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Production and applications of N-doped carbons from bioresources: A review

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ABSTRACT

N-doped and metal-N-doped carbons are receiving increasing attention for environmental and electronic applications. Modifications of carbons such as biomass-derived char by N-doping allow for modulating carbons' acid-base character, adsorption capacity, catalytic performance, and electrochemical properties (e.g., electrical conductivity and capacitance). N-doped carbons are obtained from the thermal co-processing of C-rich and N-rich sources (e.g., lignocellulosic biomass, proteins, and ammonia). Although the literature is abundant in papers on producing heteroatom-doped carbon nanotubes, carbon fibers, and other high-value carbonaceous products from non-renewable sources, the number of articles reporting N-doped chars from bioresources is more limited. Thus, this paper aims to review synthesis processes and activation strategies to produce N-doped carbons from biomass resources and the uses of the resulting materials. Pyrolysis and hydrothermal carbonization offer opportunities to obtain relatively cheap, environmentally friendly N-doped carbonaceous materials with tailored properties for environmental and electronic applications. The role of the Maillard reactions in integrating N into carbonaceous products' structure is also discussed. This paper summarizes desired char properties and the relationship between chemical composition and application performance.

1. Introduction

The conversion of abundant lignocellulosic materials to fuels, chemicals, materials, and carbons is an area of growing interest from both the public and the scientific community [1]. Common lignocellulosic materials consist mainly of 40–60 wt% cellulose, 15–30 wt% hemicellulose, and 10–25 wt% lignin [2-5] besides carbohydrates and lipids, which are rich in C, H, and O. Materials of biological origin (e.g., manure, animal wastes, brewer's spent grains, food waste, yogurt, egg white, human and animal hair, crab and shrimp shells, black gram, spent coffee, soybean residues, and algae) also contain proteins and other N-containing compounds [6-17]. For example, chitin is an abundant natural biopolymer made up of N-acetyl glucosamine derived from the exoskeletons of insects and arthropods [18]. Combining C-rich with N-rich bioresources allows for obtaining desired feedstocks for N-doped carbons production. The conversion of lignocellulosic biomass to N-containing compounds is of significant importance because these compounds offer potential in pharmaceuticals, catalysis, and adsorption applications. Pyrolysis and hydrothermal carbonization, followed by chemical or physical activation methods, are the main routes used to convert biomass sources into porous, cheap, stable carbons with enhanced electrical, adsorptive, and catalytic properties [19-21,3,4,4,5, 22,23].

The substitutional doping of N, B, P, and S heteroatoms within the C polyaromatic ring systems has raised great interest due to the possibility of tuning the acid-base character of the carbonaceous material [24]. Heteroatoms alter carbon's electronic structure, enhancing electrical conductivity, chemical stability, and electron-donating properties [25]. In this review, we focus on N-doped carbonaceous materials. C and N atom's similar covalent radius (0.77 vs. 0.74 Å) allows easy insertion of N into the C polyaromatic lattice [26,27]. However, N has higher electronegativity (3.04) than C (2.55), which induces disbalances in the electronic structure of the C network. N-doping is responsible for surface basicity and improved catalytic performance in oxidation reactions [28, 29]. These changes in electronic configuration enhance energy storage and electron conductivity of carbonaceous materials [30].

Different methods have been reported to synthesize N-doped porous carbons [24]. For example, one method employs NH_3 as the N-source in the carbonization process at elevated temperatures (over 700 °C) [31-33]. However, this process has some drawbacks due to the corrosive nature of NH_3 and its low selectivity since only a fraction of the N is retained in the char [34,35,23]. Moreover, C yield in char is lower when pyrolysis and gasification are conducted under NH_3 than under the N_2 environment [32].

In protein-rich biomass, N is present in organic and inorganic forms. Organic N is in the form of amines, amides, pyridines, and pyrroles, whereas inorganic N is in the form of ammonium, nitrate, or nitrite. Organic-N accounts for most of the N in biomass (for example, in sludge, 88 wt% of N is organic, and 12 wt% is inorganic) [36]. Char derived from protein-rich biomass such as food waste, municipal waste, and animal waste is likely to possess high N content in its polyaromatic structure. Table 1 shows the chemical formula of the main N bio-precursors used in producing N-doped carbons reported in the literature and the number of papers reporting their pyrolysis and carbonization. However, the knowledge required to design N-doped

carbons for devoted applications is dispersed in the literature. According to Science Direct, the number of papers with the keywords: "N-doped carbon (NDC)" has been steadily growing: 1980–1990: 8383 papers, 1990–2000: 18,227 papers, 2000–2010: 29,700 articles, 2010–2020: 113,343 papers. The literature includes excellent reviews on N-doped char chemistry and environmental remediation (see Table 2), but only a few focus on the undelaying mechanisms governing the formulation of the engineered chars from bioresources for targeted ecological applications. Therefore, the objective of this review is to present a comprehensive picture of the preparation of N-doped carbon, strategies for N-rich char production from bioresources, and the use of N-enriched char for environmental applications.

2. Preparation of N-doped carbons

Two methods are employed for the preparation of N-doped carbon. The first method, *ex-situ/artificial doping* (i.e., post-treatment method), involves carbon sources processed at elevated temperatures using N precursors such as ammonia, urea, amines, pyrrole, and acetonitrile

Table 1
Main precursors used to produce N-doped chars and the number of papers reporting their pyrolysis and carbonization.

N-Precursor		Keywords and number of publications
Ammonia (NH_3)		N-doped Carbon + Ammonia: 27,645 papers* Ammonia + pyrolysis: 29,696 papers Ammonia + carbonization: 10,373 papers
Urea (NH_2-CO-NH_2)		N-doped Carbon + Urea: 17,786 papers Urea + pyrolysis: 13,400 papers Urea + carbonization: 5706 papers
Pyrrole (C_4H_4NH)		N-doped Carbon + Pyrrole: 9562 papers Pyrrole + pyrolysis: 6763 papers Pyrrole + carbonization: 2881 papers
Acetonitrile (CH_3CN)		N-doped Carbon + Acetonitrile: 15,080 papers Acetonitrile + pyrolysis: 10,903 papers Acetonitrile + carbonization: 3101 papers
Polypeptide/proteins RCH (NH_2) COOH)		N-doped Carbon + protein: 25,392 papers Protein + pyrolysis: 25,963 papers Protein + carbonization:9582 papers
Chitosan ($C_{56}H_{103}N_9O_{39}$)		N-doped Carbon + Chitosan: 12,216 papers Chitosan + pyrolysis: 6280 papers Chitosan + carbonization: 4345 papers
Melamine ($C_3H_6N_6$)		N-doped Carbon + Melamine: 8566 papers Melamine + pyrolysis: 5776 papers Melamine + carbonization: 3877 papers
Chlorophyll ($C_{55}H_{72}MgN_4O_5$)		N-doped Carbon + Chlorophyll: 1985 papers Chlorophyll + pyrolysis: 3751 papers Chlorophyll + carbonization: 826 papers
Hemoglobin (Heme b group) ($C_{43}H_{32}FeN_4O_4$)		N-doped Carbon + Hemoglobin: 3279 papers Hemoglobin + pyrolysis: 1910 papers Hemoglobin + carbonization: 826 papers

* As of November 2022.

Table 2

Reviews published in the last five years on N-dopped char chemistry and environmental applications.

Authors	Main points of the review
Yadhav and Dixit [30]	Synthesis, characterization, and prospective applications of nitrogen-doped graphene
Lin et al. [37]	Metal ions doped carbon quantum dots: Synthesis, physicochemical properties, and their applications.
Shaik et al. [38]	Nitrogen-doped nanocarbon (NNCs): Status and future opportunities
Jeon et al. [25]	Nitrogen-doped carbon nanomaterials: synthesis, characteristics, and applications
Fan et al. [39]	Algal biorefinery to value-added products by using combined processes based on thermochemical conversion
Hammi et al. [40]	Chitosan is a sustainable precursor of nitrogen-containing carbons.
Leng et al. [41]	Pyrolysis of protein-rich biomass
Cheng et al. [42]	Impact of feedstock composition on lignin and protein pyrolysis
Sun et al. [43]	Algae pyrolysis for char production
Liang et al. [44]	Melamine self-assembled graphitic carbon nitride-based materials
Chin et al. [45]	Magnetic char crosslinked chitosan for metal removal
Kurian and Paul [46]	Use of green sources for carbon synthesis
Leng et al. [47]	Nitrogen transformation in hydrochar during hydrothermal carbonization of biomass containing N
Ababneh and Hameed [48]	Chitosan-derived hydrothermally carbonized materials and their applications
Aragón-Briceño et al. [49]	Hydrothermal carbonization of wet biomass
Ansari et al. [50]	Recent advances in biosensing, bioimaging, and therapy
Vinodh et al. [51]	Chitin and chitosan-based biopolymer-derived electrode materials
Shirsath and Henchion [52]	Bovine and ovine meat co-products valorization opportunities
Khajavian et al. [53]	Chitin and chitosan-based products: preparation strategies aided by deep eutectic solvents
Seow et al. [54]	Char production from different biomass wastes by recent carbonization technologies

[55]. In this process, the N-containing gases decompose at high temperatures and generate highly reactive radicals [25], which react with carbonaceous materials and form N-containing functionalities [24,56]. The *ex-situ* N-doping process requires thermal treatment introducing N sources in activated carbon materials. The second method involves in situ functionalization with a feedstock containing N [27]. Carbohydrate-containing N functional groups such as glucosamine or chitosan have been used as carbon and N-precursors [57,58]. Compared to the post-treatment route, the in-situ approach is more straightforward and has immense potential to get high N content and a stable product. Both methods result in a good distribution of doped atoms [59]. In the in-situ doping synthesis, both N doping and the synthesis of C materials co-occur. Chemical vapor distribution, solvothermal routes, ball-milling, and plasma-arc discharge methods are the most widely used [25,30]. In the literature, treating biomass at elevated temperatures under N-containing gas (mainly ammonia) is reported to be the most common pyrolysis process. [32] studied a simple one-step process

for N-doped porous carbon using a cellulose filter under NH_3. N-doped char exhibited more than double the unit area of capacitance compared to the untreated material. Fig. 1 shows a schematic of both the *ex*-situ and the *in*-situ routes for preparing N-doped carbons.

2.1. Feedstock

In nature, protein-derived biomass sources are widely available [41] a; [60]; [61]; [62]; [10]; [63]. Ideally, protein-rich wastes such as agricultural waste, food waste, municipal solid waste, distillers' grains, dairy manure (solid digestates), and algae are used for producing N-doped chars. Among them, sewage sludge and algae are common N-rich biomass sources. Compared to algae, sludge contained significant amounts of ash, which has better catalytic properties. The aquatic microalgae biomass is primarily composed of proteins, carbohydrates, fats, and lipids in varying proportions. As reported in the literature, microalgae doped char has higher N-efficiency, with well N-fixation. Table 3 shows the C and N content of some types of waste biomass. For example, organic N in protein is an essential component of dairy manure. Crude protein in dairy manure is about 15–20 (wt% dry basis) [64]. Solid digestates from manure anaerobic digestion are also rich in N and P [65]. Food wastes contain lipids, proteins, and digestible sugars, suggesting that they also constitute a potential source for energy-dense fuels and N-doped carbon materials. Moreover, the marine environment contains about half of the world's biodiversity. Marine organisms are a major source of novel compounds, including polysaccharides, proteins, and lipids. Chitin is a polysaccharide widely available in the marine environment [66]. Every year, ~100 billion tons of chitin are produced worldwide by mollusks, insects, fungi, and other related organisms [67]. Alkali deacetylation of chitin produces a bio-compatible, bio-degradable, and non-toxic nitrogenated biopolymer called chitosan (with two reactive primary amino groups) that contains up to 7.5 wt% of N [9].

Table 3

Low-cost materials of biological origin that could be used as N-rich sources to produce N-doped chars.

| Biomass	C (wt %)	N (wt %)	References
Agricultural wastes	27–65	0.5–4.5	Chen et al. [68]
Forestry waste	44–53	0.5–1.3	Agamuthu [69]
Potato skin	71.3	2.6	Önal et al. [70]
Sewage sludge	25.5	4.9	Alvarez et al. [71]
Manure and solid digestate	20–48	1.4–7.8	Arazo et al. [72]
Fresh cattle manure	49.4	3.3	Font-Palma [73]
Sesame de-oiled cake	55.8	1–9	Volli and Singh [74]
Brewers spent grains	46.6	3.9	Sanna et al. [75]
Blue-green algae	48.0	11.4	Falco et al. [12]; Soto-Sierra et al. [76]
Chitin chitosan	40.0	7.5	Sarwar et al. [77]
Coconut shell	48.6	0.14	Daud and Wan [78]
Almond shell	50.5	0.2	González et al. [79]; Dhyani and Thallada [80]
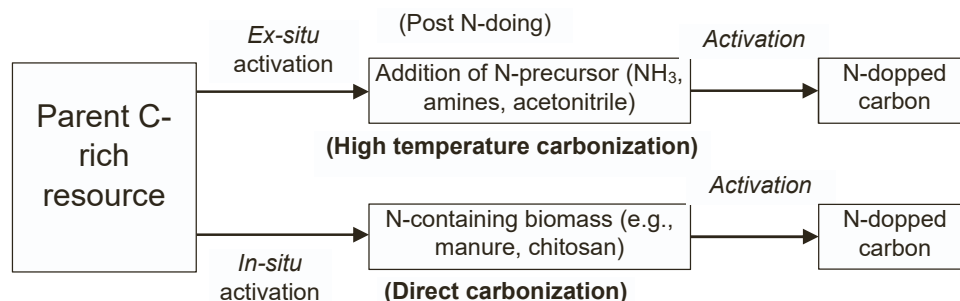
Fig. 1. Schematic of *ex*-situ and *in*-situ routes to prepare and activate N-doped carbons.

Chitosan is abundant in nature as a food waste from the marine industry. These two examples of N-rich materials (i.e., dairy manure and chitosan from marine sources) show enormous potential for producing chars with high N content. Evidently, the main parameter controlling N content in biochar is biomass composition. During pyrolysis/carbonization, the N available in parent biomass is either collected in the volatile fraction (bio-oil) or integrated into the bio-char structure [41,42]. The presence of heteroatoms (e.g., N, O) contributes to the creation of crosslinking centers that increase char yield. Pyrolysis/carbonization parameters (T, P, and residence time) influence the distribution of N and the type of the N-containing compound in the carbonaceous solid product obtained (e. g., in pyridinic or pyrrolic forms) [41].

2.2. Thermochemical processes for char production

2.2.1. Pyrolysis

Pyrolysis involves converting carbonaceous materials into a liquid product, gases, and char in an inert atmosphere (under N or Ar) or an atmosphere with limited content of O_2 at temperatures between 300 and 1000 °C [81]. Pyrolysis technologies are often divided into fast/flash pyrolysis, intermediate pyrolysis, and slow pyrolysis [82] based on the heating rate, residence time, and temperature used. Slow pyrolysis is also often referred to as carbonization. Pyrolysis/carbonization temperature is a critical factor affecting char's the aromaticity and size of the polyaromatic ring system. At temperatures between 400 and 600 °C, pyrolysis and carbonization reactions produce volatile products and char [83]. Overall, the pyrolysis process involves consecutive steps such as dehydration (below 200 °C) and depolymerization (at 200–500 °C) of hemicellulose, cellulose, and lignin [83-85]. Weight losses are minimal as the temperature of the process is increased (i.e., at 600–2000 °C). However, essential changes in the solid structure happen (e.g., formation of pores and surface area, increase in the size of polyaromatic ring systems, and formation of graphitic structures). Processes to maximize char yield (for example, flash carbonization) could result in higher C conversion efficiencies with char yields over 30 wt%. This process could require a pressured environment [86].

The N-containing ring systems derived from protein carbonization can be five-membered, six-membered, and heterocyclic rings. Amine and other nitrogenated groups react with carbonyl or other oxygenated groups to form covalent bonds and precursors for developing carbonaceous residues [25]. A series of reactions such as direct cyclization, dimerization, dehydration, dehydrogenation, deamination, cross-linking, depolymerization, fragmentation, and isomerization occur [41,85,87]. Typically, a fraction (close to 50%) of the N is collected in bio-oil. High N content in bio-oil is a challenging problem and inhibits bio-oil application as a fuel. In addition, it needs extra steps, such as denitrogenating, to upgrade into energy [47]. The transformation behavior of N during carbonization reactions is poorly understood [88].

2.2.1.1. The pyrolysis of N-containing waste biomass.
From the perspective of green chemistry, using waste biomass as a precursor for producing high-value carbons has several advantages, such as environmental friendliness, inexpensiveness, and natural global abundance [89]. Depending on the N source, N in the charred product can be in the form of pyridinic o pyrrolic groups [90]. Using melamine as a nitrogen source and calcium chloride as an activator in ethylene glycol (forming a well-mixed phase) enhanced BET-specific surface area (892 m^2/g), with more microporous, and 5.63 wt% N content [91]. The synthesis of N-doped meso/microporous carbon using renewable biomass bamboo fungus as a precursor via a two-step pyrolysis process has been reported. First, the bamboo fungus was carbonized via direct pyrolysis at 300 °C for 2 h under N_2. Then, the char mixed with KOH and completely dried slurries were carbonized at 700, 800, and 900 °C for 2 h. The resulting char showed excellent porosity and a high surface area

with N doping of 3.2 wt% [91].

[92] reported a one-step production of N-S-doped hierarchical C groups from ant powder. The products showed high surface area, interconnected macro, meso, and micropores, suitable pore size distribution, and appropriate N-atom doping. In another study [93], the pyrolysis of pig compost was investigated. The N content of the residue chars decreased with increasing pyrolysis temperature. Most N-containing tar and ammonia evolved below 500 °C due to lipid, protein, and carbohydrate thermal decomposition [93]. The algae pyrolysis mechanism has also been investigated to understand N conversion thoroughly [94]. The studies showed that N in algae is mainly in protein (90%), accompanied by inorganic N_{ic}-N. Cracking of pyridinic N and pyrrolic N at high temperatures produced HCN and NH_3 [94]. The proposed mechanism in the study helps to control the emission of hazardous N-containing pollution and having high-value N-containing (functional groups) pyrolytic char. Tian et al. [95] also investigated the pyrolytic behavior of sewage sludge and reported different N-containing compounds, such as pyrrolic -N, protein-N, and amine -N, in char structure.

Reaction pathways of protein-containing biomass pyrolysis: In most organic wastes, proteins are the primary sources of N [96]. Frequent amino acids in proteins include glycine, alanine, leucine, lysine, light amides, and amines. During pyrolysis, protein macromolecules break down into smaller fragments, and several heterocyclic compounds, including pyridines and pyrroles, are also formed [97]. N is commonly found in carbonaceous materials derived from proteins such as pyrrole-N, amide-N, and pyridine-N. The peptide-N bonds were transformed to N-heteroaromatic carbon compounds during pyrolysis/carbonization, and some amide-N within char decreased with increasing pyrolysis temperature. The total N content within bio-chars increases, followed by a reduction as pyrolysis temperature increases [98]. [99] summarized major products and proposed reaction pathways based on identified compounds. Fourteen nitrogenous gas and seventy-two tar products were identified [99]. The compounds identified agree with the studies of [100-103]. Table 4 shows the major identified protein fragments in the pyrolytic gases pyrolytic oil derived from some biomass sources. It is seen, for example, that the identified compounds from sewage sludge and food waste are similar.

N-containing compounds (both in gas and solid phase) include linear chains (e.g., long-chain nitriles, fatty acids amides, acetamide, and carbamide) and heterocyclic species (e.g., pyrroles, indoles, pyridines, benzimidazole), which are produced from protein and pyridine structure. The compounds listed in Table 4 show that several heterocyclic compounds directly originate from the rupture of proteins [99]. Protein moieties also react with carbonyl groups of sugars, forming Amadori compounds via the Maillard reaction. The products of diketopiperazines

Table 4
Major N compounds were identified in pyrolysis gases and tars of pyrolyzed waste biomass.

Waste organic types	Main N-compounds	References
Compounds identified in pyrolysis gas		
Sewage sludge/ Food waste	HCN, NH_3, NO, Propane-nitrile, Heterocyclic compound pyrrole, nitriles, Pyridine	Debono and Audrey [99]; Tian et al. [95]; Zhou et al. [104]
Sewage sludge (Only)	4-methyl pentanetrile, two pyrrolidinones, Acetamide	Debono and Audrey [99]
Animal bones	2,5-pyrrolidinedione	Purevsuren et al. [105]
Pig Compost	NH_3	Meesuk et al. [93]
Compounds identified in pyrolysis bio-oil		
Sewage sludge/ Food waste	Heptadecanenotrile, 9-octadecenitrile, DKP, amides, amines.	Debono and Audrey [99]; Cao et al. [106]
Food waste (only)	Hexadecanenitrile, more DKP groups, different oximes groups	Debono and Audrey [99]
Animal bones	Heptadecanenitrile	Purevsuren et al. [105]

(DPK) are created from these intermediate Amadori products [99,107]. Finally, the DKPs further break down and form light amino compounds. The detailed process of the mechanism of the Millard reaction is discussed in Section 2.2.4.

[95] studied the N transformation mechanism of sludge pyrolysis products using TG-FTIR-MS. At different heating rates (10, 50, and 200 °C/min) and temperatures (150–800 °C), the N in the sludge is mainly retained in the following forms: pyrrolic-N, protein-N, and pyridinic-N, amine-N. Above 300 °C, the pyrrolic-N and pyridinic-N are decomposed to heterocyclic-N, followed by the release of HCN. Conversely, amine-N is transferred to N-containing compounds and releases NH_3 at 300 °C [104]. However, the decomposition of protein N is not symmetrical as other N-moieties. At the temperature range of 300–550 °C, amine-N is decomposed, transferred to heterocyclic-N, and finally decomposed to NH_3. In addition, above 500 °C, the heterocyclic-N is decomposed and forms HCN. The macromolecular protein is first cracked into small molecular amine compounds in this stage; then, the deamination process occurs, generating NH_3. Finally, at higher temperatures (600–900 °C), the N compounds are converted to nitriles and produce HCN [104]. From a chemical viewpoint, the aromatic hetero-N compounds are formed via Diels-Alder reactions, and the aliphatic compounds are formed from decarboxylation and decarbonylation of long-chain acids ruptures of heterocyclic compounds [108]. Fig. 2 shows a schematic of the conversion of N species in sewage sludge during pyrolysis.

Pietrzak et al. [110] studied the pyrolysis reaction of brown coal (at 500–700 °C) in an ammonia/urea environment and found that the char structure introduced N in the form of pyridinic-N, pyrrolic-N-N, imine-N, amine-N, and amide-N. Chen et al. [26,111] investigated the co-pyrolysis of lignocellulosic biomass and amino acids and proposed reaction pathways based on identified N species. The dominant tracks were Maillard reaction, cyclization, ketonization, deamination, and cracking. Hyphenated techniques, such as TG-FTIR and Py-GC/MS, confirmed the presence of N-species and helped to propose the reaction mechanism. In addition, [35] showed that the thermo-catalytic conversion of biomass under ammoniation produces different N-heterocycles (e.g., pyrroles, pyridines, and indoles). The carbohydrate undergoes dehydration, decarboxylation, and decarbonylation reactions, producing reactive intermediates such as ketones, aldehydes, acids, alcohols, furfurals, and anhydro-sugars. At the same time, proteins decompose into linear chains and N-heterocyclic compounds (e.g., pyridines, indoles, and quinolines). As the temperatures increased, many reactions (deoxygenation, deamination, and polymerization) were responsible for forming N-based polycyclic compounds [43,112]. Previous studies also focused on the pyrolysis products of waste biomass and their potential impacts on the environment [95]. During pyrolysis, the transformations of organic materials produce many hazardous materials, including S and N-containing compounds, heavy metals, dioxins, and furans [100-103]. The N oxides (NO_x) are of concern because they are responsible for ozone layer depletion and acid rain.

2.2.1.2. Synthesis of N-doped carbons. N proximity to C in the periodic table, with its equal atomic radius, facilitates its introduction into the carbon lattice structure [24,25]. Thus, N is the most common atom used to dope carbonaceous materials. N substitution in the carbon framework favors the formation of the pentagons and hexagons, increasing the reactivity of neighboring carbon. N-doped carbon-based nanomaterials have numerous advantages, such as adsorbents, catalysis, and semiconducting materials, among other uses. [95] studied the synthesis of heteroatoms (N and S) co-doped via direct pyrolysis of glucose, sodium carbonate, and thiourea. Prepared N-S doped porous carbons showed remarkable hierarchical porosity and surface area. These prepared materials also showed adsorptive and oxidative removal of pharmaceutical contaminants [113]. A simple, facile, and cost-efficient method was reported to prepare N-doped carbon nanotubes using ball milling. Melamine and urea were used successfully as N precursors in carbon sources (Nanocryl 3100). As a solvent-free method, this process incorporates copious amounts of N-groups (such as quaternary N, pyrrolic, and pyridine) into the carbon structure.

[114,115] studied the pyrolysis of cellulose and additives (with high N content, such as dicyandiamide) and found that the organic N interacted during pyrolysis to increase char yield. Ayiania et al. [116] synthesized N-doped chars via carbonization followed by ammonification at elevated temperatures (above 800 °C) and quantified surface functional groups via deconvolution of X-ray photoelectron (XPS) spectroscopy results. The study found that the most prevalent C-N bonding configurations in synthesized N-doped carbon materials were pyrrolic-N, pyridine-N, graphitic-N (or quaternary-N), and pyridine N-oxide (see Fig. 3). The leading functional group on the surface char includes five-membered rings, six-membered rings, and heterocyclic rings [41]. Pyrrolic N refers to N atoms that contribute with two p electrons to the π system and quaternary N atoms that generally substitute C atoms in a hexagonal ring [30].
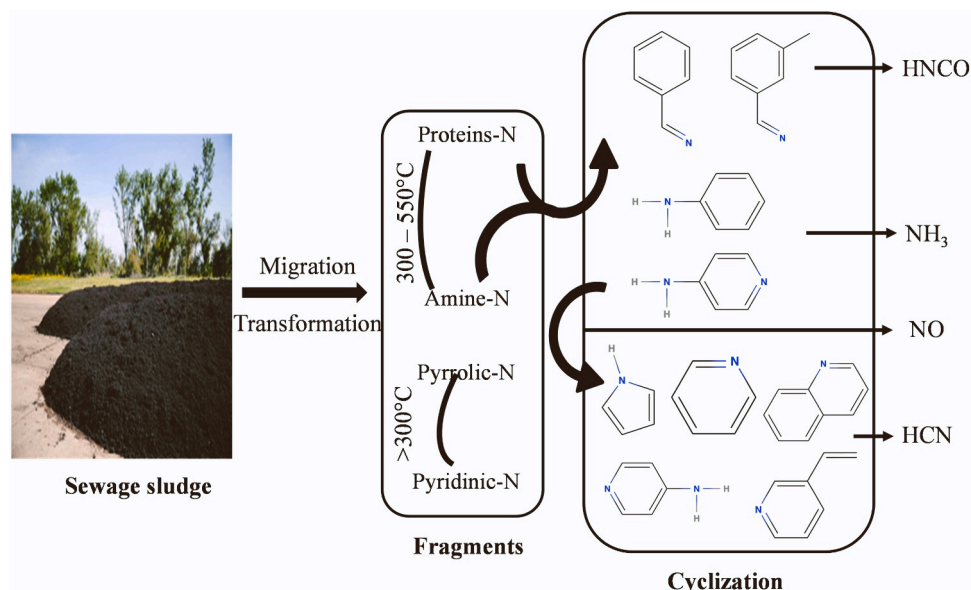

Fig. 2. Reaction pathway for N compounds in sewage sludge during pyrolysis (adapted from [109] [99]; Zhou et al., 2017).
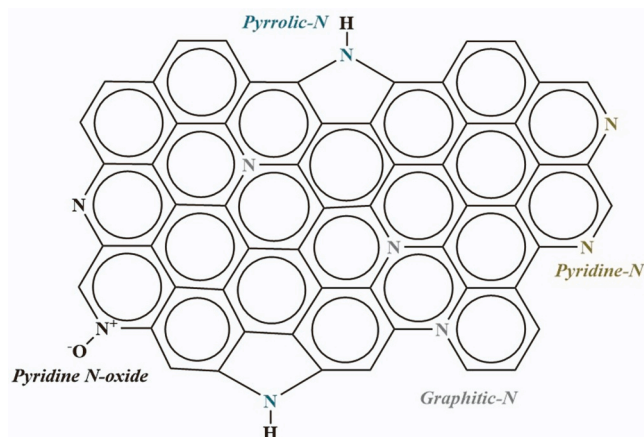
Fig. 3. C-N bonding states into the N-doped carbon nanomaterials Redrawn from Joen et al. [25]; Yadav and Dixit [30].

Pyrolysis of N-containing compounds followed by activation has been extensively studied in the literature [25,117]; [100-103]; [118]. Many N-containing compounds, such as ionic liquids and organic polymers, have been used as precursors of N-doped porous carbons [119]. Ionic liquids (heteroatom-containing organic salts) have been used in molten electrolytes for battery applications. Currently, ionic liquids are used in other fields, such as chemical synthesis, catalysis, CO_2 capture, and materials science. Ionic liquids-derived porous C with controlled hetero atoms provide a reliable platform to study metal-support interaction [17,120]. [121] synthesized N-doped carbons via the hydrothermal treatment of glucose in the presence of model proteins. The resulting N-doped carbonaceous nanoparticles showed a high surface area of up to 8 wt% N.

2.2.2. Hydrothermal carbonization

Hydrothermal carbonization (HTC) is a thermochemical conversion process under aqueous conditions that simulates natural coalification [122] and is employed mainly for processing materials with high moisture content. HTC is simple, low-cost, and can process wet biomass. It was first introduced by Bergius as early as 1913 [123]. In HTC, biomass is processed under mild temperatures (180–260 °C, in a closed system under autogenous pressure), resulting in the formation of solid

products [124]. During HTC, hydrolysis, dehydration, decarboxylation, aromatization, and re-condensation co-occur as part of an inter-connected reaction network [122]. As shown in Fig. 4, the N-containing compounds (mostly from proteins) degrade to amino acids, and the cellulose/hemicellulose degrades to oligomers and monomers such as glucose, xylose, and fructose. Hydrolysis breaks down biomass, forming biopolymers through the cleavage of ester and ether bonds. The reaction between aldehydes and amines (Mannich reaction) and the Maillard reaction are critical for integrating N into the growing carbonaceous product [47]. In the dehydration process, water is removed (causing hydroxyl groups elimination and the formation of carbonyl and C-C double bonds). Decarboxylation occurs by removing CO_2 (carboxyl group) from the biomass. Aromatization occurs through dehydration and decarboxylation, and double-bonded functional groups replace single-bonded groups in the carbon structure, forming Amadori compounds. In the second stage, the unstable Amadori compounds would generate imine and pyrazine derivatives via dehydration and conden-sation. The furans/aldehydes condense with amino groups and form imines (Schiff's base). Some imines would be converted to N-heterocy-clic compounds (pyridines, pyrroles) through dehydration and ring closure [125]. Overall, the combination of proteins and carbohydrates via the Maillard reaction forms Amadori products that further react, forming a broad range of N-containing heterocyclic aromatic com-pounds. Finally, the reactive groups recondense [126,127].

Compared to its equivalent dry thermochemical processes (i.e., py-rolysis/carbonization), a considerable amount of N is lost during HTC, decreasing N content in synthesized materials [23] [34]. Some authors suggest that the carbohydrate fractions may follow similar reaction pathways under dry and wet conditions. Still, the significant component of proteins mostly available in sludge and manure may change the re-action pathways in a hydrothermal environment [123]. The main dif-ference between pyrolysis and hydrothermal carbonization is the removal mechanism by which thermal depolymerization products are removed [128]. In pyrolysis, the primary pyrolysis products are removed by evaporation [128] and thermal ejection [129]. In HTC, the oligomeric products are removed via solubilization in the liquid envi-ronment [130]. The solubility in the fluid environment depends on the properties of the solvent [131]. In addition, in pyrolysis, the vapor products removed from the biomass particle can continue reacting and form a secondary carbon in the vapor phase [132]. During HTC, the oligomeric and monomeric products solubilized in the liquid phase can
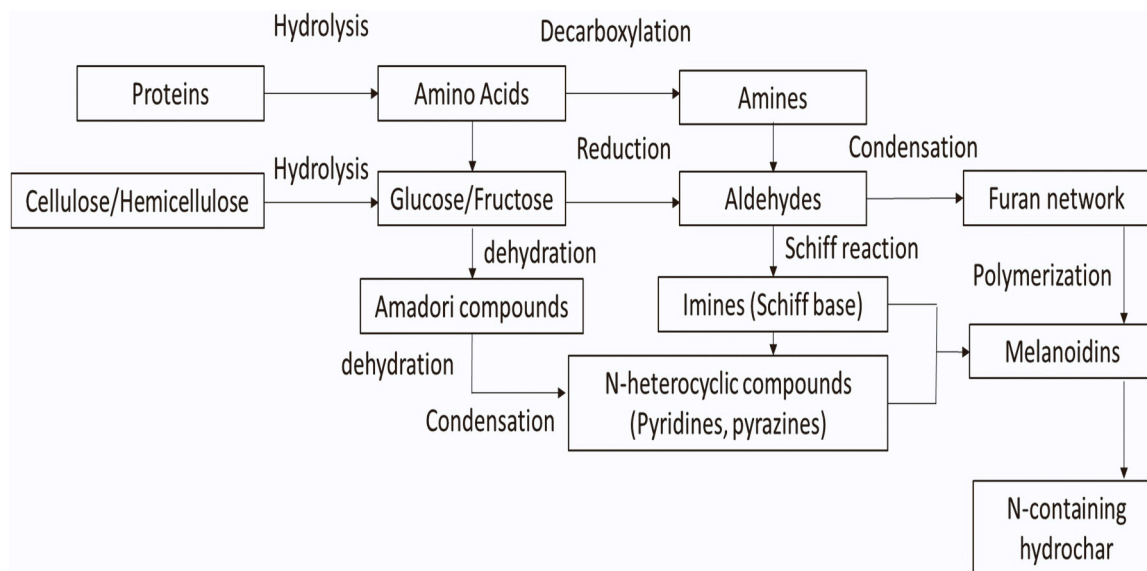

Fig. 4. The proposed mechanism to produce N-doped chars via HTC Modified from Leng et al. [47].

continue reacting to form larger polyaromatic ring systems that can lose solubility in the solvent and form carbon microspheres (Khan et al., 2018).

In HTC, it is hypothesized that the first step under hot pressure water is decomposing the peptide bond (the bond between carboxyl and amine groups) into amino acids. Almost in parallel, cellulose, hemicellulose, and lignin are also subjected to hydrolysis and cracking depolymerization reactions. Although the depolymerization process is slower than carbohydrate hydrolysis, the environment promotes the degradation of proteins into several amino acids, such as leucine, isoleucine, and phenylamine. The amino acids from, for example, sewage sludge initially undergo a Maillard reaction with the sugars stage. These brown polymeric compounds are melanoidins [133]. As shown in Fig. 5, during the Maillard process, the first step is a reaction between dissolved fractions of carbohydrates and protein, which undergo a series of responses, including hydrolysis and dehydration, thus contributing to the formation of N-doped poly-aromatic hydrochars. When the molecular weight of the organic materials in the solution grows, its solubility decreases, and the material reprecipitates to form micrometer size N-doped carbon spheres. The undissolved fraction is believed to follow solid-solid conversion, as in pyrolysis, following condensation, dehydration, and decarboxylation to develop char-like products. Consequently, during HTC, two types of chars are produced: one that is water-soluble and one that is water-insoluble.

Various amino acids have been employed as model compounds in the literature to study reaction mechanisms during HTC [123]. The HTC of glycine and alanine showed that decarboxylation and deamination are the main reactions during the process [134,135]. The typical Maillard reaction between D-glucose and glycine showed that Amodori products, followed by fission product rearrangement and aldol condensation, formed brown nitrogenous polymers (melanoidins) [134]. Similarly, He et al. [136] reported that HTC of sewage sludge results in a uniform brown hydrochar, with a nut-like smell, which confirmed the Maillard process. Some researchers showed that using optimum temperature and residence time dominated the Maillard process. The sewage sludge carbonization at 140 °C (240 mins residence time) and 160 °C (60–120 min residence time) showed no Maillard reaction products. Conversely, the HTC process at 180 °C (15 min) dominated the Maillard process [133]. Therefore, efficient HTC processes depend on the composition of protein-containing biomass (protein species, amino acids) and processing temperatures.

HTC is appropriate for obtaining N-doped porous carbon, especially from biomass containing high moisture (e.g., animal manure, sewage sludge, spent gains, and different aquatic biomasses). In previous studies, algae and sewage sludge have been used as C and N precursors during the HTC process [137]. Since the microalgae possess high photosynthetic efficiency and are fast-growing plants, these precursors are promising renewable resources for N-rich hydrochars. [12] studied a one-step green route to get N-doped carbon via HTC of aquatic micro-algae with high N content. Adding glucose to the initial microalgae mixture is beneficial for enhancing fixation and incorporating N (higher N conversion efficiency). Nevertheless, dairy manure, other aquatic plants, and the most abundant agricultural wastes are limited to the HTC process due to low-value products. Therefore, future research should focus on these abundant feedstocks for facile, sustainable N-doped carbon synthesis. Cross-linking agents such as glucose and dehydrating agents (e.g., acids and bases) could enhance char yield and fix N loss during the process.

The addition of citric acid to the HTC of macro-algae (*S. horneri*) (also known as seaweeds) increased char yield by up to 50% [138,139] obtained N-doped hydrochars with high surface area and adsorption capacities upon chemical activation with KOH. Similarly, [100], [101], [102] and [103] reported an improved HTC process from inexpensive sugar with an ionic liquid to obtain nano-porous N-doped carbon (with a surface area of 572 m^2/g), followed by post-heating treatment. [140] studied microalgae carbonization and found that reaction conditions influence char composition. Xiong et al. [141] reviewed the HTC of swine manure and reported a slight loss of N content in chars. Increasing the reaction temperature from 200° to 280°C slightly decreased the N content in the hydrochars (from 2.8 to 1.5 wt%). Similar studies under low temperatures (140–220 °C) were conducted by [142] who reported comparable N retention in all hydro cars, with an increase in carbon content. Baccile et al. [121] synthesized nano carbons using albumin (as N-sources, model proteins) with glucose via the HTC process. The resulting carbon had around 8 wt% N.

Previous studies reported HTC followed by pyrolysis to fix N in the carbon structure using, for example, spent grains [143]. The study showed a high char yield, increasing N-doped carbon chars. Protein-containing biomass offers the potential for synthesizing N-doped carbonaceous materials. However, N mineralization and immobilization still need to be better understood, and their economic viability has yet to be proven. Therefore, a detailed understanding of the HTC process, the influence of process parameters, and reaction mechanisms are needed to synthesize N-doped carbons from protein-rich biomass. The analysis of products using state-of-the-art techniques such as XPS and NMR showed that the N is stored in stable heterocyclic aromatic structure forms such as pyrrole, pyridine, and quaternary-N.

2.2.3. Selective carbonization

Selective carbonization is a concept in which the knowledge gained on C and N carbonization reactions is used to design a process to maximize the integration of feedstock C and N into polyaromatic
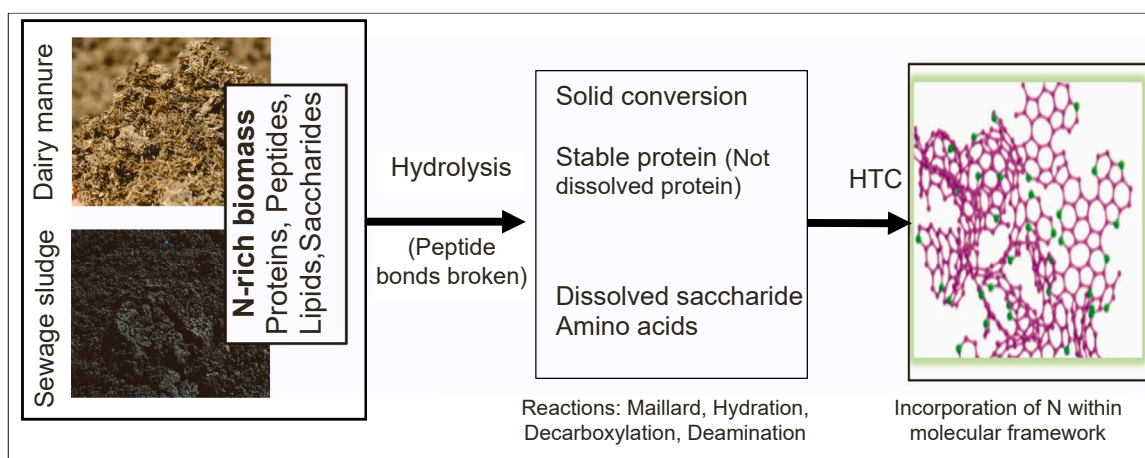

Fig. 5. Hydro-char formation pathway from N-rich biomass (such as dairy manure and sewage sludge) during HTC.

carbonaceous products. Maximizing C and N conversion efficiency is the primary goal of selective carbonization. Because of the importance of the Maillard reaction in controlling the interactions between the N source and carbohydrates, controlling C: N in the feedstock is likely to be one of the essential variables for achieving high N and C conversion efficiency. As compared to slow pyrolysis (which results in around 20% char yield), selective carbonization could result in a higher char yield (up to 50%) [114,115]. To maximize C conversion efficiency, the O contained in the biomass must be removed in water. Conditions facilitating dehydration reactions will result in higher char yields and C conversion efficiencies. The elimination of H_2O needs an acid catalyst for oxygen protonation at the a-position. In the absence of catalyst, dehydration reactions in pyrolysis conditions typically happen at around 300 °C. [144] noted that the peak temperature for completely protonated alcohols is close to 100 °C. The ionic mechanism is launched by the high-temperature boiling tars (oligomers) produced from the cracking of cellulose, hemicellulose, and lignin [144]. More studies are needed to accelerate dehydration reactions in pyrolysis and HTC conditions.

Dehydrating agents are reported in the literature as cross-linking agents. Blasi et al. [145] studied the pyrolysis of wood catalyzed by $ZnCl_2$. The use of this Lewis acid enhanced char yield and modified char properties. [146] reviewed the catalytic action of $ZnCl_2$ in the substrate (corn-cob) pyrolysis and reported it as highly favoring dehydrating and cross-linking agents. However, using $ZnCl_2$ can be harmful and detrimental to the environment [146]. Alternatively, calcium carbide (CaC_2) has been reported as a dehydrating and binding agent in synthesizing high-value chemicals. Phosphoric acid is known as an activating agent during pyrolysis. Zhao et al. [127] studied phosphorous-catalyzed biomass conversion to improve carbon loss and char stability. Different P-bearing materials were tested to fix carbon loss during pyrolysis. Chu et al. [147] tested bio-chars and raw biomass using H_3PO_4. Due to acid catalysis and crosslink, maximum char yield with high surface and micropores was reported. Dehydration reactions produce carbonyl groups, which are critical for the Millard reaction. N integration into the char structure is mediated by dehydration reactions with carbonyl groups [88,148]. The careful N-rich biomass selection and modulating of C:N ratios by co-carbonization can help to create in-situ N-doped chars [88,148]. A limited number of studies have been conducted to maximize char yield from N-containing biomass. Therefore, it is necessary to understand the Maillard reaction of protein carbonization and N mineralization.

2.2.4. Maillard reaction

The French physicist and chemist Louis Camille Maillard (1878–1936) described for the first time the "Maillard reaction" [149] as a non-enzymatic browning reaction that occurs between reducing sugars (carbonyl groups) and amino groups (proteins, peptides, and amino acids), as depicted in Fig. 6 [150,151]. The Millard reactions have been intensively studied in food processing and storage, where the formed Amadori compounds are the main precursors of aroma, color, and flavor. Amadori compounds and melanoidins have health-promoting properties [149]. Although the mechanism of the Maillard reaction is well understood in the food and flavor industry, its impact on the carbonization of feedstocks containing proteins is less understood. Thus, strategies for controlling this reaction during the production of N-doped

carbons warrant further investigation. N-containing recalcitrant organics can be produced during the Maillard reaction.

Maillard reactions take place in both "dry" and "wet" processes and are influenced by operating temperature, pH, and the composition of reactants (e.g., the C:N ratios). The chemistry behind the Maillard reaction is complex as it proceeds through several steps, i.e., dehydration, condensations, cyclization, rearrangements, and isomerization. In the first step, sugars and amino acids follow a condensation process and form a Schiff base. The included base can afterward rearrange to form an Amadori product. In the second stage, dehydration and fragmentation occur in the sugar molecules; amino acids are also degraded, and color formation occurs through various reactions to form low molecular weight compounds. Then, high molecular weight melanoidins are formed via polymerization and cross-linking of low molecular weight melanoidins [152-154]. The Maillard reaction products are believed to be heterogeneous mixtures of compounds such as aldehydes, ketones, carbonyls, and heterocyclic amines with large molecular weight compounds [123]. Melanonids are negatively charged molecules whose molecular weight depends on reaction time and temperature. Researchers reported high molecular weight (>10 kDa) of melanoidins from food model compounds under higher temperatures or longer residence times, such as (121 °C for 30 min) or 100 °C for more than 48 h [155,156]. Also, studies of hydrothermal sludge pretreatment at 165 °C showed intense color-dissolved organic N (melanoidins) with high molecular weights (>10 kDa). The main factors involved in the reaction are the carbon precursors (sugars) and types of amino acids (proteins). Maillard reaction in HTC of protein-containing biomass depends on reactants' carbon and N content, pH, and temperature.

2.3. Char activation strategies

Biomass-derived N-doped carbon has normally been synthesized using chemical and physical methods or combinations of both [30]. Physical activation is reported in the presence of air, CO_2, stream, and other oxidizing and reducing agents. A drawback of physical activation (via gas such as ammonia and CO_2) is that it suffers from exothermic reactions (e.g., the reaction of carbon with oxygen), making the reaction challenging to control, which decreases carbon yield. In chemical activation, various activation agents, such as KOH, NaOH, K_2CO_3, $MgCl_2$, and $AlCl_3$ are used [157]; Tan et al., 2016). The use of these agents enhanced surface area and pore size. KOH has been reported as an activation agent to optimize surface area [158,159]. Fan et al. [160] stated that highly porous carbon with high surface area by altering the K_2CO_3 /chitosan ratio and activation temperature. Compared to physical activation process, chemical activation is preferable as lower temperatures can be used. The maximum char yield can be achieved by removing oxygen in the form of water using acids. An improved $ZnCl_2$ chemical activation has been reported, which increased the surface area of activated carbons [161]. Similarly, (Branca., 2010) pyrolyzed corncobs using $ZnCl_2$ and reported cross-linking agents and high char yield. [145] used $ZnCl_2$ as dehydrating as well as a cross-linking agent during wood pyrolysis.
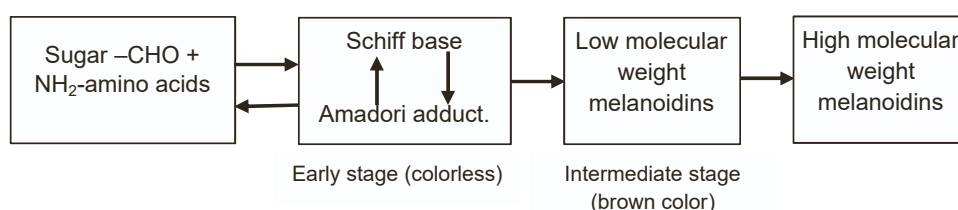
Fig. 6. Schematic representation of Maillard reaction [123,152].

2.4. Strategies to control N loss during selective carbonization

2.4.1. Optimum reaction reactants (co-carbonization)

Although co-pyrolysis with different activators/dopants has been reported in the literature, the idea of co-carbonization and its synergetic effects has been limited to studying protein-containing biomass. Municipal sludge is rich in polysaccharides (20–40 wt%) and proteins (30–50 wt%). Consequently, in the thermal conversion of these materials, the Maillard reaction plays a vital role [162]. Researchers studied the effect of N content in Maillard reaction products, varying the C:N ratios from 7.4 to 26.3 [163] and showed that, by increasing the process temperature, the 1:1 molar ratio of reducing sugar to glycine increased dissolved organic N content in the liquid phase. The reactivity of short-chain amino acids, color development, and amine utilization rates were much higher than single amino acids. The co-carbonization of metal-rich feedstocks (e.g., sewage sludge) with unpolluted biomass (e. g., bamboo, hazelnut shell, and walnut shell) helped to reduce the risk of metal leaching [164]. Additionally, the co-carbonization of seaweed (high ash-bearing biomass) with lignocellulosic biomass (terrestrial biomass) showed a high adsorption capacity for cationic dye removal from industrial effluents [165]. Recently, [88] studied the co-carbonization of cellulose and different N-containing compounds and reported modulating C:N ratios effect on char yield and N-conversion efficiency. More importantly, the use of food waste (chitosan) could be a better additive to get high N-content in char.

2.4.2. Suitable reaction conditions

As discussed in Section 2.4.1, the C:N ratio (quantitative ratio of amino N to reducing sugar) is the main factor controlling the Maillard reaction. Besides, other processing parameters, such as time, pH, and temperature, directly affect the Maillard process. The temperature affects the molecular weight, biodegradability, color, and composition of Maillard products (melanoidins). [166] studied HTC of model compounds protein and glucose to simulate low lipid microalgae and reported that the temperature and time affect the severity of the Maillard reaction during the hydrothermal process. The availability of the reactive forms of sugar and amino groups is responsive to pH changes. The open chain of reducing sugar and the un-protonated form of an amino group at high pH is considered reactive moieties. However, if pH decreases during the process, the amino group will be protonated and gradually lose its reactivity with sugar. This mechanism is fundamental during the hydrothermal process in which the formation of short-chain fatty acids (drop in pH) occurs with the Maillard reaction. [167] studied the glucose-lysine system without buffer addition and reported a drop in pH during the process, resulting in less dark polymerized melanonids products.

2.4.3. Use of cross-linking agents

Metallic ions such as magnesium, calcium, aluminum, and iron and their corresponding salts are commonly used in wastewater treatment and sludge management. Melanoidins (anionic compounds) could bind with metallic ions. The Amadori products formed at the early stage of the Maillard process are able to bind metallic ions with remarkable coagulation capacity. Several metallic ions, such as transition metallic ions (e.g., Fe^{+3} and Mn^{+2}), are used in wet Maillard reactions to increase dissolved organic N in the liquid phase. Metallic cations (e.g., Al^{+3} and Ca^{+2}) show reduced dissolved organic N in the liquid phase. Therefore, selecting suitable metallic ions during dry Maillard reactions could be a potential strategy to fix inorganic N loss during the selective carbonization process. Morales et al. [168] examined the iron (II) binding ability of food-originated melanoidins and concluded that no relationship exists between browning reaction and iron complex. The cross-linking of proteins occurs during the final stage of Maillard reactions. Nevertheless, the impact of crosslinking agents on reducing N losses during carbonization still needs to be studied.

2.5. Chemical nature of N-doped carbons

Understanding the chemical nature and the theoretical formation mechanism of N-doped carbons may lead to better process performance and, therefore, may allow for obtaining improved products [169,170]. Comprehending the N-doped carbons formation mechanism requires an understanding of the reactions involved, intermediates, and products, as well as the molecular structures implicated. Nevertheless, the complex nature of the large systems that make up bioresources represents a tremendous challenge for molecular modeling and formation mechanism studies [171]. Such complexity is due to the multilevel character of the process used to produce the N-doped carbons [172], the multiple reaction steps that occur in the process, the multiphase interaction [173-176], the multiscale of the phenomena involved in the process [177], as well as the challenges to interpreting data from the characterization [178]. Some authors have approached this matter by addressing not the entire system but by analyzing particular molecules that represent the carbon and studying how the N molecules interact with it. Characterization techniques also have been used to understand and validate the importance of N-doped carbons, including the improved surface and electronic properties attributed to nitrogen in a quaternary form that leads to an electron-enriched carbon surface and therefore stimulates the mobility of metals and other components [170, 179] studied N-doped carbon nanotubes with N functional groups in distinct positions within the structure employing XPS data with electrochemical measurements. They claimed that pyridinic-N functionalities are more stable than pyrrolic-N, which is consistent with the findings. The authors concluded that the first stage in the process is that the unstable pyrrolic functionalities transform into pyridinic-N, followed by an immediate transition into the quaternary center and valley N functionalities (when enough temperature is provided) (Fig. 7).

Hu et al. [180] explored the formation mechanisms of N-doped carbon dots prepared from the citric acid in monoethanolamide (MEA) by employing TEM, time-dependent UV–vis, FTIR, and thermogravimetric analysis. The authors proposed the following formation mechanism: polymerization (dehydration of MEA that reacts with citric acid leads to the formation of large-sized polymers), aromatization (heating promotes the formation of C=C and C=N bonds and therefore aromatic clusters are formed), nucleation (critical supersaturation of aromatic clusters allows the nucleation of N-C dots), and growth (polymers are "sacrificed" to enable the N-C dots to increase).

A mechanistic pathway for including N groups in the hydrothermally produced carbon was proposed by Latham et al. [181], who employed Synchrotron-based Near-Edge X-ray Absorption Fine Structure (NEX-AFS). They identified C=N and C-N functionalities and N groups such as pyridinic, pyrrolic, and tertiary amine nitrogen surface groups. Two main reaction pathways were suggested: (i) dehydration and (ii) retro-aldol condensation, followed by a cascade of parallel reactions due to the incorporation of NH_4^+ into the hydrothermal solution, which facilitates the formation of N-functional groups that are more stable than oxygen-functional groups (valid for subcritical temperatures and pressures). Other authors also claimed that the incorporation of N happens via the Strecker pathway [182] and the decarboxylative Strecker degradation pathway [183].

Huang et al. [184] analyzed diverse types of N species in N-doped carbons and found that the oxidation degree of the carbon precursors was correlated with the N content of the N-doped carbon obtained. As more O was present in the source, more N content was in the product. The incorporation of N within the structure was related to O-containing groups because these groups react easily with the reductant, replacing those O groups with N. Additionally, pyridinic N and graphitic N presented a linear relationship with total contents of C=O/–COOH (located at the edges) and –OH/–C(O)C– (located at plane) functional groups, respectively. These results are promising and of interest to test in carbon sources such as biomass with high O content. More importantly, these results were consistent with DFT calculations obtained by other authors
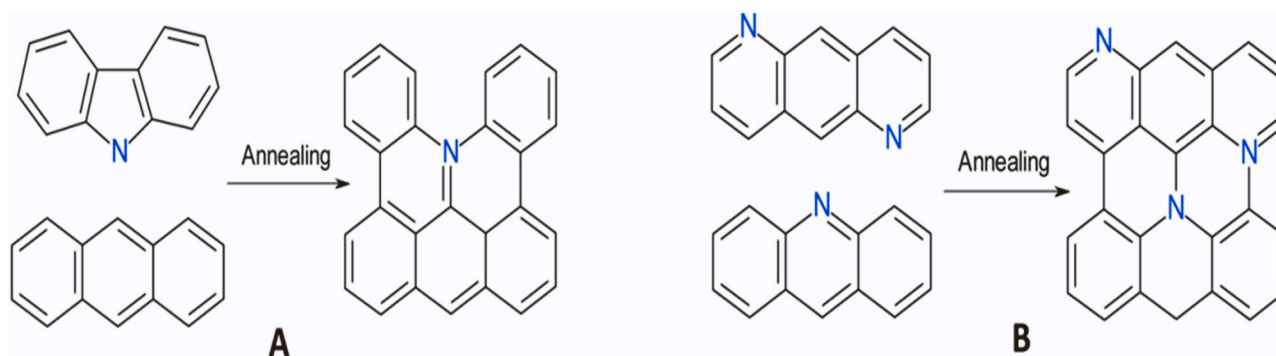
Fig. 7. Condensation reaction by annealing, (A) from pyrrolic-N (N 2) to N-Q$_{valley}$ (N 4), (B) from pyridinic-N (N 1) to N-Qs (N-Q$_{center}$ and N-Q$_{valley}$) [179].

[185,186].

[169] used the first principle-based models of diverse polyaromatic structures containing N functional groups and compared the effect of incorporating those in the char Raman spectra. The authors employed models' compounds, including acridine, carbazole, acridine, anthracene, amino anthracene, and coronene, to identify the vibrational modes of the different N groups. After comparing DFT modeling and experimental results, they proposed the most representative vibrational features observed in the molecular structures evaluated. Based on the peak assignment, they meant to perform a deconvolution of the Raman spectra of the N-doped chars. They found that the N effect has more impact in the region between 1400 and 1550 cm^{-1}, and single- and double-point defects are responsible for the intensity in the valley region of the spectra. Mixed experimental techniques, characterization (SEM, XPS, XRD, TEM, surface area), and DFT modeling to predict how the incorporation of N in the ring system structure of the carbon interacts with its properties have also been employed [169]. The authors hypothesize that a single-point defect is created within the carbon ring structure during the N-doped process, and the C-C bond is broken and replaced by pyridinic groups. Then, a metal atom (Mg or Ca) is embedded in the matrix and connected to the N functional groups creating a more stable structure (See Fig. 8). They also evaluated the hypothesis at the system's edges and theoretically compared both edges and internal active sites by quantifying the stability of Mg and Ca. Internal active sites (Fig. 8c) were more likely to occur due to their negative adsorption energy. The functional groups formed for cellulose char doped with N and metals (Mg and Ca) are probably phthalocyanine-like structures.

The thermodynamic stability of N functionalities in graphene structure was also studied by [116], who evaluated how temperature and pressure affect graphene stability using DFT modeling. The study was essential to understand how the treatment properties affect the graphene electronic properties and, therefore, unable to tune the process. Base models of pristine graphene, graphene nanoribbon terminated with H, and graphene nanoribbon completed with both H and O were analyzed. N groups, such as pyridinic, were incorporated within the graphene structure and the edges. They claimed that results on Gibbs's reaction energy evidenced that low temperatures and high pressures favor incorporating N into the graphene structure. Additionally, they concluded that pyridinic N is most likely formed at the graphene edge structure, pyrrolic N is the most stable structure, and the functional group in the edge of the graphene structure (either H, N, or O) has not a significant effect on the electronic system of N functionalities.

A recent work studied how N was transformed during pyrolysis to produce N-doped chars [187]. The authors analyzed various N functional group evolution in gas-liquid-solid phases during the co-pyrolysis of biomass. Some groups identified in the process include quaternary-N, pyrrolic-N, pyridinic-N, NH$_3$, and HCN. They found that pyrolysis temperature is vital in distributing the N functionalities within the N-doped products and, therefore, the N pathway. At lower temperatures, leucine cyclization and dimerization of aspartic acid were pathways for pyridinic-N in char, whereas proline decarboxylation and leucine cyclization originated pyrrolic-N in char. Besides, high temperatures promote the cyclo-condensation of pyridinic to quaternary-N or HCN via further cleavage. Accordingly, theoretical studies mixed with characterization techniques have been meaningful in elucidating and clarifying how N-doped carbons are created. However, most of the work reported in the literature is for carbon nanotube materials, and only some work has been reported for biomass as a carbon precursor. One of the possible pathways reported can be described as follows (Eq. 1) [188]
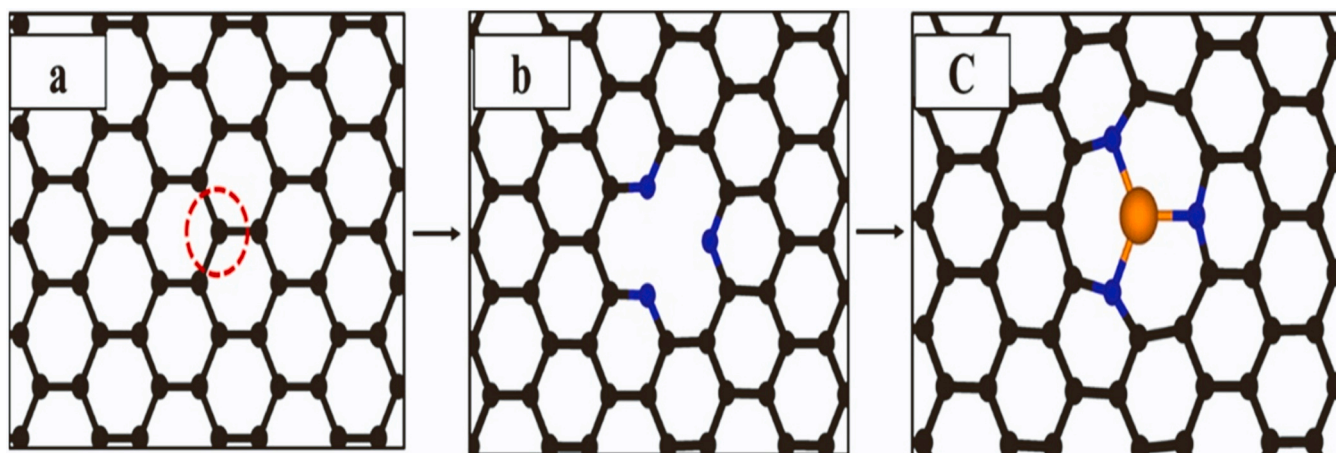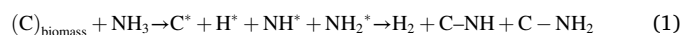
$$(C)_{biomass} + NH_3 \rightarrow C^* + H^* + NH^* + NH_2^* \rightarrow H_2 + C-NH + C-NH_2 \tag{1}$$

Fig. 8. Schematic representation of the creation of N-metal graphene models [169].

Therefore, the introduction of ammonia in the biomass pyrolysis process increases H formation, generating many N-containing species and decreasing the production of O-containing species [189,190]. Nevertheless, the formation mechanism of the reactions involved in the process deserves further study. DFT calculations suggest that the formation of graphitic-N from NH_3 and defective carbon structures with O groups, such as carbonyl or hydroxyl groups, is feasible at elevated temperatures from an energy point of view [185,186]. Therefore, not only the reduction agent used to produce N-doped carbons is essential for reduction and doping, but defects and O functional groups should also be considered.

2.6. Characterization of N-doped carbon

After preparing N-doped char, it is important to determine its bulk and surface chemical composition, surface morphology, and other related physic-chemical properties. Fig. 9 shows the most common analytical techniques used to characterize carbonaceous materials. N-functional groups on the char surface are in five-membered rings and six-member rings. There are several studies on analytical techniques for characterizing N-doped carbon materials. For example, MacIntosh et al. [191] investigated N and P as dopants in graphene structure using XPS, SEM, TEM, and NMR. These are the primary analysis techniques widely used to characterize N-doped porous carbons. TEM is used to determine the lattice constant of char. STM (Scanning tunneling microscopy) is also used to understand N-functionalities on char as it is a powerful tool for investigating char electronic properties. XPS is the standard for quickly quantifying C, N, O, and other elements on the char's surface. Ayiania

et al. [192,193] used first principle-based models of various poly-aromatic structures containing N-functionalities to inform the deconvolution of XPS and Raman spectra of N-doped chars. Raman spectroscopy is used to quantify graphitic content. Besides Raman, FTIR (Fourier transform infrared) can be used to identify the chemical functional groups on N-doped char. X-ray absorption spectroscopy (EXAFS) can be used to characterize the poly-aromatic structure of char according to the mechanism of multiple scattering resonances. X-ray spectroscopy (EDS) uses an electron microscope to determine the elements' molar fraction on the char's surface. Gas adsorption or BET analysis is used to measure specific surface area, pore size/distribution, and porosity of porous materials such as biochars.

Hyphenated techniques such as TG-FTIR-MS and Py-GC-MS have been reported to monitor N-species' evolution during pyrolysis [84] [195]. These analytical tools can study intermediates and the transformation of N species under different heating rates. [95] studied the evolution of N-containing organic compounds during the pyrolysis of sewage sludge. The identified compounds were heterocyclic-N, amine-N, and nitrile-N. Zhou et al. [104] reported the distribution of N-containing products during the pyrolysis (300–900 °C) of sewage sludge using Py-GC/MS and Py-FTIR. These studies confirmed that N-transformation during pyrolysis could be monitored by using hyphenated techniques. A more detailed description of analytical methods used to characterize N-doped chars can be found elsewhere [24,196].

3. Applications of N-doped carbons

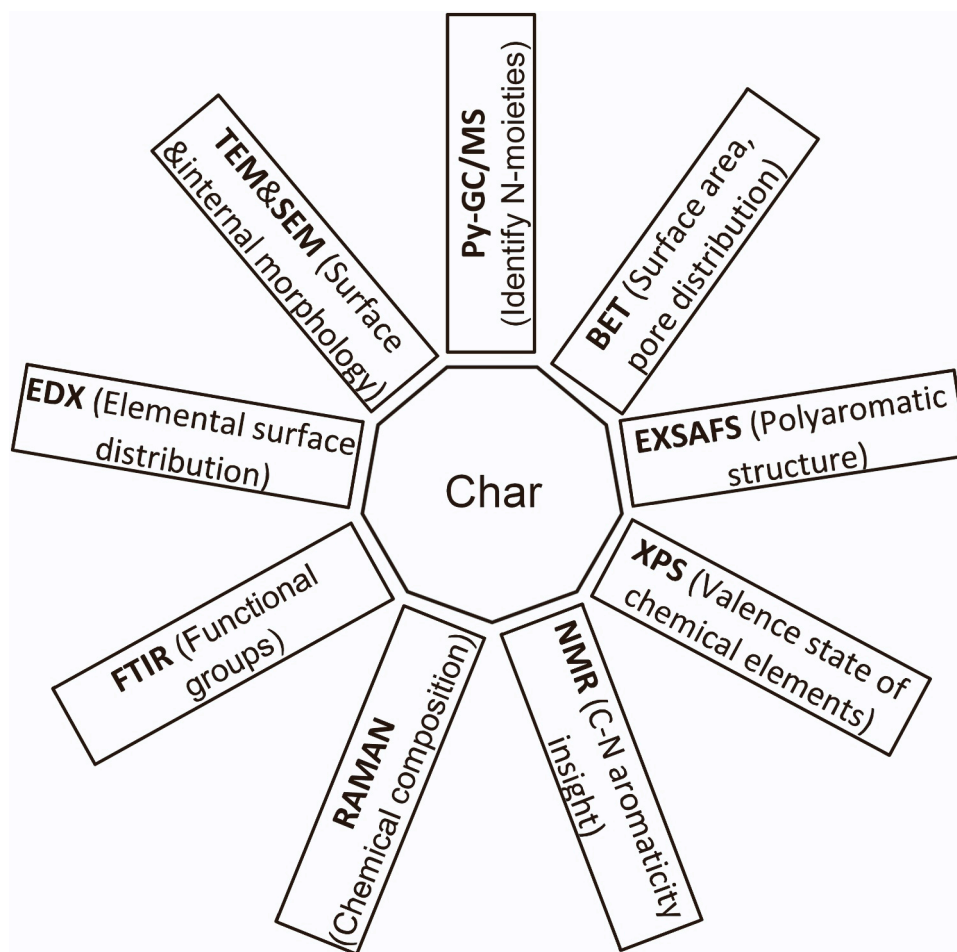N doping on carbon structure can efficiently alter carbon's intrinsic

Fig. 9. Methods for char characterization
Adapted from Wang and Wang [194].

optical and electrical properties. Compared to pristine C-structure (neutral, with poor reactivity), the N-doping improves carbon nanomaterials' surface energy and chemical activity and, thus, conductivity and electrocatalytic properties. Also, the prepared porous N-doped nanocarbon showed metal-free catalysts in organic synthesis reactions [197]. Also, the N-doped char shows adsorption capacity because of its tunable surface area and large surface functional groups. The adsorption of organic contaminants mainly depends on the electrostatic force, chemisorption, and π-π interactions. The N-doped char can also be used as a soil amendment that improves soil fertility. This section discusses some applications of N-doped carbon material.

3.1. Adsorption

Preparing N-doped porous carbons (NdPC) from unique, cheap, and abundant protein-containing biomass could make these carbons suitable adsorbents for treating acid and other poisonous gases. Also, the magnetic chars from waste biomass can be sorbents for other contaminations. Adsorption is the primary mechanism to remove organic pollutants and heavy metals from gases using chars. Adsorption also plays a vital role in catalysis. The main paths for char adsorption are an electrostatic attraction, pore-filling, π-π electron donor-acceptor interaction, H-bonding, complexes adsorption, hydrophobic interactions, precipitation, and partition of uncarbonized carbon [198-200] discussed the mechanisms for removing organic pollutants by several types of char. Functionalization of carbon materials that modify the carbon surface as adsorbents is responsible for adsorption capacity [201]. Introducing various functional groups (heteroatoms) into carbon materials via grafting and modifying carbon materials' surface chemistry is crucial for enhanced adsorption capacity. Among the typical modification processes (i.e., oxidation, nitrogenation, and sulfuration), nitrogenation is the most popular technique for generating N-containing functional groups. The introduction of N onto carbon materials significantly enhances its surface's polarity, increasing its specific interaction with adsorbates. It is believed that the heteroatoms bonded to the surface of carbon (N functional groups) modify and improve the surface properties of carbon adsorbents (e.g., specific surface area, pore-size distribution, and pore volume) as well as enhanced structural stability [202].

In general, several mechanisms work under unique environments to remove specific contaminants. Chemisorption (i.e., ion exchange, surface complexation, and precipitation) plays a more key role in removing heavy metals from aqueous solutions than physisorption (electronic interaction and physical adsorption). In addition, the NH_2 and NH functional groups, along with O-containing functional groups on the carbon surface, are also responsible for heavy metals removal [87]. Physical sorption occurs between these two positively charged amino groups in an aqueous medium and the delocalized cloud of electrons associated with aromatic groups on the surface of carbonaceous chars. This creates cation-π interactions with the C=C aromatic bonds. Furthermore, the presence of primary N functional groups can improve the adsorption of acidic gases due to the solid π-π exchange between the gases' quadrupole moment and the polar site associated with N functional groups [203].

Mood et al. [204] studied the use of N-doped char as an adsorbent for phosphate removal from aqueous solutions with positive results. The same research group studied the synergetic effects of N and Mg co-doped engineered chars from different feedstocks (e. g., digested diary fiber, wheat straw, and Douglas Fir). Introducing metallic ions (Ca and Mg) stabilized the N fixation, and P adsorption significantly increased Mood et al. [205]. Han et al. [206] synthesized suitable N-doped activated carbon materials from renewable leather wastes. The prepared N-doped carbon materials via KOH activation showed a high surface area and increased surface N species. The large surface pyridinic N in the char surface served as an excellent adsorbent to remove phenols (up to 282 mg/g).

N-doped chars can also be used to remove antibiotics. Antibiotics are present in municipal sewage discharges, livestock manure, slurry, and pharmaceutical wastewater [207]. Chitosan-derived chars are efficient adsorbents to remove tetracycline from water. Huang et al. (2020) studied the performance of ball milling chars from bagasse, bamboo, and hickory chips to remove antibiotics from wastewater treatment plants. Because of the N-doped chars' excellent physiochemical and adsorptive properties, these chars offer potential as remediates for various potential emerging contaminants. [164]studied the use of plant biomass (sawdust) as a carbon source, urea-phosphate as an activator, and N to prepare adsorbent char. The one-step synthesis showed a microporous structure and N-rich functional group (pyridinic, pyrolytic-N), which exhibited good CO_2 and H_2S adsorption capacity. N-containing functional groups on the char provide more active sites for CO_2 adsorption [208]. Additionally, the presence of N-atoms' presence on micropores during synthesis promotes acid-base interaction between carbon and CO_2 molecules, resulting in CO_2 adsorption. [209] synthesized walnut shell-based NdPC with urea modification and KOH activation, which showed CO_2 adsorption of 14.03 mmol/g. Similarly, [189] reported coconut shell-derived NdPC with CO_2 uptake of around 4.8 mmol/g. The work showed that the N moieties in char, such as N-5, N-6, and N-Q, are beneficial to CO_2 adsorption, but N-5 contributes a much higher CO_2 adsorption than N-6 and N-Q. Researchers have proposed the reaction mechanism of N-containing char surface and CO_2 at the molecular and atomic levels via density functional theory (DFT) [210].

Besides CO_2, the adsorption capacities of NdPC materials to other gases such as H_2S, SO_2, NO, and NO_2 have also been reported. Researchers studied the H_2S adsorption mechanism and reported that introducing N-species in char structure is responsible for H_2S removal. [211] showed the role of thermally stable groups such as N-Q and N-6 (available in N-doped carbon) for H_2S adsorption. The process of SO_2 removal via adsorptions has also been studied by Shao et al. [212]. The N-doped carbon showed maximum adsorption capacities from 57.8 to 156.2 mg/g compared to normal chars. Since NO and NO_2 are the two most common N oxides in nature, the capabilities of NdPCs were also evaluated to remove/absorb these gases. Therefore, incorporating N (N-groups) in the carbon structure positively impacts the capacity of NdPC to clean acid gases (i.e., to remove CO_2, H_2S, SO_2, and NO_2 from gases).

3.2. Uses of N-doped and N-metal-doped chars as catalysts

3.2.1. Uses of N-doped chars

Catalysts are essential in many fields, such as fuels production, fine chemicals processing, and novel synthesis processes [213,214,120]. Carbonaceous materials have received much interest as catalysts due to their eco-friendly and stable nature, cost-effectiveness, and availability (Dharecently). Carbon-based non-metallic materials are sustainable alternatives to metal-based catalysts. The structure of carbon-based catalysts and functional groups (e.g., O-containing groups), defects, and persistent free radicals play crucial roles in catalytic reactions [215]. The performance of carbon-based catalysts could be enhanced by modification methods, such as introducing specific metals to the structure of carbon-based catalysts [14].
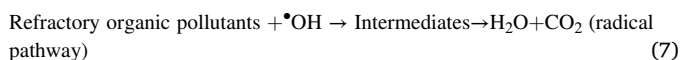
Although the modification of carbon-based materials through metal loading is expected to increase the catalytic activity of carbon-based catalysts, the high price of some metals and possible secondary contamination resulting from metal leaching could limit this approach [216]. Introducing N to the structure of carbon-based materials can alter and improve electronic properties, such as easy availability of lone pairs of electrons, charging, and polarizability [217]. N-doped chars are metal-free catalysts with a unique ability to tune electronic activity [218-220]. N insertion can create carbon defects, increasing the substrate's edge exposure and enhancing the catalytic activity [219]. Therefore, N-containing biomass precursors can be great chemical catalysts and catalysts supports for various reactions [221]. Table 5 shows

Table 5
Works on the application of N-doped catalysts in chemical reactions.

N-doped catalyst preparation	Reaction	Application	Reference (s)
Prepared by using chitosan and melamine	Oxidation-Reduction reaction (in alkaline media)	Efficient electrocatalytic activity	Rybarczyk et al. [223]
Chemical vapor deposition method (Fe (NO$_3$)$_3$ on Y-Al$_2$O$_3$)	Selective oxidation (H$_2$S to S)	Metal-free catalyst	Chizari et al. [224]
Cellulose followed by immunization (slow carbonization)	Wet oxidation process	Removal of phenolics from aqueous solutions	Tews et al. [225]
Bamboo shoot (8 wt %N)	Conversion of N$_2$ to NH$_3$	Electrocatalyst	Li et al. [83]
Pyridinic N	Oxidation-reduction reaction	Superior metal-free catalyst	Lv, Qing, et al. (2018)
Metal nanoparticles supported on N-doped carbon.	Catalytic reactions	Hydrogen production	David et al. (2019)
Biomass-derived electrocatalyst activated by KOH	Oxygen reduction reaction	Fuel cells and metal-air batteries	Kaare et al. [226,227]
Chitosan (by HTC) Pd-loaded N-doped carbon	Selective hydrogenation	Phenol to cyclohexanone	Wu et al. [228]
Glucose+Al(NO$_3$)$_3$ (wet impregnation followed by thermal treatment) supported ZnO.	Transesterification	Synthesis of ethyl methyl carbonate	Sun et al. [112]

some examples of the uses of N-doped char as catalysts. Atomic sizes of N and C are similar, facilitating the replacement of C with N in carbon-based materials without significant lattice distortion [222]. The difference in electronegativity of C and N causes defect structures in C-based materials and high electron transfer capacity, which enhances the catalytic performance of C-based materials.

In N-doped char, delocalized π electrons from sp^2 C and N species and ketonic groups could donate and transfer an electron to activate oxidants (e.g., peroxymonosulfate) and generate radicals such as SO$_4^{\bullet}$ and $^{\bullet}$OH, which oxidize refractory organic pollutants into less toxic compounds and could further mineralize refractory organic pollutants into CO$_2$ and H$_2$O via radical pathways, following Eqs. (2)–(7) [229].

$$HSO_5^- + e^- \rightarrow {}^{\bullet}OH + SO_4{}^{2-} \tag{2}$$

$$HSO_5^- + e^- \rightarrow SO_4^{\bullet} + OH^- \tag{3}$$

$$SO_4^{\bullet} + H_2O \rightarrow {}^{\bullet}OH + SO_4^{2-} + H^+ \tag{4}$$

$$HSO_5^- \rightarrow SO_5^{\bullet} + H^+ + e^- \tag{5}$$

Refractory organic pollutants $+ SO_4^{\bullet-} \rightarrow$ Intermediates$\rightarrow SO_4{}^{2-} + H_2O + CO_2$ (radical pathway) (6)

Refractory organic pollutants $+ {}^{\bullet}OH \rightarrow$ Intermediates$\rightarrow H_2O + CO_2$ (radical pathway) (7)
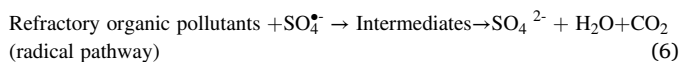
Refractory organic pollutants can also be oxidized through non-radical pathways in the presence of N-doped carbon-based catalysts. In the non-radical pathway, refractory organic pollutants are degraded through direct electron transfer without generating free radicals [229, 185,186]. Several studies have used N-doped porous carbon as a catalyst for the oxidation of organic pollutants. [230] studied N-doped reduced graphene oxide as a bifunctional catalyst for removing bisphenols from water. The study showed that N-doped enhanced char's adsorption and catalysis ability (via synergistic effect between adsorption and catalysis). Tews et al. [225] studied the N-doped char as a catalyst for the wet oxidation of phenol-contaminated water and proposed a reaction

mechanism. The results suggested that adding N functional groups on the char surface can produce a primary character that enhances electron transfer. Also, the high concentration of pyridine groups on char catalyst promotes the propagation of hydroxy free radicals. Furthermore, the dipyridine group was most energetically favorable in binding and splitting O$_2$. Guo et al. [231] reported that the carbon atom was modified as Lewis's base after successfully incorporating N onto the char structure (in pyridinic-N). Hence, the pyridinic group promoted electrocatalytic performance in the redox reaction. Liang et al. [232] reported that the graphitic-N was responsible for the high oxygen reduction reaction. Introducing the pyridinic N group to C-based catalysts could improve the catalytic activity for the oxygen reduction reaction (ORR) [28]. Pyridinic nitrogen can donate π electrons to electron-poor reactions such as ORR [226,227]. N doping causes the increase of positive charge density on C atoms and affects O$_2$ chemisorption, which could weaken the O-O bond and promote ORR [226, 227,23].

N-doped carbon (char) based catalysts could be great solid acid/base catalysts for esterification and transesterification reactions. The biomass-based solid acid catalyst derived from biomass carbonization followed by ammonification and sulfonation can be significantly substituted as green catalysts in biofuel synthesis [233]. N-doped carbon, as a catalyst, changes the surrounding electronic environment, showing catalytic activity of selective oxidation reaction [25,234]. Ma et al. introduced nonmetal elements (urea for N, sodium dodecyl sulfate for S), transition metal elements (MnSO4 for Mn, FeSO4 for Fe), and alkali/alkaline-earth metal elements (CaCl2 for Ca, NaCl for Na) to char structure to evaluate their catalytic performance in trichloroethylene reduction with a layered iron (II, III) hydroxide as reductant [14]. The results showed that N-doped char exhibited the highest catalytic activity among different modification methods used in the study, compared to other modified chars (trichloroethylene dechlorination rate increased by 3.5) (Ma et al., 2022).

3.2.2. Uses of N-metal-doped carbons

Interest in using carbon nanostructures doped with transition metals and N for catalyst applications have been reported for a relatively long time because it was found that C nanostructures doped with Bo, N, and other atoms change electronic properties or chemically activate local sites of the carbon nanostructure [235]. However, recent years have witnessed an emerging interest in nitrogen and metals co-doped char for environmental and energy applications and as catalysts [236,237,220, 238]; with promising results to replace conventional metal- or metal-oxides-based catalysts (Shi et al., 2020). Metals commonly used for this purpose include Fe, Cu, Mn, Co, and Ni [237]. Fe and N co-doped char exhibited a cost-effective catalyst with excellent catalytic performance and activation oxidation stability [229,239]. Moreover, magnetization appears as a good strategy for separating and recycling the catalyst [240]. You et al. [240] synthesized a magnetized N-doped char catalyst to activate peroxymonosulfate for the degradation of ciprofloxacin hydrochloride. Results showed that, after 80 min of reaction, the degradation efficiency of ciprofloxacin hydrochloride reached 92.6%, and SO$_4^-$·,·OH, and ^{1}O$_2$ participated in the degradation of ciprofloxacin hydrochloride.

Metal and N co-doped carbon (N-metal-doped) catalysts are expected to replace noble metal catalysts for the electrochemical reduction of CO$_2$ into CO. The metal and pyridine sites showed catalytic activities in the CO$_2$ reduction reaction [241]. CO can be further converted to CH$_4$ in the presence of Fe-N carbon [241]. Conversion of CO$_2$ to fuels could be a promising strategy for reducing CO$_2$ emissions in the air and fighting global warming [242]. Tripkovic et al. studied the catalytic activity of the metal-functionalized porphyrin-like structures toward the electrochemical reduction of CO$_2$ and CO to methane or methanol. The Rh−porphyrin-like functionalized graphene exhibited an excellent catalyst activity for producing methanol from CO [243].

Zhong et al. [238] produced a rod-like N-Cu-doped carbon for O

reduction reactions (ORR) in microbial fuel cells with air cathodes. The power density of these cathodes was almost twice that of the control (activated carbon) (i.e., 1760 mW/m^2). Homogeneous distribution of Cu, N, and C was observed in the rods, which resulted in abundant active sites for electrocatalytic reactions. Moreover, the high surface area of the catalyst (1398 m^2/g) provided the required access and active adsorption sites for O in ORR. In another work, Ji et al. [236] reported that the performance of biofuel cells can be improved by using N-Fe-doped carbon nanotubes. Therefore, both Cu and Fe offer the potential for busting the performance of biofuel cells. The effectiveness of using Fe^0/Fe$_3$C nano-particles for co-doping N-doped carbons for environmental applications has been reported by [244]. The co-doped carbon can remove Carbamazepine from wastewater entirely in a few minutes, suggesting that N-Fe-doped chars offer practical interest for wastewater remediation.

3.3. Electrochemical devices (energy storage devices)

Solar and wind energy are promising green energy sources. However, medium, and large-scale energy storage facilities are necessary for better synchronizing the production and use of electricity produced using these sources. As efficient energy storage systems, Li-ion and Na-ion batteries can be used for energy storage (e.g., at a commercial scale and in electric vehicles). However, the high cost and shortage of sources (e.g., Li) may restrict their economic viability [26,245]. The conversion of C-based materials (e.g., cellulose and biomass) to energy-related materials, including energy storage, has been investigated intensively, with promising results [87,245]. Compared to transition metal oxides, carbonaceous materials offer low cost, high conductivity, environmental friendliness, and inertness [27]. Supercapacitors are high-power storage devices in which N-doped carbon materials are desirable materials. Recent reviews by [246] and [247] thoroughly revised the advantages of using N-doped graphene carbon for energy storage. In particular, N-doped carbon materials offer potential use in supercapacitors [248]. Table 6 shows some works involving studies on N-doped carbon for supercapacitors.

In supercapacitors, the charge storage mechanism is the electrostatic attraction of electrolyte ions onto the surface of the electrode material. N and P's main surface functional groups provide another feature of charge storage via electrochemical redox reactions. Industrial biomass residue, algal biomass, sewage sludge, livestock manure, and poultry litter are rich in heteroatoms (N and P) and heavy metals (Cu, Zn). The thermal processing of these wastes to char helps immobilize these heavy metals in the char matrix. Thus, chars with these transition metals can provide charge storage properties as energy storage devices (i.e., supercapacitors). However, it is necessary to explore innovative research methods from renewable resources by controlling N configuration in the carbon structure in the future.

3.4. Application of N-doped carbon materials in biology and biomedicine

Carbon nanomaterials are of interest in various biomedical applications such as sensing and detection, photothermal therapy, and drug delivery due to the exceptional properties (e.g., biocompatibility and biodegradability) of these materials [261,262]. Heteroatom doping (e.g., N doping) can modify the photophysical properties of carbon-based materials (e.g., carbon dots) and improve their performance in biomedical applications [262]. N-doped carbon dots (N-CD) has a lone pair of electrons on N and free radicals on their surface, which makes them effective antimicrobial agent with high efficacy and low cytotoxicity [263,264]. Saravanan et al. synthesized N-CD, and the results indicated that N-CD had antimicrobial properties against Gram-negative Escherichia coli and Gram-positive Staphylococcus aureus [264].

N-doped carbon-based quantum dots have attracted considerable attention for their application for Photodynamic therapy (PDT), which is used to treat diseases such as cancer [50]. PDT is a photochemical

Table 6

Works on N-doped carbons for supercapacitors reported in the literature.

N-doped carbon preparation	Characteristics/ Applications	Reference (s)
Carbonization of waste lotus stem pyridine(N-6) and pyrrolic (N-5)	High-performance supercapacitor	Yan et al. [23]
Prepared by using N-containing salt (tetrasodium salt of EDTA) (Carbonization)	High capacitance	Xu et al. [139,249]
3D N-doped graphene (Carbonization)	High power density	Elessawy et al. [250]
Prepared by using soybean waste residue via HTC	Excellent capacitance performance	Ferroeso et al. [251]
Prepared by using Silk cocoon (Animal-based wastes)	High electron density (with specific capacitance)	Long et al. [252]
Pine nut with melamine as N precursor	Superior capacitance	Guan et al. [253]
Wheat straw (Melamine as N-source), (Carbonization)	High capacitance performance	Liu et al. [254]
Agriculture waste tobacco (Melamine) and molten zinc salt	High specific capacitance (346 F/g)	Liu et al. [158]
Cornhusk, human hairs, egg white (as C and N precursors)	Supercapacitor performance	Song et al. [255]; Qian et al. [175]; Li et al. [256]
Acorn shells (carbonization/ activation)	Ultrahigh capacitance supercapacitors	Gopalkrishnan et al. [254]
Peanut shells (melamine as the N source)	Lithium-ion batteries	Liu et al. [257,258]
Lotus parts (leaves, flowers, fruits, stems) under simple carbonization	High specific capacitance (160 F/g)	Atchudan et al. [248]
Yak dung (Carbonization at 800 °C)	High specific capacitance (346.3 F/g)	Hu et al. [259]
Chitosan (activation with KHCO3) followed by HTC	High specific capacitance (406 F/g)	Tong et al. [208]
Poultry litter (activation with KOH) followed by carbonization	High specific capacitance (229 F/g)	Pontiroli et al. [260]

reaction between light and photosensitizing agents in which reactive oxygen species (ROS) are generated and damage cancer cells and pathogens [50]. N-doped carbon-based quantum dots can act as photosensitizing agents to generate ROS and destroy cancer cells [50,265]. N-Doped carbon-based materials have been applied to drug-delivery systems. Burkert et al. successfully used N-doped carbon nanotube cups (NCNCs) for drug-delivery systems and anti-cancer immunotherapies [261]. In a study by Shi et al., highly fluorescent and super biocompatible N-doped carbon quantum dots were synthesized from lignin and successfully used for cellular imaging and intracellular irons detection [266]. Table 7 shows some works involving studies on N-doped carbon for applications in biology and biomedicine. The corresponding results show promising results to further the study of using N-doped carbon materials for medical applications.

3.5. Other applications. Soil application of densified N-doped char

Modern agroecosystems ensure food security and reduce greenhouse gas emissions [273,274]; [275]. Reducing synthetic fertilizer inputs and increasing C sequestration by adding char as a nutrient-rich N-fertilizer are alternative ways to reform agro-farming systems [276]. Although synthetic fertilizers have been used to improve crop yield productivity, the fertilizer commonly leaches into the environment and emits greenhouse gases. When N-rich materials such as manure, food waste, and yard waste are composted, special care is needed to ensure a target N:C ratio; otherwise, part of the N is released into the air in the form of ammonia and NO$_x$. Ideally, densified chars are not subject to fast microbial attack/oxidation [277]. Compared to composting and synthetic

Table 7
Works on N-doped carbons for applications in biology and biomedicine reported in the literature.

N-doped carbon	Characteristics/Applications	Reference (s)
N-doped carbon dots	Antimicrobial properties against Gram-negative Escherichia coli and Gram-positive Staphylococcus aureus	Saravanan et al. [264]
N-doped carbon-based quantum dots	Photodynamic therapy	Ansari et al. [50]
N-doped carbon nanotube cups	drug-delivery systems and anti-cancer immunotherapies	Burkert et al. [261]
N-doped carbon quantum dots	Cellular imaging and intracellular irons detection	Shi et al. [267]
N-doped carbon dots	Antimicrobial properties and skin permeability	Demirci et al. [263]
N-doped graphene quantum dots	Nucleus-targeted drug delivery	Ju et al. [265]
N-doped carbon dots	Bioimaging applications	Atchudan et al. [268]
N-doped carbon dots	Bioimaging applications	Wang et al. [269]
N-doped carbon quantum dots	Bioimaging applications	Guo et al. [270]
N-doped carbon nanodots	Drug delivery and bioimaging applications	Gomez et al. [271]
N-doped carbon quantum dots	Fluorescence sensing, bioimaging application, and anti-counterfeiting	Tan et al. [272]

Table 8
Works on N-doped carbons for other applications.

N-rich chars	Characteristics/Applications	Reference (s)
Wheat straw biochar	Reduce N leaching, keep soil N retention	Sun et al. [281]
Animal manure-based hydrochars	Photodynamic therapy	Ro et al. [277]
Biochar on application of reclaim water	Treating wastewater effluents	Yao et al. [33]
Manure biological and thermochemical treatment	Waste management and economic values	Awasthi et al. [278]
Manure and compost-based fertilizer	Improved crop yield compared to conventional composting	Evanylo et al. [279]
Cow manure-based chars	Increased soil pH and C-sequestration	Faye et al. [276]

fertilizers, the densified chars are made up of natural and unprocessed sources and are considered stable, sustainable, biodegradable, and environmentally friendly [278,279]. Many wastes containing N-rich molecules (e.g., dairy manure, animal waste, food waste, and yard waste), can be feasible cheap resources for densified chars. The addition of N-doped densified chars proved favorable for plant yield with a steady flow of nutrients for plant uptake via slow release of nutrients from the char. [33] reported that the uptake of N (ammonium/nitrate) and P (Phosphate) is decreased by char application, reducing their frequency in soil leachates by a high proportion. The densified N-doped char makes N more available for plant uptake for crops that cannot fix the N. In addition, the C content in the soil increases by applying char and helps to maintain the pH level by adding N-doped char (which is alkaline in nature). Compared to traditional composting, these densified chars are not subjected to fast microbial attack/oxidation. It has been reported that char effectively reduces the leaching of N in the form of NH_3 [280]. Table 8 shows some works on N-doped carbons for soil amendment and other applications.

4. Summary and future perspectives

N-doped carbon materials offer enormous potential in environmental remediation and electronic applications. The widely available protein-containing biomass and other N-containing biomolecules are promising sources for producing N-doped carbons. Their natural abundance and self-doping heteroatom content (N), resulting in chars with high specific surface area and porous structure, provide opportunities for several applications. Unlike external doping, co-carbonization avoids toxic chemicals by providing uniform doping using cost-efficient processes. N-doped carbon-based materials have been investigated as catalysis for gas adsorption and energy storage. However, mass production of these materials from renewable waste resources is necessary to expand the use of char at large scales, such as soil amendment, environmental remediation, and carbon sequestration. Since the N content of char is low, modulating the C:N ratio helps to fix N in the char structure and can be used as fertilizer. However, more work is needed to investigate the role of N-doped carbon in many organic synthesis reactions that could easily replace expensive, corrosive metal (acid-base heterogeneous) catalysts. Also, more efficient synthesis methods are needed to produce these materials from biogenic N and C sources, and more work is required to advance the analytical techniques (such as solid-state NMR and scanning tunneling microscopy-STM) and their interpretation for N-doped chars.

CRediT authorship contribution statement

Kalidas Mainali: Conceptualization, Writing – original draft, Writing – review & editing. **Sohrab Haghighi Mood**: Conceptualization, Writing – original draft, Writing – review & editing. **Manuel Raul Pelaez-Samaniego**: Conceptualization, Writing – review & editing. **Valentina Sierra-Jimenez**: Conceptualization, Writing – original draft, Writing – review & editing. **Manuel Garcia-Perez**: Conceptualization, Writing – original draft, Writing – review & editing.

Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Manuel Garcia-Perez reports financial support was provided by Washington State Department of Agriculture.

Data availability

Data will be made available on request.

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