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Review Paper

Transforming *Sargassum* into Valuable Solid Carbon Materials: A Review of Synthesis Methods †

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The valorization of *Sargassum* into valuable carbon materials is not only driven by the need to manage its massive accumulation on beaches every year, posing ecological, economic, and health challenges, but also by its unique composition rich in carbohydrates and bioactive materials, making it an ideal precursor for carbon-rich materials. Among various thermal transformation methods, hydrothermal carbonization, slow pyrolysis, and microwave-assisted methods (microwave-assisted hydrothermal carbonization and microwave-assisted pyrolysis) have been employed to convert *Sargassum* into solid carbon-based materials, such as hydrochar, biochar, carbon quantum dots (CQDs), and activated carbon. These materials have demonstrated a significant role in diverse applications, including water purification, soil enhancement, energy storage, catalysis, and biomedical fields. This review discusses these synthesis methods, essential pretreatment processes to prepare *Sargassum* before the synthesis processes, and posttreatment processes aimed at enhancing the carbon material properties. Finally, the work also discusses the challenges of *Sargassum* valorization and presents future directions. Given the scale of *Sargassum* overgrowth affecting coastlines in the Caribbean, Gulf of Mexico, and Atlantic Ocean, this review is significant in highlighting strategies for transforming marine biomass into high-value carbon materials and provides a comprehensive analysis of the existing studies on the conversion of *Sargassum* into carbon materials.

Keywords: *Sargassum* valorization, carbon materials, algal biomass, green synthesis, materials synthesis

1. Introduction

Sargassum (**Fig. 1(a)**), a genus of brown macroalgae commonly known as seaweed, plays a vital role in marine ecosystems and biodiversity. It forms large floating mats on the ocean's surface, creating unique habitats that support a diverse range of marine species (Van Tussenbroek et al., 2017). These mats carry a resident plankton population, composed of organic-rich microorganisms that sustain, nourish, and support the food web for a variety of marine species, inhabitants of *Sargassum* mats such as young fish, small invertebrates like shrimp and crabs, fungi, epiphytes, and juvenile sea turtles (Franks et al., 2012; Lobban et al., 1985; Van Tussenbroek et al., 2017; Yangthong, 2017). Beyond serving as a food source, the expansive *Sargassum* mats provide essential shelter and protection for their inhabitants from predators (Casazza and Ross, 2008; Coston-Clements et al., 1991; Witherington et al., 2012). These floating mats offer safe resting and hiding spaces, allowing species to grow and thrive in the open ocean (Coston-Clements et al., 1991). Some inhabitants, such as *Sargassum* fish and certain invertebrates, have evolved specialized adaptations for life within *Sargassum*, developing unique shapes and behaviors to increase their survival. Moreover, *Sargassum* contributes to global ecological balance through photosynthesis, consuming CO_2 and releasing O_2 into the atmosphere (Robledo et al., 2021). These combined roles make *Sargassum* an important part of maintaining healthy and balanced marine ecosystems. *Sargassum* is found across the globe, flourishing primarily in tropical and subtropical waters. It is abundant in the Atlantic Ocean, particularly the Sargasso Sea, a region in the North Atlantic near the Caribbean and east of the Florida Straits. Research indicates that the Sargasso Sea absorbs approximately 7 % of the world's net CO_2 emissions, underscoring its critical role in global carbon cycling (Milledge and Harvey, 2016). *Sargassum* primarily reproduces through vegetative growth and fragmentation, thriving in conditions with high nutrient levels, warm water, and ocean currents (Oxenford et al., 2021). Its growth is particularly noticeable during the spring and summer, fueled by water upwelling, in which deep, cold, and nutrient-rich waters rise to the surface, replenishing nutrients and driving *Sargassum* growth (Lapointe et al., 2021).

In recent years, *Sargassum* has increasingly invaded coastlines worldwide (**Fig. 1(b)**), forming massive accumulations along shorelines (Chávez et al., 2020). The first

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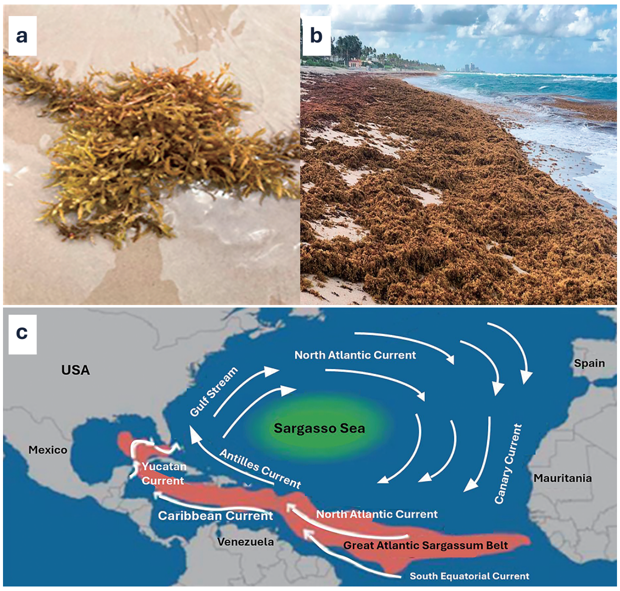

Fig. 1 **(a)** A photo of Pelagic *Sargassum*. **(b)** A photo shows *Sargassum* piled up on a beach in Palm Beach County, Florida (SciTechDaily, 2021). **(c)** The Sargasso Sea and Great Atlantic *Sargassum* Belt in the North Atlantic. Reprinted from López Miranda et al. (2021) under the terms of the CC BY 4.0 license. Copyright: (2021) The Authors, published by Frontiers Media S.A.

major *Sargassum* bloom in the central Atlantic was recorded in 2011, and over the years, the bloom developed into the "Great Atlantic *Sargassum* Belt (GASB)" extending from West Africa to the Caribbean Sea and into the Gulf of Mexico (**Fig. 1(c)**), spanning over approximately 3,000 km^2 area (Oxenford et al., 2021; Wang et al., 2019). The exact causes of the massive influx of *Sargassum* remain unclear, but several contributing factors have been proposed (Schmidt, 2023; Van Tussenbroek et al., 2017). A major factor is the nutrient runoff from the Amazon River, driven by deforestation and land-use changes, which enriches the Atlantic Ocean with nitrogen (N) and phosphorus (P), promoting *Sargassum* growth (Giffard et al., 2019; Van Tussenbroek et al., 2017). The movement of Sahara dust across the Atlantic from North Africa carries iron and essential nutrients that further stimulate *Sargassum* growth and expansion (Van Tussenbroek et al., 2017). Climate change and increased ocean temperatures cause upwelling of water, which enhances nutrient content and decreases sea surface temperature, creating favorable conditions for *Sargassum* growth (Wang et al., 2019). Since 2011, the Atlantic Ocean has experienced unusual wind patterns and increased storm activity, which further drives the spread and accumulation of *Sargassum* along the coastal regions (Oyesiku and Egunyomi, 2014; Wang et al., 2019), called *Sargassum* inundation event (SIE) (Trinanes et al., 2023). Forecasting SIEs to specific locations is extremely difficult because of the unpredictability of winds and ocean currents. However, the National Oceanographic and Atmospheric Association (NOAA)'s Coast Watch (NOAA, 2025) for the Atlantic region and the University of South Florida's Optical Oceanographic Lab have developed statistically based *Sargassum* Inundation Risk reports that predict the likelihood of SIEs in the Tropical Atlantic and the Gulf of Mexico (Putman et al., 2023).

The overabundance of *Sargassum* on beaches and along shorelines poses a significant threat to the coastal ecosystem, human health, and local economy (Van Tussenbroek et al., 2017; Williams and Feagin, 2010). Environmentally, dense *Sargassum* mats block sunlight from reaching the water column, inhibiting photosynthesis in corals and seagrasses, leading to their decline and disrupting the marine food web (Hoegh-Guldberg and Bruno, 2010; Paredes-Camacho et al., 2023). As *Sargassum* decomposes in shallow waters or on beaches, it depletes dissolved oxygen, releases excess organic matter, and emits harmful gases like hydrogen sulfide and ammonia, altering water chemistry and potentially causing acidification. These changes threaten marine ecosystems by damaging coral reefs and seagrass beds, causing fish and invertebrate die-offs, and disrupting sea turtle nesting and hatching (Hoegh-Guldberg and Bruno, 2010; Paredes-Camacho et al., 2023). Additionally, the release of these gases poses health risks to humans, including neurological and cardiovascular issues, nausea, and headaches. Economically, *Sargassum* accumulation harms tourism by making beaches unattractive, emitting a foul hydrogen sulfide odor, and obstructing public access to shores and boats, deterring visitors (Schmidt, 2023; Van Tussenbroek et al., 2017). Furthermore, it burdens coastal communities with clean-up costs. Local fisheries are also impacted due to the disruption of marine habitats and potential declines in fish stocks (Townhill et al., 2021). These combined impacts make excessive accumulation of *Sargassum* in the coastal areas adjacent to local communities a pressing issue for both the environment and society (Orozco-González et al., 2022; Robledo et al., 2021; Van Tussenbroek et al., 2017).

Governments of affected shorelines have implemented various strategies to mitigate the impact of *Sargassum* invasions (Chávez et al., 2020; López Miranda et al., 2021). These include installing floating barriers or booms (Oxenford et al., 2021; Robledo et al., 2021) to prevent *Sargassum* from reaching beaches, developing specialized beach cleaning equipment for swift removal and disposing of the *Sargassum* in landfills or valorizing collected *Sargassum* into valuable products, thus transforming an ecological challenge into a potential opportunity (Lee et al., 2023; Milledge and Harvey, 2016; Orozco-González et al., 2022). Disposing of *Sargassum* in landfills or redirecting it away from local beaches is neither a long-term solution nor the best way of managing this natural marine resource (López Miranda et al., 2021; Schmidt, 2023). Instead, transforming *Sargassum* into value-added materials or products presents a more viable approach, creating economic opportunities while mitigating its environmental impact. *Sargassum* has recently gained attention for its potential in "green synthesis" (Orozco-González et al., 2022) of carbon materials due to its high carbon content, making it an excellent precursor for producing carbon materials such as carbon quantum dots (CQDs) (Castañeda-Serna et al., 2022), hydrochar (Spagnuolo et al., 2023), biochar (Farobie et al., 2022), porous carbon (Jia et al., 2020), activated carbon (Francoeur et al., 2021), and graphene (Prakoso et al., 2024). These materials have wide-ranging applications in energy, energy storage, catalysis, environmental remediation, agriculture, and healthcare, positioning *Sargassum* as a valuable resource rather than a coastal burden.

In general, wet *Sargassum* contains 70–90 % water by weight, and the dry *Sargassum* biomass is mostly composed of organic and inorganic compounds and elements. After drying the *Sargassum*, its moisture content can range from 8.50 % to 18.80 % of its dry weight (Davis et al., 2021; Tonon et al., 2022). Organic compounds consist of carbohydrates (up to 68 %), proteins (up to 16 %), and lipids (up to 6 %) of the dry weight, along with small amounts of vitamins, minerals, and secondary metabolites (Orozco-González et al., 2022; Yangthong, 2017), including sterols, terpenoids, phenolics, and pigments such as chlorophyll and fucoxanthin (Holdt and Kraan, 2011; Peng et al., 2011). The ash content in *Sargassum* ranges from 9 % to 44 % of the dry weight and includes elements such as Ca, Al, Cl, Cu, Fe, Mg, Na, K, Mg, S, P, and Si (Devault et al., 2021; Rushdi et al., 2020), trace toxic elements such as Pb, As, and I, and metal oxides such as CaO, SO_3, and Na_2O (Rodríguez-Martínez et al., 2020). Among the commonly studied species, *Sargassum natans* and *S. fluitans*, the dominant pelagic types in the Atlantic, are rich in carbohydrates (alginate, fucoidan, and mannitol) but have low protein and lipid contents. Benthic species, such as *Sargassum horneri* and *S. muticum*, found in East Asia and Europe, respectively, contain higher phenolic content and, in some cases, substantial lignocellulosic components (Ai et al., 2022; Xu et al., 2013). While *Sargassum* species share similar overall components, their relative composition varies by species, location, and season (Davis et al., 2021; Duran et al., 2024). Studies show that *Sargassum natans* VIII contains higher phenolic and polysaccharide content than *S. fluitans* III, enhancing its potential for functionalized carbon synthesis (Vázquez-Delfín et al., 2021). Most pelagic species are low in lignin, simplifying processing, while certain benthic types like *Sargassum horneri* contain up to 27 % lignin, which can increase yield and aromaticity of the synthesized carbon material (Ai et al., 2022; Xu et al., 2013). This variability underscores the importance of species selection in optimizing the properties of carbon materials. The details of the composition of *Sargassum* and its different species are provided in the J-STAGE Data (https://doi.org/10.50931/data.kona.29672663).

Like other types of biomass, *Sargassum* can be converted into carbon materials using various thermal transformation methods, such as hydrothermal carbonization (HTC) (Sharma et al., 2020), pyrolysis (Dhyani and Bhaskar, 2019), microwave-assisted (MW) techniques (Gao et al., 2021; Luque et al., 2012), and advanced approaches like joule (Jiang et al., 2019), plasma (Wang et al., 2015), and laser (Ye R. et al., 2017) heating techniques (Zhang et al., 2021). These methods differ based on the type of precursor used, synthesis mechanism, process parameters, and the final products (Tobío-Pérez et al., 2022). During the thermal transformation processes, amorphous biomass precursors are decomposed into small molecular intermediates, which then undergo polymerization, aromatization, carbonization, and graphitization processes to obtain carbonaceous materials (Chen et al., 2020; Zhang et al., 2021). The process parameters play a crucial role in determining the yield, structural characteristics, and surface properties of the synthesized carbon materials, which can be tailored for various applications (Ao et al., 2018; Castañeda-Serna et al., 2022; Zhang et al., 2021). Among these methods, HTC, pyrolysis, and MW techniques are particularly preferred due to their versatility, efficiency, low operating costs, and simplicity. Herein, we aim to provide a comprehensive review of these three synthesis methods for synthesizing solid carbon materials from *Sargassum*. We describe their synthesis mechanisms, key process parameters, and the types of carbon materials produced. We also discuss the comparison between these methods and their products. Finally, we conclude with the challenges associated with the synthesis of carbon materials from *Sargassum* and provide a future outlook, focusing on the research needed to improve synthesis methods.

Pretreatment is a crucial step in preparing *Sargassum* before converting it into functional carbon materials. Once harvested, *Sargassum* pretreatment involves washing, drying, ensiling, grinding, cutting and chopping, sieving, and chemical treatment. These steps ensure the removal of impurities and prepare the *Sargassum* feedstock for optimized conversion to high-quality carbon materials (Maneein et al., 2018). The details of the *Sargassum* pretreatment procedures are provided in the J-STAGE Data (https://doi.org/10.50931/data.kona.29672663).

2. Synthesis of *Sargassum*-derived carbonaceous materials

This section presents an overview of the synthesis methods, including the synthesis mechanism, key parameters, such as temperature, reaction time, and reaction conditions, using the relevant studies, and the comparison among the synthesis methods. **Fig. 2** illustrates the various processes used to convert *Sargassum* into valuable carbon materials.

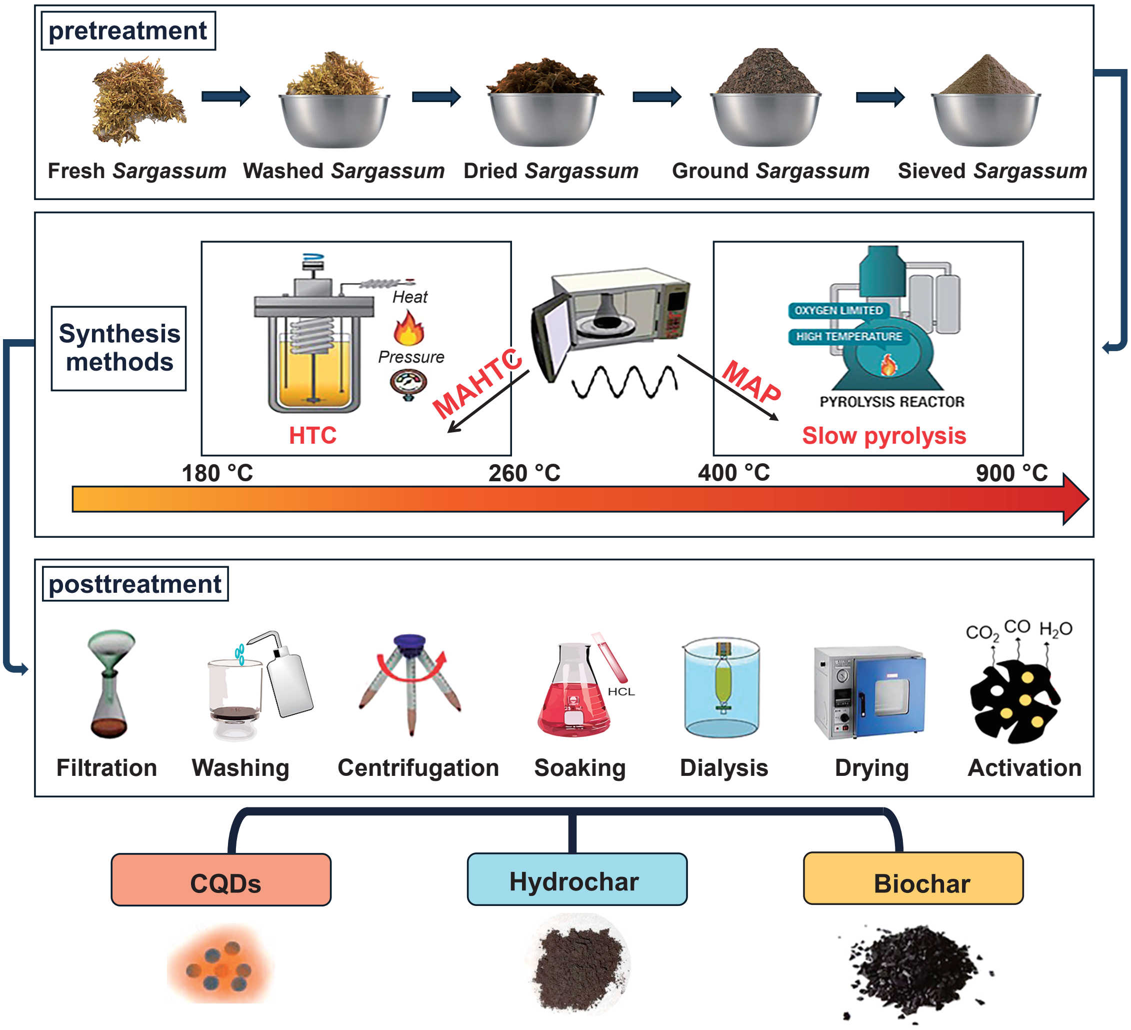

Fig. 2 Schematic of the complete process for converting *Sargassum* into carbon materials, including pretreatment methods, synthesis methods, and posttreatment methods.

2.1 Hydrothermal carbonization (HTC) method

The HTC process occurs in a water-rich environment under subcritical conditions, typically at temperatures between 180–260 °C and pressures ranging from 2 to 6 MPa, with reaction times varying from a few minutes to several hours (Funke and Ziegler, 2010; Soroush et al., 2024; Wang et al., 2018). Although wet *Sargassum* can be directly used in this process, it is commonly dried and ground into a fine powder to ensure a high surface area (SA) of *Sargassum* powder particles and uniform particle size, thereby promoting faster reactions and ensuring product uniformity (Castañeda-Serna et al., 2022; Spagnuolo et al., 2023). The process yields three product streams: 1) hydrochar, a carbon-rich solid material with enhanced surface functionality, accounting for approximately 50–80 % of the initial *Sargassum* biomass conversion into hydrochar; 2) a liquid-phase mixture of soluble organic compounds, including CQDs and inorganic species, representing about 5–20 % of the original biomass; and 3) a gaseous product, composed mainly of CO_2 and traces of CO, CH_4 and H_2, formed from the remaining fraction of the biomass (Ischia et al., 2022; Spagnuolo et al., 2023; Wang et al., 2018).

HTC involves a series of mechanisms, including hydrolysis, dehydration, decarboxylation, polymerization, and aromatization, to transform *Sargassum* into carbonaceous materials (Funke and Ziegler, 2010; Jin, 2014; Soroush et al., 2024). These reactions do not occur in a strict sequence but rather form a parallel network of different complex reaction pathways, which are divided into three main steps. A schematic of the HTC of *Sargassum* is constructed in Fig. 3. The first step is hydrolysis, a process that relies on the unique properties of water to act as a heat transfer medium and a catalyst at elevated temperatures and pressures to drive chemical reactions. As the temperature rises under subcritical conditions, water's dielectric constant decreases due to the weakening of hydrogen bonds, while the ionization constant increases, promoting the dissociation of water molecules into hydroxide ions (OH^-) and hydronium ions (H_3O^+) (Kritzer and Dinjus, 2001; Marcus, 1999). This enhanced ionization initiates hydrolysis reactions, where

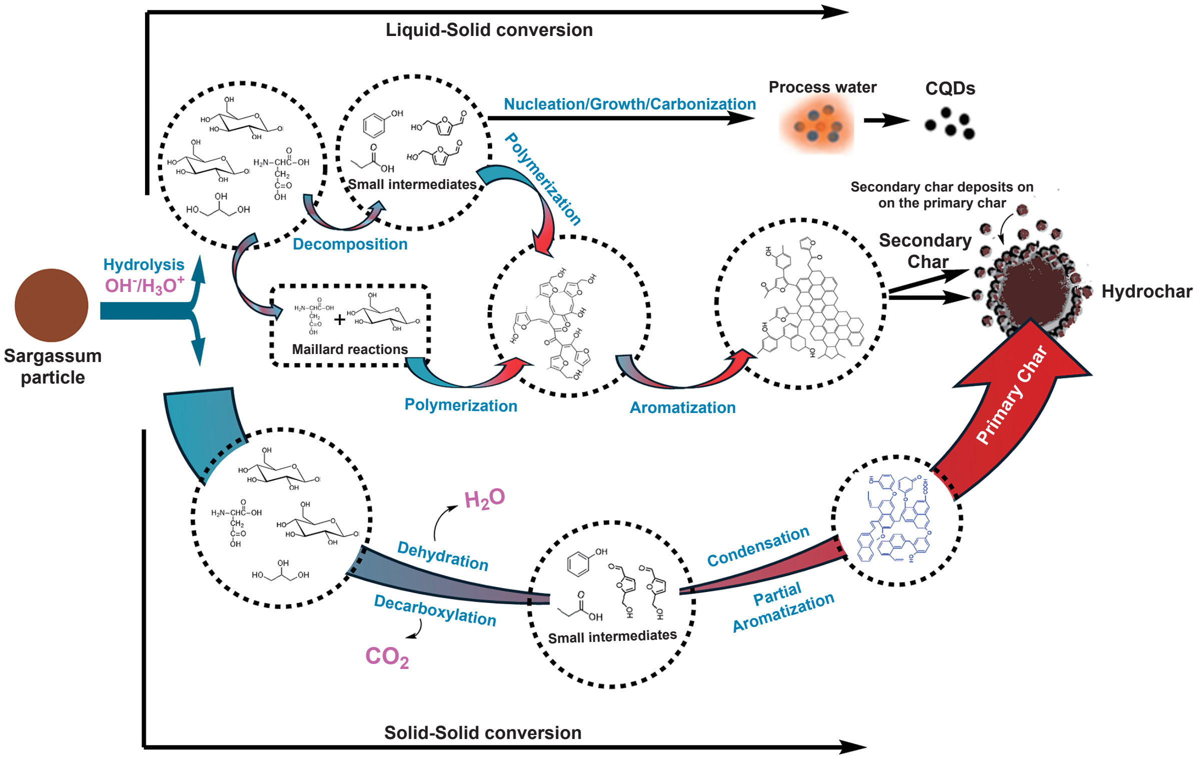

Fig. 3 Schematic of the formation mechanism of carbon materials (hydrochar and carbon quantum dots, CQDs) through the HTC process.

these ions break down the complex organic components in *Sargassum* (carbohydrates, proteins, and lipids), resulting in smaller reactive intermediate molecules (Kambo and Dutta, 2015b; Soroush et al., 2024; Zhang et al., 2021). Carbohydrates in *Sargassum* (alginate, fucoidan, laminarin, and mannitol) are hydrolyzed into simpler sugars such as glucose, fucose, and mannose. H_3O^+ ions protonate glycosidic bonds, enhancing their cleavage, while OH^- ions facilitate sugar ring opening, accelerating the decomposition process (Zhang et al., 2023). These sugars further degrade into smaller reactive intermediates, including furans, organic acids, and aldehydes (Kruse and Dinjus, 2007; Reza et al., 2014; Yang, 2015). Similarly, proteins undergo hydrothermal hydrolysis, initially breaking down into peptides such as glutamine, asparagine, and tyrosine. These peptides are further cleaved into amino acids such as glutamic acid, alanine, and glycine. H_3O^+ ions protonate peptide bonds, promoting their fragmentation, while OH^- ions deprotonate side chains, facilitating the generation of smaller nitrogenous compounds such as amines, organic acids, and phenols (Alonso-Riaño et al., 2024; Biller and Ross, 2011; Kruse and Gawlik, 2003). Lipids in *Sargassum* primarily consist of triglycerides and glycerol, which also undergo hydrolysis. Triglycerides, composed of three fatty acids linked to a glycerol backbone, are hydrolyzed into free fatty acids such as palmitic, stearic, and arachidic acid, along with glycerol (Biller and Ross, 2011; Dunne et al., 2015; Kruse and Dinjus, 2007). Glycerol may further decompose into smaller molecules like acetic acid, ketones, and glycols. H_3O^+ ions catalyze the cleavage of ester bonds in triglycerides, while OH^- ions break the carbonyl carbon in the ester linkage of glycerol and fatty acids (Biller and Ross, 2011; Dunne et al., 2015; Kruse and Dinjus, 2007). Step two includes intermediate reactions: dehydration, decarboxylation, and Maillard reactions. Once hydrolyzed, these intermediates undergo a variety of reactions within the aqueous medium. Sugars and sugar derivatives dehydrate to form furfural and HMF (5-hydroxymethylfurfural), which serve as pivotal precursors for polymerization and carbonization (Ischia et al., 2022; Zhuang et al., 2019). Concurrently, decarboxylation reactions simplify the molecular structure by removing carboxyl groups, leading to the release of CO_2 as the primary gaseous byproduct, along with traces of H_2, CO, and CH_4 (González-Arias et al., 2024; Karayıldırım et al., 2008; Velebil et al., 2016). Proteins, when reacting with reducing sugars in the same environment, engage in Maillard reactions, which are non-enzymatic browning pathways that lead to the formation of nitrogen-containing heterocyclic compounds. These compounds eventually participate in the formation of hydrochar and carbon dots and contribute to their characteristic color and odor (Lucian et al., 2018; Zhuang et al., 2019). Step three includes polymerization and aromatization, which are subsequent mechanisms that transform these reactive intermediate molecules into more stable carbon-rich materials (Ischia et al., 2022; Zhuang et al., 2019).

Polymerization occurs through the condensation reaction of molecules containing hydroxyl, carbonyl, and carboxyl functional groups, forming larger polymeric structures through covalent bonding (Danso-Boateng et al., 2022; Prakoso et al., 2022; Wang et al., 2018). Aromatization rearranges larger molecules into stable aromatic ring structures. This process is driven by the need to maximize thermodynamic stability, as aromatic structures with conjugated double bonds are more stable than irregular polymer chains. The moderate temperatures and pressures used in HTC prevent complete carbonization, retaining some of the oxygen-containing functional groups (Funke and Ziegler, 2010; Nicolae et al., 2020; Zaccariello et al., 2022). While both hydrochar and CQDs originate from the same general synthesis mechanisms, their formation pathways diverge under hydrothermal conditions (Lucian et al., 2018; Zhuang et al., 2019). Hydrochar is formed via two main pathways based on the physical phase of reactions: (A) primary char formation (solid-solid conversion) (Nicolae et al., 2020; Soroush et al., 2024). In this pathway, some of the original *Sargassum* biomass that remains insoluble (i.e., undissolved) undergoes direct thermal transformation through dehydration, condensation, decarboxylation, and partial aromatization (Ischia et al., 2024; Volpe and Fiori, 2017). This route leads to the formation of primary hydrochar, which retains some morphological features of the original *Sargassum* (Knezevic et al., 2010; Titirici and Antonietti, 2010); (B) secondary char formation (liquid-solid conversion), where the soluble organic intermediates (i.e., Furfural, HMF) in the aqueous phase undergo condensation, polymerization, and aromatization reactions (Soroush et al., 2024; Titirici et al., 2012). These reactions result in secondary char, also referred to as “coke,” which appears as microspheres or globular carbonaceous particles (Karayıldırım et al., 2008; Volpe and Fiori, 2017). In parallel, reactive species such as amino acids and sugars interact to yield nitrogen-doped carbon structures through Maillard reactions, evidenced by the brown coloration of hydrochar (Zhuang et al., 2019). The formation of secondary chars is favored at higher temperatures, longer residence times, and higher biomass loadings, where dissolved organics reach supersaturation and precipitate as solid carbon spheres in nano/micro-scale deposits onto primary char surfaces (Ischia et al., 2022; Titirici and Antonietti, 2010). The mechanism explained here is based on non-lignocellulosic biomass and not based on *Sargassum* directly, as studies on the hydrothermal carbonization of *Sargassum* are lacking. **Table 1** represents the comparison between the properties of primary and secondary hydrochar derived from biomass through the HTC synthesis method. Hydrochar derived from *Sargassum* has diverse applications due to its porous structure, surface functionality (hydroxyl, carboxyl, and amine groups), and thermal stability (Chambers et al., 2024; González-Fernández et al., 2024). González-Fernández et al. (2024) demonstrated that hydrochar derived from *Sargassum* biomass exhibits significantly high adsorption capacities for heavy metals, specifically Cd(II) and Pb(II), due to the presence of functional groups on the hydrochar surface. Their study reported maximum adsorption capacities of approximately 140 mg/g for cadmium and 340 mg/g for lead, exceeding those of many previously studied similar adsorbents (González-Fernández et al., 2024). Chambers et al. (2024) demonstrated the successful valorization of *Sargassum* into hydrochar via hydrothermal carbonization followed by KOH activation. The resulting hydrochar exhibited excellent physicochemical properties, including a high SA (up to 1404.09 m^2/g) and abundant oxygen-containing functional groups (up to 452.82 μmol/g). These features enabled an exceptional adsorption capacity for methylene blue, reaching 714.29 mg/g at 37 °C (Chambers et al., 2024).

On the other hand, CQDs may form during secondary

Table 1 Comparison between the properties of primary and secondary hydrochar derived from biomass through the HTC synthesis method.

Property	Primary Hydrochar	Secondary Hydrochar
Formation pathway	Solid–solid conversion (retains initial biomass structure)	Polymerization of dissolved intermediates in the liquid phase (e.g., HMF, furans)
Morphology	Irregularly, retains initial sargassum particle morphology	Spherical microspheres (nano/micron-sized)
Carbon content	Low (retains O/N from original biomass)	High (extensive aromatization, dehydration)
Oxygen content	High (retains –OH, –COOH groups)	Low (more hydrophobic)
Aromaticity	Moderate (partial condensation)	High (condensed polyaromatic structures)
Thermal stability	Good (cross-linked but less graphitized)	Excellent (highly condensed carbon).
Surface functional groups	More polar (–OH, –COOH, $–NH_2$)	Fewer polar groups (more hydrophobic)
Formation conditions	Dominates at lower temperatures (180–220 °C), short residence times	Favored at higher temperatures (>220 °C), long residence times
Contribution to the yield	~60–80 %	~20–40 %

char formation in the liquid phase. Their formation mechanism, although not fully understood, appears to involve three key stages: First, small aromatic clusters nucleate from reactive intermediates like furans and phenolic compounds, released during *Sargassum* breakdown (Ischia et al., 2024; Nicolae et al., 2020). Second, these nascent clusters grow through aggregation while being passivated by surface functional groups (–COOH, –OH) from the hydrothermal medium. Importantly, Maillard reactions between sugars and amino acids contribute to N-doped carbon structures that significantly enhance the fluorescent properties of CQDs (Das et al., 2019). Finally, the CQD structures develop from the most soluble molecular fragments that avoid incorporating into larger secondary char particles, undergoing gradual carbonization and surface functionalization under hydrothermal conditions (Kapitonov et al., 2018; Papaioannou et al., 2019). The resulting CQDs exhibit size-dependent photoluminescence, which is also tunable by the presence of surface functional groups and degree of carbonization (Paul and Kurian, 2021). *Sargassum* is rich in nitrogen (N) and sulfur (S), which enables the production of S- and N-doped CQDs without an external doping agent (Bressi et al., 2023; Godavarthi et al., 2017; Tian et al., 2021). Godavarthi et al. (2017) synthesized N-doped CQDs from *Sargassum fluitans*, with a quantum yield of 18.2 % and excellent fluorescent properties, making them suitable for sensitive DNA detection (**Fig. 4(a)**). Similarly, Tian and co-authors (2021) produced nitrogen–sulfur co-doped CQDs from *Sargassum carpophyllum*, demonstrating excellent photostability and high selectivity as fluorescent sensors for Cr(VI) and ascorbic acid detection (**Fig. 4(b)**).

Several key parameters influence the properties of the synthesized carbon materials via the HTC process, including reaction temperature, residence time, water-to-biomass ratio, reactor pressure, and initial *Sargassum* particle size (Nizamuddin et al., 2017; Pauline and Joseph, 2020). Among these, temperature, resident time, and particle size are the most critical, directly affecting the yield, carbon content, SA, porosity, and functionality (Heilmann et al., 2010; Heilmann et al., 2011; Xu et al., 2013). Reaction temperature determines the extent of biomass decomposition and the efficiency of carbon conversion (Pauline and Joseph, 2020). Higher temperatures enhance water ionization and provide thermal energy, accelerating organic decomposition (Nizamuddin et al., 2017; Yusuff et al., 2019).

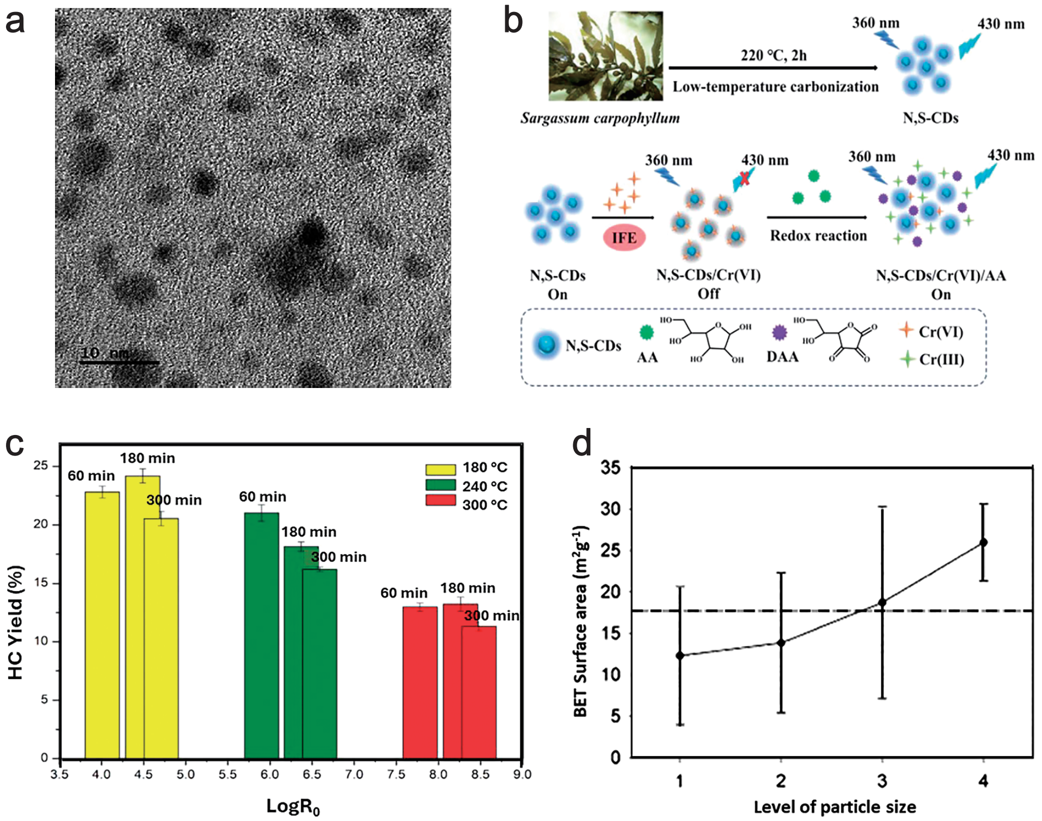

Fig. 4 **(a)** HR-TEM image of nitrogen-doped carbon dots (NCDs). Adapted with permission from Godavarthi et al. (2017). Copyright (2017) Elsevier. **(b)** Synthetic route of N,S-doped carbon dots (N,S-CDs) from *Sargassum carpophyllum* and detection mechanism of Cr (VI) and ascorbic acid (AA). Adapted with permission from Tian et al. (2021). Copyright (2021) Royal Society of Chemistry. **(c)** Percentage yield of hydrochar as a function of log R_0, where R_0 is the severity factor used to quantify the combined effects of temperature and residence time on the reaction. Adapted with permission from Spagnuolo et al. (2023). Copyright (2023) Royal Society of Chemistry. **(d)** Sargassum particle size affects HTC efficiency and relation with surface area (SA). Adapted with permission from Xu et al. (2013). Copyright (2013) American Chemical Society.

Furthermore, high temperatures lower water viscosity, facilitating deeper penetration in the biomass and enhancing decomposition. This leads to increased carbon content, C/O ratio, and C/H ratios but reduces hydrochar mass yield (Nizamuddin et al., 2017). This trend was demonstrated by Bazrafshan et al. (2024), who showed that the SA increased from 15 m^2/g to 17 m^2/g by increasing the temperature from 180 °C to 260 °C, whereas the yield decreased from 4.8 (wt.%) to 4.3 (wt.%). However, Spagnuolo et al. (2023) noted that elevated temperatures caused dehydration, increased aromaticity, and altered SA, initially increasing due to volatile release but later decreasing due to pore collapse from condensation and polymerization of small intermediates. Residence time affects reaction completion, allowing more time for hydrolysis, dehydration, condensation, and polymerization (Nizamuddin et al., 2017; Sharma et al., 2020). Shorter reaction times generally preserve porosity, while prolonged time promotes further condensation, aromatization, and carbonization, which can lead to pore collapse and reduced SA. Spagnuolo et al. (2023) found that shorter reaction times (60 minutes at 180 °C) yielded more mesoporous hydrochar synthesized from *Sargassum muticum* and with lower mass yield than longer reaction times of 180 minutes at the same temperature, where excessive condensation and carbonization led to pore collapse and reduced SA. They also found that the optimal conditions for producing high SA are 60 minutes at 240 °C, attributed to the balance between enhanced thermal decomposition and the formation of a more porous structure. This balance between reaction time and temperature is critical because prolonged reaction times at elevated temperatures can cause structural shrinkage and loss of SA due to advanced carbonization (**Fig. 4(c)**).

The initial particle size of biomass significantly influences the HTC process, affecting hydrochar yields and properties. Smaller particles improve water–biomass interaction, enhancing dehydration and decarboxylation, leading to a higher stable carbon content but a lower mass yield. Moreover, the SA decreases due to pore blockage from deposition and decomposition of secondary hydrochar on primary hydrochar (Heidari et al., 2018; Meehnian et al., 2016; Moreno-Chocontá et al., 2024). In contrast, larger particles undergo less extensive structural breakdown and tend to maintain more open pore structures, resulting in higher SA in the final product (Liu et al., 2010). In addition to the SA and yield, particle size also influences the physical morphology of hydrochar. Primary hydrochar, which forms in the initial stages of the HTC process, is typically larger and retains the shape and texture of the original biomass (Knezevic et al., 2010; Titirici and Antonietti, 2010). Xu et al. (2013) observed that smaller *Sargassum* particles (<150 μm) yielded hydrochar with lower O/C and H/C ratios, indicating better decomposition, while larger particles (>600 μm) produced hydrochar with higher SA (26.0 m^2/g vs. 12.3 m^2/g), as shown in **Fig. 4(d)**, where level-1 (<150 μm), level-2 (150–300 μm), level-3 (300–600 μm), and level-4 (>600 μm) represent different particle size ranges. The water-to-biomass ratio and pressure are important yet often underestimated parameters in the HTC of *Sargassum* (Liu et al., 2010; Xu et al., 2013). A high water-to-biomass ratio can lead to excessive hydrolysis, reducing the yield, whereas a low water-to-biomass concentration ratio promotes polymerization and solid hydrochar formation (Funke and Ziegler, 2010). Water content also affects reaction pathways (Liu et al., 2010); higher ratios slow polymerization, favoring smaller molecules, whereas lower ratios encourage larger carbonaceous structures (Favas and Jackson, 2003; Kambo and Dutta, 2015a). To the best of the authors' knowledge, studies specifically investigating the influence of process parameters on hydrochar derived from *Sargassum* remain limited, indicating a need for more focused research in this area.

2.2 Pyrolysis synthesis method

Pyrolysis is a thermochemical process that involves the thermal decomposition of organic materials at high temperatures in an oxygen-deficient environment, typically using inert gases such as nitrogen or argon (Ethaib et al., 2020; Shafizadeh, 1982). The dried and ground *Sargassum* is placed in a pyrolysis reactor, where it is subjected to high temperatures under an inert atmosphere to prevent combustion and facilitate the breakdown of complex organic molecules (Farobie et al., 2022; Tobío-Pérez et al., 2022; Tripathi et al., 2016). Pyrolysis is widely used to convert *Sargassum* biomass into valuable carbon materials, where *Sargassum* particles undergo a sequence of reactions, including decomposition, polymerization, aromatization, and carbonization (Vamvuka, 2011), which leads to the production of three main products: a black solid carbonaceous substance (biochar), a liquid product (bio-oil), and a gaseous mixture (syngas) (Qin et al., 2020; Yang et al., 2019). Based on the operating conditions, pyrolysis is classified into three categories (Ethaib et al., 2020; Foong et al., 2020; Gupta and Mondal, 2022): slow, fast, and flash pyrolysis. Slow pyrolysis operates at temperatures ranging from 400 °C to 900 °C, with a low heating rate (0.1–10 °C/s) and a long residence time (several minutes to hours) (Kebelmann et al., 2013; Yaashikaa et al., 2019). It is particularly favored for producing biochar because a longer duration provides sufficient time for polymerization and aromatization to form a stable carbonaceous structure (Demirbas and Arin, 2002; Gupta and Mondal, 2022; Milledge et al., 2015; Tripathi et al., 2016). Fast pyrolysis occurs at temperatures similar to those of slow pyrolysis but with a higher heating rate (10–200 °C/s) and shorter residence time (a few seconds), favoring the production of bio-oil (Bridgwater, 2003, 2012). Flash pyrolysis operates at even higher temperatures and ultra-fast heating (>1000 °C/s) with a very short residence time (<1 s), mainly producing syngas for energy generation or as a precursor for chemical synthesis (Demirbas and Arin, 2002; Foong et al., 2020).

Since slow pyrolysis primarily produces biochar, our discussion will focus exclusively on this method. Slow pyrolysis starts with dehydration, reducing the moisture content of *Sargassum* particles at low temperatures (<200 °C), followed by the decomposition of complex organic molecules as the temperature increases. At high temperatures, the organic matter breaks down into simpler compounds, leading to the release of gases such as CO_2, CO, CH_4, and H_2, as well as volatile organic compounds. The decomposition of *Sargassum*, driven by thermal energy, breaks down its biochemical components—lipids, proteins, and carbohydrates—into smaller molecules such as aldehydes, ketones, alcohols, fatty acids, amines, amides, furans, and pyrroles. These smaller molecules are the building blocks for the subsequent stages of pyrolysis. Polymerization follows, where some of the decomposed molecules undergo cross-linking to form more stable structures, laying the foundation of biochar. Aromatization then occurs, forming aromatic rings within the polymeric structures, followed by carbonization, which increases carbon content and enhances the stability of biochar. During carbonization, non-carbon elements such as hydrogen, oxygen, and nitrogen are lost in the form of gases, leaving behind a highly carbonaceous solid material, biochar (Li and Jiang, 2017; Wang et al., 2013; Yang et al., 2019). The biochar yield from slow pyrolysis of *Sargassum* typically ranges from 25 % to 35 %, depending on the pyrolysis temperature and residence time (Brownsort, 2009; Gupta and Mondal, 2022). A schematic of the biochar formation mechanism through the slow pyrolysis of *Sargassum* is constructed in **Fig. 5**.

Several factors, including pyrolysis temperature, residence time, initial particle size, and biomass biochemical composition, influence the outcome of slow pyrolysis. Biochar yield tends to significantly decrease with increasing pyrolysis temperature (Gupta and Mondal, 2022; Tripathi et al., 2016; Zhang et al., 2021). This trend can be attributed to the enhanced thermal decomposition and devolatilization of the biomass at higher temperatures. As the temperature increases, more volatile compounds are released, leaving behind a reduced solid residue (biochar) (Guedes et al., 2018; Gupta and Mondal, 2022). Furthermore, elevated temperatures promote secondary reactions—tar cracking and gas-phase reforming, which further contribute to biochar degradation and increase the generation of non-condensable gases like CH_4, CO, and CO_2 (Gupta and Mondal, 2022; Isahak et al., 2012; Yang et al., 2019). An increase in pyrolysis temperature has been shown to enhance biochar porosity and SA. Poo et al. (2018) conducted pyrolysis at different temperatures (up to 700 °C) on three biomasses, including *Sargassum fusiforme*, and found that the specific surface area (SSA) of the resulting biochar (*S. fusiform* derived biochar, SFB) increased with temperature (**Fig. 6(a)**) due to the enhanced decomposition and formation of micropores, which grow

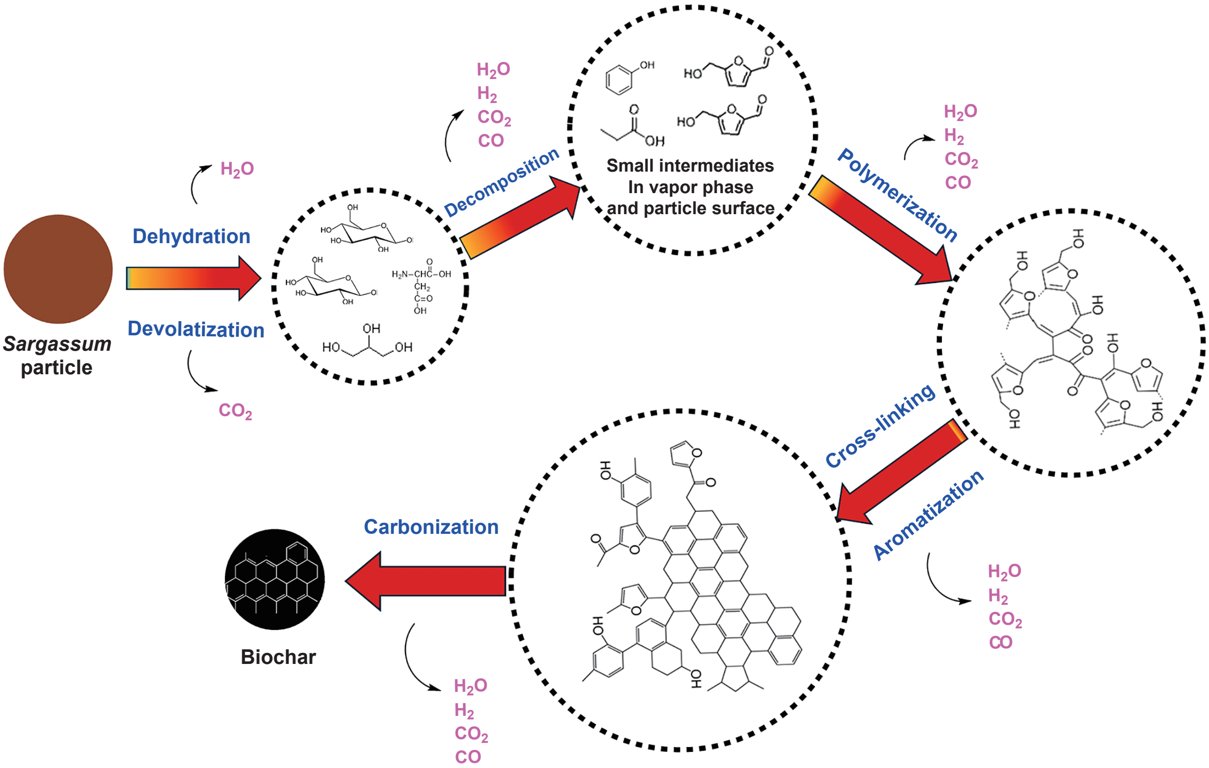

Fig. 5 Schematic of biochar formation mechanism through slow pyrolysis process.

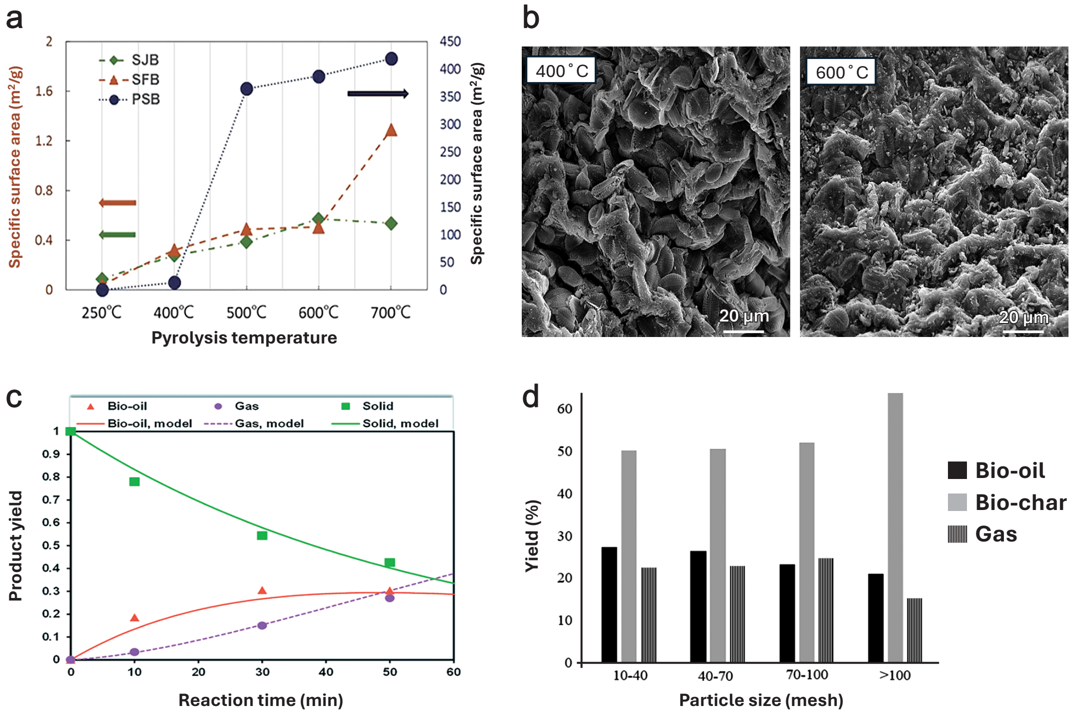

Fig. 6 **(a)** Specific surface area of the *Sargassum fusiforme*-derived biochar (SFB) as a function of pyrolysis temperature. Adapted with permission from Poo et al. (2018). Copyright (2018) Elsevier. **(b)** SEM images of biochar derived from *Sargassum* at 400 °C and 600 °C pyrolysis temperatures. Adapted with permission from Song et al. (2019). Copyright (2019) Elsevier. **(c)** Retention time effect on pyrolysis product yield. Adapted with permission from Farobie et al. (2022). Copyright (2022) Royal Society of Chemistry. **(d)** Particle size effect on pyrolysis product yield. Adapted with permission from Refki et al. (2024). Copyright (2024) IOP Publishing.

proportionally with higher pyrolysis temperatures. However, Divya et al. (2019) demonstrated the pyrolysis of *Sargassum wightii* at temperatures ranging from 700 °C to 900 °C and found that biochar produced at 700 °C showed the highest specific capacitance (354 F/g), possibly as a result of high SA, porosity, and graphitic structure, indicating significant electrochemical performance. **Fig. 6(b)** shows the SEM images of biochar derived from *Sargassum* sp. synthesized at different pyrolysis temperatures (Song et al., 2019). At 400 °C, spindle-like particles with a clearer structure and no granular material were formed, whereas at 600 °C, further carbonization led to a more compact and collapsed morphology, indicating that higher temperatures cause structural breakdown and densification of the biochar.

The residence time, the duration for which the *Sargassum* biomass remains exposed to high temperatures, also significantly affects the biochar yield and properties. Prolonged residence times allow for more thorough decomposition, enhancing the conversion of the solid biomass into bio-oil and gas at the expense of biochar yield. Longer residence times promote secondary pyrolysis reactions, including cracking and reforming, which decompose the residual char into volatiles and gases. However, extended residence time can also enhance the aromatization and carbonization process, potentially improving the biochar's carbon content and thermal stability (Choi et al., 2016; Kaewpanha et al., 2014). Farobie et al. (2022) conducted a comprehensive study on the slow pyrolysis of *Sargassum* within a residence time range of 10 to 50 minutes, observing that longer residence times resulted in lower biochar yields due to continued decomposition and the release of volatiles (**Fig. 6(c)**), and exhibited improved carbon content and lower H/C and O/C ratios, indicative of enhanced carbonization.

The particle size of *Sargassum* biomass significantly influences the efficiency and outcomes of the pyrolysis process (Jonsson, 2016; Paletta et al., 2024; White et al., 2011). Smaller particle sizes, having high SA, enhance their heating efficiency and uniformity, leading to faster breakdown of the biomass, polymerization, and aromatization (Jonsson, 2016; Paletta et al., 2024; White et al., 2011). Refki et al. (2024) have demonstrated that smaller *Sargassum* particles, particularly those in the size range of >100 mesh, exhibit a noticeable increase in biochar yield (**Fig. 6(d)**) compared to larger particles (10–40, 40–70, 70–100 mesh). The biochemical composition of *Sargassum* can also play a significant role in determining the pyrolysis products, as different components decompose at different temperatures (Raveendran et al., 1996). Lipids, proteins, and carbohydrates degrade at different temperature ranges, contributing to the overall pyrolysis mechanism. Lipids begin to degrade at around 190 °C, forming aldehydes, ketones, and long-chain fatty acids. Proteins and carbohydrates start to degrade between 200 °C and 350 °C, producing nitrogenous compounds such as amines, amides, and pyridines, as well as light organics like furans and pyrroles (Ruiz, 2021; Saber et al., 2016). At higher temperatures (350–500 °C), the remaining proteins and carbohydrates are converted into volatiles through depolymerization, decarboxylation, and cracking. Non-volatile materials degrade into CO_2 and CO at temperatures above 550 °C (Lee et al., 2020; Ranzi et al., 2008; Ruiz, 2021; Saber et al., 2016).

Numerous research efforts have utilized *Sargassum* either as a standalone biomass, in combination with other feedstocks, or by incorporating activating agents under slow pyrolysis conditions, where tube furnace reactors are used for batch pyrolysis due to their simplicity, safety, and efficient heat transfer (Chen et al., 2014). In this type of reactor, dried *Sargassum* powder is kept in a crucible inside a tube furnace, and an inert gas is continuously supplied into the furnace. Continuous nitrogen flow ensures an oxygen-free environment, a uniform temperature in the furnace, and the collection of volatile gases from the reactor. Most studies utilize activation after pyrolysis to enhance the SA of carbon materials. Wang et al. (2025) carbonized coal/*Sargassum* blends at 600 °C under nitrogen, followed by activation with KOH at 800 °C, resulting in supercapacitor electrodes with significantly enhanced electrochemical performance. Similarly, Attokkaran et al. (2024) pyrolyzed *Sargassum tenerrimum* with a manganese-based eutectic mixture (700–900 °C, N_2), identifying 800 °C as the optimal temperature for producing MnO_2-functionalized carbon for energy storage. Rosas-Medellín et al. (2025) demonstrated that pyrolysis at elevated temperatures (700–1000 °C under N_2) yielded sulfur-doped bio-carbons with outstanding oxygen reduction reaction activity, suitable for fuel cell applications. Xu et al. (2024) advanced this approach by integrating K_2FeO_4 as a chemical activator during the pyrolysis (600–800 °C, N_2) of *Sargassum thunbergii*, with 720 °C producing heteroatom-doped graphitic carbon that achieved a capacitance of 325.5 F/g.

2.3 Microwave-assisted (MW) method

The MW method of carbon materials is based on dielectric heating of the precursor. The microwave uses electromagnetic waves in the frequency range of 300 MHz to 30 GHz to heat the precursor (*Sargassum* here) to convert it into valuable carbon materials (Vanetsev and Tretyakov, 2007; Widawati et al., 2024). These electromagnetic waves interact directly with polar molecules of the solvent and precursor, and the electric dipole of the polar molecules continuously realigns with the oscillating electric field (Ethaib et al., 2020; Motasemi and Afzal, 2013; Yang et al., 2019). This rapid molecular movement generates heating through dielectric loss and friction between molecules. This molecular-level heating is efficient and uniform, enabling decomposition and carbonization of the precursor. Ionic components, such as salts, also contribute to heating through ionic conduction in response to the oscillating electric field of microwave (Ethaib et al., 2020; Yang et al., 2019). Microwave-assisted heating offers significant advantages over conventional heating methods, which rely on convection or conduction and often result in heterogeneous temperature distribution within the reaction vessel and sample. The uneven temperature, from the hotter outer vessel to the cooler center (outside-in heating), may result in incomplete reactions and non-uniform products. In contrast, microwave radiation bypasses the microwave-transparent reaction vessels and is directly absorbed by the solvents and precursors, leading to direct molecular activation or heating throughout the solution volume, thereby allowing faster reactions and uniform product properties (Díaz-Ortiz et al., 2019; Tierney and Lidström, 2005).

For MW processing, *Sargassum* is ground into smaller particles and gently dried to retain sufficient moisture, aiding microwave absorption (de Medeiros et al., 2019). The MW method encompasses several variations, with microwave-assisted hydrothermal carbonization (MAHTC) and microwave-assisted pyrolysis (MAP) being the most widely used methods for processing *Sargassum* to produce carbon materials. Both methods leverage microwave to rapidly heat the *Sargassum* under controlled conditions but differ significantly in their operational environments and end products (Ethaib et al., 2020; Hesas et al., 2013; Mohan et al., 2006). MAHTC operates at relatively low power (400–1000 watts ~<250 °C) in a high-pressure aqueous environment under subcritical conditions, similar to conventional HTC but with reaction times (5–30 minutes). During MAHTC, *Sargassum* undergoes hydrolysis, breaking down polysaccharides, proteins, and lipids into smaller organic compounds. These intermediates undergo polymerization, forming larger soluble organic molecules, which then polymerize and aromatize into carbon-rich structures. The rapid volumetric microwave heating accelerates dehydration and decarboxylation, yielding solid hydrochar and a liquid phase containing CQDs with distinct properties compared to those from conventional HTC, alongside gaseous byproducts primarily composed of CO and CO_2 (de Medeiros et al., 2019; Ke et al., 2024; Nizamuddin et al., 2018). In contrast, MAP occurs in an inert, oxygen-deficient atmosphere with higher power levels (1250–2500 watts, ~400–800 °C), following the principles of slow pyrolysis. The key differences are the significantly shorter reaction times (5–30 min) and the elimination of the need for pre-drying the *Sargassum*. During MAP, the carbonization mechanism involves the thermal decomposition of *Sargassum* into volatile compounds and reactive intermediates, which subsequently undergo polymerization into larger carbon structures, aromatization into condensed aromatic rings, and finally carbonization into stable, carbon-rich biochar. MAP yields biochar, bio-oil, and syngas with distinct physicochemical properties compared to traditional slow pyrolysis methods (Ao et al., 2018; Lozano Pérez et al., 2024; Nizamuddin et al., 2018).

Key process parameters in the MW method are microwave power and residence time, which significantly influence the product yield, composition, and properties (Soroush et al., 2023). Other parameters, such as precursor characteristics such as particle size, dielectric properties, and the use of microwave absorbers, also influence microwave absorption and heating uniformity, controlling the product properties (Ke et al., 2024; Vignesh et al., 2022). Microwave power, directly linked to the heating rate and reaction temperature, governs the efficiency of thermal decomposition and carbonization (Dong et al., 2021). Higher microwave power increases the energy input, resulting in faster heating rates and elevated reaction temperature, which accelerates thermal decomposition and promotes the formation of carbon materials with a higher degree of carbonization (Du et al., 2011). Iturbides et al. (2024) reported that increasing microwave power during the MAP of *Sargassum* mixture of fluitans and natans enhanced the energy absorption, resulting in high decomposition of the precursor, decreasing the oxygen-containing functional groups, and increasing the carbon content of the biochar. However, excessive power can cause localized overheating, resulting in non-uniform products and higher gas yields at the expense of bio-oil and biochar. Therefore, careful optimization of microwave power is crucial for achieving desired biochar properties and yield. Widawati et al. (2024) demonstrated that during the MAP process of *Sargassum*, the char yield decreased with increasing temperature, indicating enhanced thermal decomposition at higher energy inputs (**Fig. 7(a)**).

Residence time, the duration for which *Sargassum* biomass is exposed to microwave heating, significantly influences the distribution of products in microwave-assisted methods. Prolonged residence times typically enhance the degree of polymerization, aromatization, and carbonization, resulting in carbon materials with more ordered carbon structures (Gautam et al., 2019; Vignesh et al., 2022). In their study on MAP of *Sargassum* species, Gautam et al. (2019) observed that longer residence times (>200 s) led to

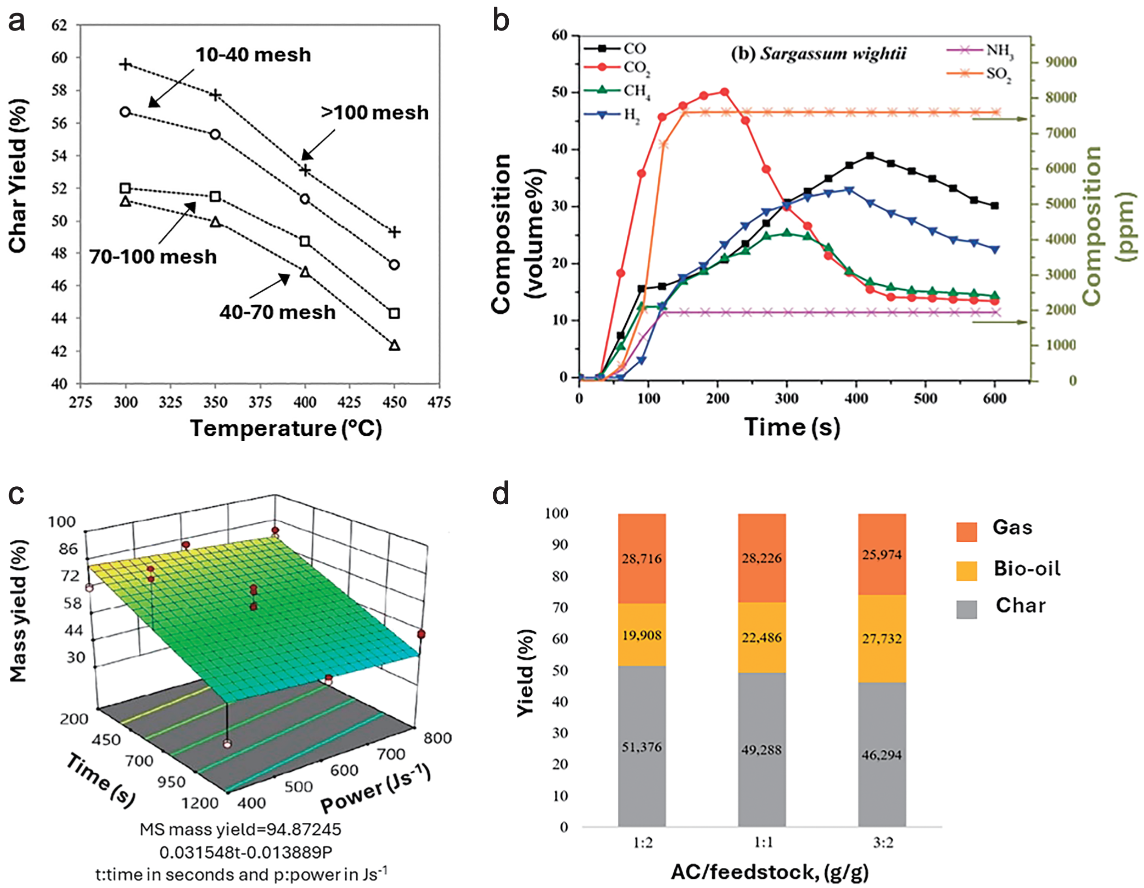

Fig. 7 **(a)** Effect of reactor temperature on char yield. Adapted with permission from Widawati et al. (2024). Copyright (2024) Gadjah Mada University. **(b)** Effect of residence time on char yield. Adapted with permission from Gautam et al. (2019). Copyright (2019) Sustainable Energy & Fuels. **(c)** Incident microwave power, and resident time on final product yield. Adapted with permission from Castañeda-Serna et al. (2022). Copyright (2022) Elsevier. **(d)** Product yield at different *Sargassum* feedstock-to-CAC (coconut activated carbon) ratios. Adapted with permission from Widawati et al. (2024). Copyright (2024) Gadjah Mada University.

increased secondary cracking reactions, further releasing volatile gases and altering the composition of biochar with increased carbon content (**Fig. 7(b)**). Castañeda-Serna et al. (2022) found that increasing microwave power and residence time significantly decreases the mass yield of CQDs from *Sargassum* (**Fig. 7(c)**), suggesting that excessive energy input or prolonged exposure time promotes biomass decomposition and volatilization, reducing solid yield. The particle size plays a crucial role in microwave absorption and heat dissipation, governed by the concept of penetration depth (δ). Penetration depth is defined as the distance from the surface of the material inward where microwave power decreases to 1/e (approximately 37 %) of its initial output power at the surface (Motasemi and Afzal, 2013; Zhang et al., 2017). Smaller particles absorb microwaves more quickly and efficiently throughout their volume, which results in rapid and uniform heating. In contrast, larger particles have higher volume for microwave absorption, resulting in higher overall temperatures. However, if the penetration depth is significantly smaller than the particle radius (r), heat transfer to the particle core is reduced, leading to uneven temperature distribution (Jie et al., 2022). Widawati et al. (2024) found an optimal particle size (40–70 mesh) effective for microwave absorption and effective heating of the particles, leading to nearly complete decomposition and low biochar yield. Conversely, smaller particle sizes (>70 mesh) resulted in lower volatile yields and higher biochar yields because of the high bulk density of *Sargassum* sp. in the reactor, which delays microwave energy from reaching the center of the reactor.

The dielectric properties of *Sargassum*, dielectric constant (ε') and dielectric loss tangent ($\tan\delta$), play a crucial role in MW methods. ε' reflects the material's capacity to store microwave energy, while $\tan\delta$ quantifies the amount of stored energy that is dissipated as heat (Fini and Breccia, 1999; Gautam et al., 2019; Ingole et al., 2016). *Sargassum*'s naturally high moisture content, along with its rich mineral composition, enhances these dielectric properties, making it highly responsive to MW heating (Nascimento and Azevedo, 2013). Ruiz et al. (2021) investigated the dielectric properties of *Sargassum natans* and found that it exhibited both a low dielectric constant and a loss factor. This indicates that dried *Sargassum* alone has a limited capacity to absorb microwave energy. Therefore, it is necessary to use *Sargassum* with adequate moisture content or incorporate additional microwave-absorbing materials to achieve efficient microwave-assisted processing. In contrast, the biochar produced through MAP of *Sargassum* showed favorable dielectric properties, including a higher dielectric constant and loss tangent. These characteristics suggest that biochar can serve as an effective microwave susceptor by efficiently absorbing and converting microwave energy into heat. These dielectric and conductive properties make *Sargassum*-derived biochar promising for applications in supercapacitors, batteries, and other energy storage devices (Ethaib et al., 2020; Wang D. et al., 2022). The addition of high-dielectric microwave absorbers has been utilized to enhance the efficiency of the microwave-assisted processing of biomass (de Medeiros et al., 2019; Ethaib et al., 2020). These materials help achieve higher maximum temperatures, which enhances bio-oil yields with elevated calorific values and reduces char yield and microwave power required, making the process more energy efficient (de Medeiros et al., 2019; Ethaib et al., 2020). Widawati et al. (2024) conducted a study using activated carbon derived from coconut (CAC), which has a high dielectric loss, as a microwave absorber at various *Sargassum* feedstock-to-CAC ratios (1:2, 1:1, and 3:2). They found that the biochar yield decreases with an increasing CAC/feedstock ratio, with maximum yield at a ratio of 1:2. The decrease in biochar yield is attributed to the enhanced microwave absorption capacity of CAC, which increases the heating rate and pyrolysis temperature. As a result, more feedstock is converted into volatiles and condensable products rather than remaining as solid char (**Fig. 7(d)**).

Despite its many advantages, MW synthesis faces challenges like limited scalability due to shallow microwave penetration and small reactor volumes, restricting large-scale applications. Sensitivity to *Sargassum*'s moisture content, composition, and particle size can cause uneven heating and affect product quality (Ingole et al., 2016; Omar et al., 2011; Widawati et al., 2024). Additionally, the system pressure limits for MAHTC and potential microwave leakage pose operational concerns. Carbon dots produced via MW often show lower quantum yield compared to conventional methods, highlighting the need for further process optimization (Ingole et al., 2016; Omar et al., 2011; Widawati et al., 2024).

3. Comparison of the synthesis methods

Each of the synthesis methods discussed above differs in terms of their working mechanism and the synthesized product, and offers distinct advantages tailored to specific applications, as summarized in **Table 2**. HTC efficiently processes wet *Sargassum* with minimal pre-drying, reducing energy use while yielding hydrochar and CQDs suitable for bio-adsorption, sensing, and soil conditioning (Bressi et al., 2023; Funke and Ziegler, 2010; Jalilian et al., 2024; Libra et al., 2011). In contrast, slow pyrolysis requires dry feedstock and more energy but produces high SA biochar ideal for soil amendment, carbon sequestration, and heavy metal adsorption (Bridgwater, 2012; de Medeiros et al., 2019; Lehmann and Joseph, 2024). The distinction between hydrochar and biochar is elaborated later in this section. MAHTC combines microwave heating with HTC conditions, enabling rapid conversion of wet/partially wet *Sargassum* into hydrochar and CQDs with tailored properties, with lower energy use and shorter reaction times

Table 2 Comparison of the synthesis methods.

Parameter	HTC	Slow Pyrolysis	MAHTC	MAP
Process Description	Subcritical water, 180–260 °C, 2–6 MPa, 5 min–several hours	Inert atmosphere, oxygen-deficient, 400–900 °C, minutes to hours	Subcritical water, 180–260 °C, 400–1000 W, 5–30 min	Inert atmosphere, oxygen-deficient, 400–900 °C, 1250–2500 W, 5–30 min
Energy transfer mechanism	Water as a heat transfer medium and a catalyst	External heating (conduction/convection)	Microwave volumetric heating	Microwave volumetric heating
Sargassum feedstock requirement	Wet or high-moisture *Sargassum*	Dry *Sargassum* (pre-drying required)	Wet or high-moisture *Sargassum*	Wet or high-moisture *Sargassum*
Process energy requirement	Moderate	High	Low	Low
Heating rate	Moderate (dependent on water heating)	Slow to moderate (dependent on external heating)	Rapid (microwave-induced heating)	Rapid (microwave-induced heating)
Product	Hydrochar (spherical, low SSA), CQDs, aqueous liquid, and gas	Biochar (graphite-like, high SSA), bio-oil, and syngas	Hydrochar, CQDs (enhanced functional groups), aqueous liquid, and gas	Biochar (high porosity), bio-oil, and syngas
Surface functionality	High (retains oxygenated functional groups)	Low (loss of functional groups at elevated temperatures)	High (retains oxygenated functional groups)	Moderate (enhanced carbonization)
SA of the char	Low SSA	High SSA (can decrease at very high temperatures)	Moderate SSA	High SSA
Product uniformity	Moderate	Moderate	High (uniform heating)	High (uniform heating)
Scalability	Limited (batch processing)	High (scalable for industrial use)	Limited (batch processing)	Limited (batch processing)

(Castañeda-Serna et al., 2022; Huang et al., 2016; Kang et al., 2019). Similarly, MAP offers fast, uniform heating of wet/partially wet *Sargassum*, yielding biochar with high carbon content, porosity, and SA (Bressi et al., 2023; Castañeda-Serna et al., 2022). While MAHTC is better suited for producing hydrochar and CQDs for environmental and sensing applications, MAP excels at generating high-quality biochar for energy storage (Kang et al., 2019; Vignesh et al., 2022; Widawati et al., 2024).

Subsequently, a comparative analysis explores the distinctions between hydrochar and biochar from hydrothermal carbonization and slow pyrolysis, the impact of HTC and MAHTC on hydrochar and CQDs, and the efficiency of slow pyrolysis versus MAP for biochar production. These comparisons provide valuable insights into the strengths, limitations, and potential applications of each method. Hydrochar and biochar, derived from HTC and slow pyrolysis of *Sargassum*, respectively, exhibit distinct physicochemical properties that significantly influence their applications. Hydrochar is produced under subcritical water conditions at lower temperatures, whereas biochar is formed in an oxygen-deficient environment through slow pyrolysis at comparatively higher temperatures. The difference in reaction environment and heating conditions results in different characteristics of the synthesized material, where hydrochar typically forms as a particulate aqueous slurry, is only partially carbonized, and retains a higher concentration of polar functional groups. As a result, it exhibits a lower surface area, moderate porosity, and relatively high O/C and H/C ratios. In contrast, biochar produced under higher pyrolysis temperatures and in an inert environment undergoes greater dehydration, carbonization, and graphitization, resulting in a material with higher surface area, increased porosity, and lower O/C and H/C ratios (Arora et al., 2024; Masoumi et al., 2021). Additionally, hydrothermal processing of hydrochar washes mineral content in aqueous medium, resulting in lower ash content of hydrochar. Whereas, biochar generally has a higher ash content due to the retention of inorganic components such as Ca, K, and Mg. This compositional variation also affects pH: biochar is alkaline due to the presence of metal oxides and minerals, while hydrochar is slightly acidic owing to the retained oxygenated functional groups. Additionally, biochar is hydrophobic due to its aromatic structure and lower oxygen content, whereas hydrochar is more hydrophilic, enhancing its interaction with water-based systems (Gray et al., 2014; Libra et al., 2011; Wang et al., 2023). Another key distinction lies in the energy potential of these materials: hydrochar has a higher heating value due to greater retention of labile carbon and lower ash content from the HTC process. In contrast, although biochar is more stable and suitable for long-term carbon storage, it has a higher ash content, which reduces its energy value (Masoumi et al., 2021; Rustamaji et al., 2022; Wang et al., 2023). Hydrochar can decompose faster than biochar, and both materials are non-toxic and environmentally safe for diverse applications (Kambo and Dutta, 2015b; Mallouhi et al., 2024; Shyam et al., 2022; Wang et al., 2023; Zhang et al., 2019). **Table 3** summarizes the key differences between hydrochar and biochar produced from *Sargassum*.

Due to differences in their heating mechanisms, hydrochar properties can also vary significantly depending on whether it is produced by HTC or MAHTC. HTC-derived hydrochar is formed under uniform conductive heating in a subcritical water environment, typically preserving the original *Sargassum* structure and enriched with oxygenated functional groups. This resulted in materials with moderate porosity, low SA, and relatively high O/C and H/C ratios, reflecting partial carbonization. In contrast, MAHTC utilizes rapid, volumetric microwave heating that accelerates hydrolysis and decomposition, producing hydrochar with more developed porosity, higher SA, and lower O/C and H/C ratios due to greater dehydration and structural rearrangement. Although MAHTC may yield less solid product, it enhances carbon content and promotes better-organized carbon structure. These variations arise due to the difference in the heating mechanism that governs hydrolysis, fragmentation, and condensation pathways during carbonization (Ojewumi and Chen, 2024; Soroush et al., 2023). Similarly, CQDs synthesized from *Sargassum* via MAHTC have higher mass yields and lower energy consumption due to shorter reaction times. However, HTC yields CQDs with superior quantum yield and more efficient core doping of nitrogen and oxygen, which can enhance their fluorescence properties. Thus, HTC-synthesized CQDs are ideal for bioimaging and sensing, while MAHTC-synthesized CQDs, with higher concentrations and favorable surface chemistry, are better suited for drug and gene delivery (Ao et al., 2018; Bressi et al., 2023; Jiang et al., 2018). Castañeda-Serna et al. (2022) synthesized CQDs from pelagic *Sargassum* using both HTC and MWHTC and evaluated their quantum yield and mass yield. Microwave synthesis produced a higher mass yield, whereas the hydrothermal approach produced CQDs with superior quantum yield. Despite similar sizes and surface functional groups, structural differences influenced their optical properties. TEM/HR-TEM analysis revealed that MW-synthesized CQDs were more amorphous due to rapid heating and shorter reaction times, which does not provide sufficient time to achieve long-range crystalline order, whereas hydrothermal CQDs exhibited higher crystallite order, which correlated with the improved quantum yield. While MAHTC offers rapid processing, its scalability is limited by small reactor volumes (typically <150 mL) and pressure constraints that restrict solvent choice. In contrast, conventional HTC supports larger batch sizes, making it more suitable for industrial-scale production despite longer processing times (Nizamuddin et al., 2018; Wang et al., 2022).

Biochar from *Sargassum* produced via slow pyrolysis and MAP exhibits distinct characteristics due to their contrasting heating mechanisms and reaction conditions. Slow pyrolysis involves gradual external heating with a long residence time in an inert atmosphere, which promotes the formation of stable, compact, and graphitic carbon structures. This method generally yields biochar with lower SA and porosity. In contrast, MAP applies rapid, volumetric

Table 3 Key differences between hydrochar and biochar produced from *Sargassum*.

Property	Hydrochar	Biochar
Product state	Slurry (solid–liquid mixture)	Black carbon solid
Structure	Spherical particles with more homogeneous particle sizes	Graphite-like layers, including particles with different size ranges
SSA and porosity	Low specific SA and porosity	High SSA and porosity
Surface wettability	Hydrophilic	Hydrophobic
pH	Slightly Acidic	Alkaline
H/C Ratio	Higher	Lower
O/C Ratio	Higher	Lower
Ash content	Lower	Higher
Decomposability	More easily decomposable (fewer aromatic structures, higher labile carbon)	More stable (aromatic structure, resistant to decomposition)

microwave heating that induces explosive devolatilization, enhancing pore formation and producing biochar with significantly higher SA, high porosity, and lower ash content. The selective and uniform nature of MAP's heating mechanism also contributes to higher fixed carbon content and improved carbonization efficiency. These heating-induced differences directly impact the carbon's structural organization, adsorption capacity, and energy value, making MAP-derived biochar more suitable for applications like energy storage and environmental remediation (Gautam et al., 2019; Leng et al., 2021; Widawati et al., 2024). Overall, MAP offers a more energy-efficient, tunable route for producing high-performance biochar, though the choice of method should align with the desired properties of the product (Ethaib et al., 2020; Omar et al., 2011).

After the synthesis of carbon materials, the resulting products may not be in the desired phase and/or retain certain impurities, such as unreacted organic matter, residual salts, and minerals. Therefore, a range of posttreatment methods are employed, including filtration, centrifugation, soaking, washing, dialysis, drying, sieving, and activation (Mallouhi et al., 2024; Marsh and Rodríguez-Reinoso, 2006). Details on *Sargassum* posttreatment procedures are provided in the J-STAGE Data (https://doi.org/10.50931/data.kona.29672663).

4. Challenges and future outlook

Sargassum is a promising feedstock for carbon material synthesis due to its abundance and composition. However, research in this area is still limited, driven mainly by the recent surge in *Sargassum* blooms. Several challenges hinder its large-scale utilization to synthesize carbon materials. First, *Sargassum* supply is seasonal and unpredictable, peaking from early spring to late summer, with minimal availability during the rest of the year. Although satellite-based forecasting exists, supply interruptions remain a barrier. Species variability, affected by geography, season, and the time of harvest after landing on the beach, also impacts the composition and carbon yield (Davis et al., 2021; Tonon et al., 2022). For instance, *Sargassum horneri* has been found to contain lignin in its cell wall composition, which may alter its thermal decomposition (Ai et al., 2022; Duran et al., 2024). The Gulf of Mexico and the Caribbean receive a mixture of *Sargassum fluitans* and *S. fluitans*, making species separation impractical for industrial applications. *Sargassum* decomposes once it lands on the beach, and its composition varies with the collection time, making it crucial to harvest fresh *Sargassum*. There are insufficient and non-standard chemical analyses of *Sargassum* across the region, which means there is still considerable uncertainty in the chemical composition of *Sargassum*, which is a crucial factor in determining the synthesis yield and the carbon material properties. Another challenge is the high content of moisture, salt, ash, and micropollutants in *Sargassum*, which further complicates processing. High moisture requires energy-intensive drying, and although some methods tolerate wet biomass, most studies rely on dry, ground *Sargassum* for consistent quality. Salt removal requires large volumes of freshwater, an unsustainable approach in water-scarce regions (Oxenford et al., 2021). Furthermore, Sargassum can accumulate micropollutants and heavy metals from the ocean, including toxicants such as arsenic, raising significant safety concerns for food and pharmaceutical applications, as these contaminants may persist in the final carbon products and pose risks to human health, thereby necessitating additional purification steps (Devault et al., 2021; Rushdi et al., 2020). Post-harvesting, *Sargassum* decomposes, releasing harmful gases, making handling, transportation, and storage a challenge (López-Contreras et al., 2021; Oxenford et al., 2021; Schuhmann et al., 2022).

Regarding synthesis methods, several challenges hinder the efficient conversion of *Sargassum* into valuable carbon materials. HTC, while effective at processing wet biomass, generates a byproduct-rich aqueous phase containing organic acids and other soluble substances that need to be properly treated, recycled, or used to prevent environmental contamination (Tekin et al., 2014; Wang et al., 2018). Slow pyrolysis demands high energy input due to high temperature during pyrolysis and the need for dried *Sargassum*, resulting in increased cost and emission of volatile organic compounds (Bridgwater, 2012; Gautam et al., 2019). Additionally, most *Sargassum* pyrolysis studies have been conducted using tube or muffle furnaces, which are not suitable for large-scale applications, limiting scalability. Although the MAHTC and MAP methods provide rapid and uniform heating, they face scalability issues, limited by small reactor volumes (Nizamuddin et al., 2018; Wang Y. et al., 2022), pressure constraints, and reduced process flexibility. Furthermore, these systems require precise control and specialized safety measures to prevent microwave leakage, adding operational complexity and costs (de Medeiros et al., 2019; Ke et al., 2024). These limitations highlight the need for improved, scalable, continuous, and environmentally sustainable synthesis methods, which may include pretreatment methods specifically designed for *Sargassum*. Moreover, a synthesis method that is independent of the composition of *Sargassum* species or can relate the properties of carbon materials to the composition of *Sargassum* would be beneficial and would help in product reproducibility.

Future research should prioritize cost analysis and life cycle assessment (LCA) of synthesizing carbon materials from *Sargassum* to evaluate the economic viability and environmental sustainability of the process. Comprehensive cost analysis should account for collection, pretreatment, synthesis, and post-processing expenses, including energy, labor, and equipment for *Sargassum* harvesting.

Simultaneously, LCA can help identify environmental impacts across each stage—from biomass harvesting to end-use applications—highlighting areas for improvement in harmful gases emission, resource consumption, and waste generation. These insights are crucial for optimizing the process, guiding policy decisions, and supporting the development of scalable, eco-friendly technologies for *Sargassum*-derived carbon materials (Almela et al., 2023; Oxenford et al., 2021). In order to find potential for minimizing energy consumption, waste reduction, and resource optimization, the research should also consider the entire processing chain, from *Sargassum* pretreatment to the synthesis and applications of carbon materials (Santos et al., 2018; Vehring et al., 2007; Verma and Singh, 2015).

5. Conclusion

Sargassum has the potential to be used as a resource for the synthesis of value-added carbon material, which has several applications, suggesting an effective solution to the problem of excessive *Sargassum* accumulation on the beaches while also developing a sustainable way to synthesize carbon materials. In this article, we review the methods (HTC, slow pyrolysis, MAHTC, and MAP) to synthesize carbon materials (hydrochar, biochar, and CQDs) from biomass and studies that have demonstrated the utilization of *Sargassum* to produce carbon materials. The choice between these methods also depends on the desired carbon material properties, energy efficiency, scalability, and final cost of the product. While there have been studies on the valorization of *Sargassum* into carbon materials using these methods, the studies are diverse and need more research on understanding the synthesis mechanism of *Sargassum*-derived carbon materials and the effect of key process parameters. Moreover, several challenges remain to be addressed, such as the continuous availability of *Sargassum*, its harvesting and decomposition, the effect of changing the composition of *Sargassum* species on carbon material properties, the scalability of the synthesis processes, and the cost-effectiveness of the synthesized product—all of which warrant further investigation. Future efforts should focus on optimizing synthesis methods and developing a way to reproduce carbon material with the changing composition of *Sargassum* species, and evaluating the environmental and economic impacts of these methods.

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Data Availability Statement

Details on *Sargassum* composition and pre- and posttreatment procedures are provided in the J-STAGE Data (https://doi.org/10.50931/data.kona.29672663).

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