useful byproducts such as HC, bio-oil, and organic materials; decreasing pathogens alongside organic waste; decreasing waste volume; and potentially recuperating P and N [23].

Some researchers have employed HTC to remove P and N from solid parts and water through processing. The HTC method efficiently converts organic N into ammonia N-containing molecules, making it a viable option for N solubilisation along with recovery [24]. While P conversion is analogous to N conversions (ranging from biological to chemical), it is not efficiently solubilised by HTC under non-acidic circumstances [25]. Increasing acidic conditions can enhance both N and P recovery from liquid fractions by promoting organic-N hydrolysis and inorganic P solubilization. HTC promotes precipitation and crystallisation, enabling P recovery in solid fractions. HTC can improve nutrient removal (the procedure fluid) with densification (HC) via biomass, resulting in higher restoration of nutrients [26]. The two tactics do not appear to be compatible with one another. Few studies have looked at the destiny of N and P before and after HTC processes [27]. HTC review papers investigate numerous aspects of biomass, including feedstock, chemical interactions, operating parameters, balance of power, product characteristics (hydrocarbons and processing water), and prospective future research. The chemical composition that makes up the HTC procedure from a design standpoint and qualitatively determined critical parameters was evaluated by [28]. They investigated the technique and chemistry of creating HC from a variety of raw materials, including biomass and other waste materials. HTC improvement was examined by [29] considering process parameters, chemical interactions, and after-product attributes in energy and agricultural production. Biofuel production from biowaste, which included sewage sludge, municipal rubbish, and unfilled palm oil fruit bunches, was explored by [30]. They used HT, such as HTC, to highlight the process's advantages and disadvantages, as well as the financial instability of solid biofuel production. The benefits of HC and the review of HTC's application in converting feedstock into activated carbon were highlighted by [31]. HTC properties

and applications were investigated, including their use as substrates in supercapacitors. Carbon spheres manufactured with HTC and the use of HTC and anaerobic digestion technologies for food waste as a biomass source were studied by [32]. They explored the generation of hydrocarbons through the HTC process using lignin-based waste products and water sewage, and assessed the current status of HT (which includes carbonisation) in biomass from *algae*.

P enzyme pathways throughout HTC

HT allows for P migration into both solid and liquid fractions, unlike dry thermochemical transformations that only affect the solid fraction. Fig. 2 summarizes the probable transformation connections for P during HTC.

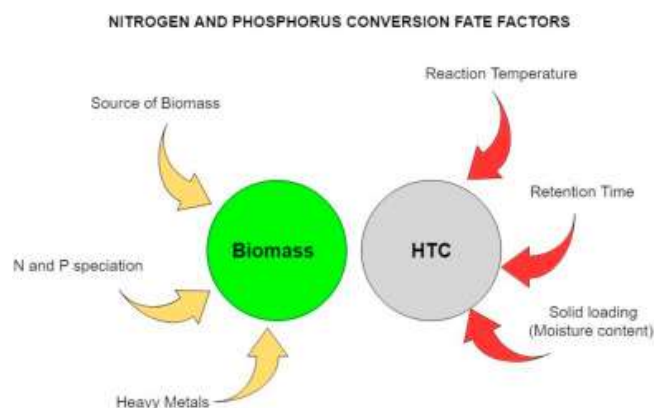


Figure 2: Scheme of possible mechanisms involved in P transformation during HTC.

Following HTC manipulation, all P molecules are usually converted into orthophosphates. Hydrolysis breaks molecular bonds in a wide range of species, including pyrophosphates, P in phytic acidic substances, and phosphor diesters, resulting in soluble orthophosphates, which migrate into the solution fraction. However, the presence of metal ions in biomass waste alters P's destiny during thermochemical process [34]. Metals such as Ca, Mg, Cu and Zn react with P to produce insoluble precipitates (phosphate salts), while Fe and Al hydroxides facilitate adsorption due to their extraordinarily high affinity for P [35]. Several studies have looked at P transformation during HTC operations to have a better knowledge of the transformation mechanisms. The distinction of P throughout HTC was studied using chicken litter and dung from cows, respectively. It has been agreed that recognizing various P types in feedstock is critical for determining P solubility before treatment [36]. Knowing molecular weight ratios of K, Ca, and Mn to P can help assess if there is adequate concentration of minerals to form precipitates along with compact P in HC [37]. It was revealed that the most common phosphate minerals in biomass include Ca, K, and Mg phosphates. To improve P recovery strategies, it is critical that one recognizes how metal cations balance and P forms present in biomass prior to undergoing thermal treatment. This

allows for improved prediction of P transformation routes during the process [38]. HC's potential as an agricultural amendment depends on its P species, with some being more chemically and physiologically accessible. Research shows that multivalent ions made of metals (e.g., Ca, K, Mg, Na, Al, and Fe) form strong connections involving P conversion mechanisms [39]. Thermal treatment causes a shift in P species, with non-soluble P becoming more predominant. Multivalent metallic elements (e.g., Ca, K, and Mg) react with HC to form precipitates, which yield non-soluble P phosphates [40]. As reaction temperature rises, organic P species break down and mix with metal cations, forming phosphate. Due to its stability, the HTC process immobilises P in polymers and apatite forms. While disintegration is not comparable to HTC, research may provide insight into how P changes after extreme temperature treatments. It was proposed by [41] that pyrolysis destroys organic P in biomass, including phytates and lipids, resulting in inorganic metal-P consortia. Throughout the HTC process, refractory phosphates like $\text{Ca}_3(\text{PO}_4)_2$ and $\text{Mg}_3(\text{PO}_4)_2$ form associations that chemo-adsorb across the HC surface [42].

Research on P and N recovery

HTC research on N and P is largely concerned with extraction and solubilisation in water used for processing, as well as the content of nutrients in HC. Due to their high nutritional value, biomass sources such as animal digestates and dung have attracted extensive attention. Other studies investigated biomass feedstocks, including food spoilage, microalgae, and waste from farming [43]. P and N high concentration in sewage trash, in particular, digestate, makes it an ideal feedstock for implementing the HTC method into treated water systems. Integrating HTC with anaerobic decomposition in the treatment of wastewater plants was suggested by [44]. The HTC technique was utilised to digest sewage solids at three temperatures: 180, 220, and 250 °C. The byproducts were consumed anaerobically. They were able to solubilize N and P up to 27 and 58%, respectively. The researchers discovered that N extraction correlated with the severity of the HTC response but not with P solubilisation [44]. HTC byproducts were investigated in granular digest recovered from an up-flow respiratory slurry membrane reactor. HC reduced N content from 9.58 to 5.49%, whereas P was nearly completely immobilised with bioavailability [45].

Existing techniques for recovering P and N

Nutrient recovery technology has advanced significantly in recent decades. Most studies prioritised P above N recovery (Fig. 3). These methods primarily target wastewater and sewage sludge, especially manure [46].

P recovery method produces nutrients with high market value, which are mostly utilised in agriculture. Choosing a P recovery procedure depends on the biomass's qualities. P recovery techniques usually involve acidic elimination and struvite rains,

with variations based on biomass characteristics and state (solid or liquid). According to [47], while there are multiple technologies and techniques for recovering P from sewage effluent, most of them lack waste management capabilities or have high operational costs. Precipitation and crystallisation are used to treat wastewater. Sludge is treated employing Seaborne. Biomass pellets are treated with BioCon. Methods to recover P for biomass, namely sewage waste, have been developed to process the aquatic fraction, sludge, and ash [48]. There are fewer N-focused recovery systems compared to P, and they mostly target liquid waste flows with high $\text{NH}_4\text{-N}$ concentrations. These techniques employ biological processes, such as bacteria, to convert melamine to N_2 , which is then fixed to create NH_3 [49].

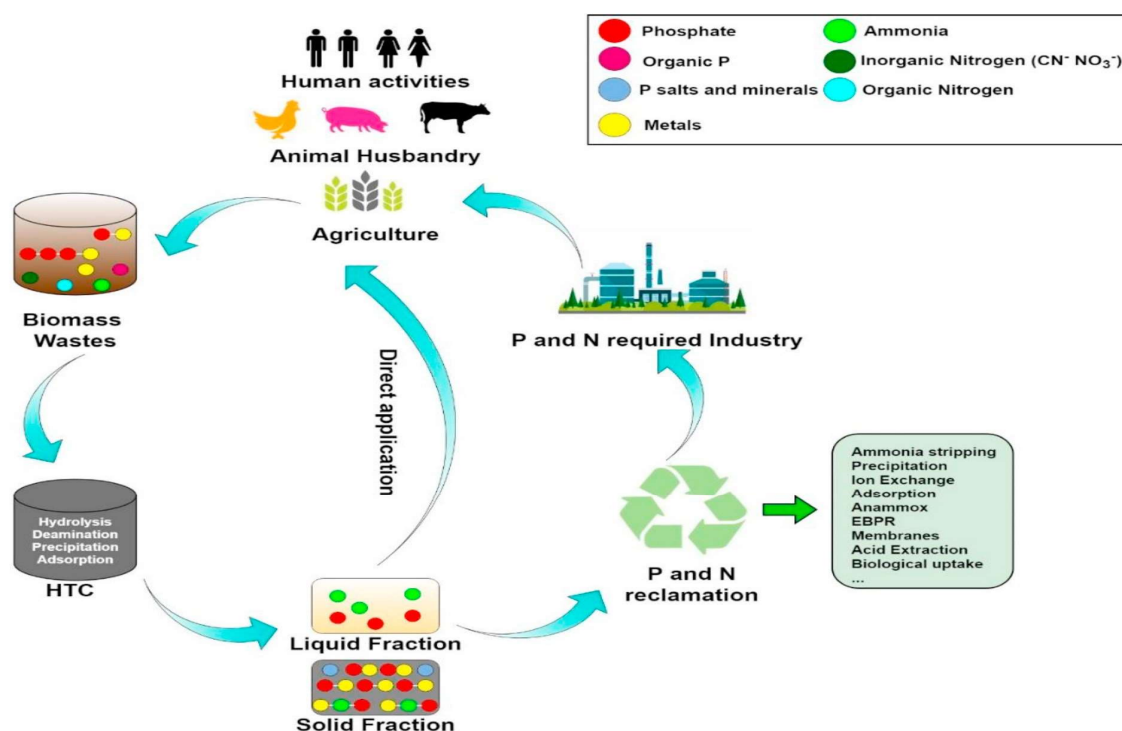


Figure 3: Schematic of the various paths for including P and N recovery as well as recovery into HTC-based processes for nutrient-rich biodegradable waste [50].

Conclusions

The HTC process is a welcome addition to N and P reclamation efforts, complementing a wide range of existing recovery methods and technology. A deeper knowledge of the processes influencing P and N changes and delivery during the HTC method is required. Results would assist in forecasting the steps that follow chemical residues and establishing an appropriate recovery technique. To increase

the recovery of nutrients, one must investigate techniques to include P and N recovery and recycling during HTC treatment of nutrient-dense bio-waste.

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Authors' contributions

N. Jafar: wrote the paper. **N. A. Khan:** corrected all figures and tables. **S. Afzal:** helped with references, DOIs. **A. A. Khan:** edited the manuscript. **A. Ali:** improved the abstract and conclusion. **H. M. B. Munir:** removed paper mistakes and modified. **M. Siddique:** removed plagiarism. **M. S. Aamir:** reviewed and removed grammar mistakes.

Conflict of interest

The author revealed no competing interests regarding the study activity.

Abbreviations

HC: hydrochar

HT: hydrothermal treatments

HTC: hydrothermal carbonization

N: nitrogen

P: phosphorus

WM: waste management

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