

Microwave assisted carbonization and activation of biochar for energy-environment nexus: A review

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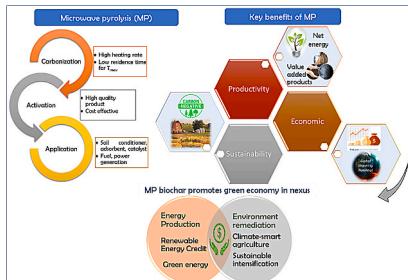
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HIGHLIGHTS

- Pyrolysis is a promising technology for biofuel production at industrial level.
- Microwave biochar production is more carbon negative than conventional heating.
- Catalytic microwave pyrolysis by biochar shows better pyrolytic volatiles reforming.
- Microwave pre-treatment reduces activation energy and increases structural stability.
- Biochar can mitigate climate change issues and achieve sustainable agroecosystem.

GRAPHICAL ABSTRACT



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ABSTRACT

Conventional thermochemical conversion techniques for biofuel production from lignocellulosic biomass is often non-selective and energy inefficient. Microwave assisted pyrolysis (MAP) is cost and energy-efficient technology aimed for value-added bioproducts recovery from biomass with less environmental impacts. The present review emphasizes the performance of MAP in terms of product yield, characteristics and energy consumption and further it compares it with conventional pyrolysis. The significant role of biochar as catalyst in microwave pyrolysis for enhancing the product selectivity and quality, and the influence of microwave activation on product composition identified through sophisticated techniques has been highlighted. Besides, the application of MAP based biochar as soil conditioner and heavy metal immobilization has been illustrated. MAP accomplished at low temperature creates uniform thermal gradient than conventional mode, thereby producing engineered char with hotspots that could be used as catalysts for gasification, energy storage, etc. The stability, nutrient content, surface properties and adsorption capacity of biochar was enhanced by microwave activation, thus facilitating its use as soil conditioner. Many reviews until now on MAP mostly dealt with operational conditions and product yield with limited focus on comparative energy consumption with conventional mode, analytical techniques for product characterization and end application especially concerning agriculture. Thus, the present review adds on to the current state of art on microwave assisted pyrolysis covering all-round aspects of production followed by characterization and applications as soil amendment for increasing crop productivity in addition to the production of value-added chemicals, thus promoting process sustainability in energy and environment nexus.

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1. Introduction

Energy security is one of the indispensable components concerned to decrease energy dependency with self-sufficient and sustainable economic growth. To ensure energy security with environmental sustainability and mitigating global climate change, the over consumption and exhaustion of finite fossil fuels deteriorating the environment has to be procrastinated. It can be enhanced by instigating the development of modern technologies to process alternate energy sources like biomass to bioproducts, biofuels, chemicals and energy (Guerriero et al., 2016; Yang et al., 2018).

Biomass, a non-exhaustive renewable energy source, serves as an alternative green fuel through various biochemical and thermochemical conversions because of its sustainability, low pollution, and its capacity to alleviate the environmental pollution caused by fossil fuel combustion. By looking into the strength, weakness, opportunities and threat (SWOT) analysis of using biomass energy at industrial level, the strength and opportunity are focusing on promoting the economy and energy supply. However, the weakness and threats indicate further development and more need for research to validate it as an efficient alternative (Nunes et al., 2019). The estimates obtained using system-generalized method of moments (a statistical method using the principles of maximum likelihood, least square estimates and instrumental variables to generate estimates with large data irrespective of its distribution pattern) for the year 1990–2017 indicated the effect of biomass consumption energy to be positive. Besides, it was statistically significant over non-renewable energy on sustainable development with the increase of level of per capita and promote the sustainable developmental goals in 2030 (Guney and Kantar, 2020). Liu et al. (2017) analysed the fates of chemical elements of biomass during the pyrolysis process and reported that studies on selecting the technologies for efficient conversion of biomass into desired products with reduced number of undesired products are limited.

Henceforth, an integrated approach of biomass waste management should be formulated and encouraged since the technical end-of-pipe solutions alone do not signify the process efficiency (Zhang et al., 2017; Morone et al., 2019). Chen et al. (2021) has recently reviewed the life cycle of torrefied biomass and suggested that the economic viability and environmental performance could be enhanced through valuation of thermochemical conversion products by means of biorefinery on circular economy. Leong et al. (2021) has presented the low carbon circular bioeconomy using algal biomass in terms of biorefinery perspective in order to produce biofuel, biochemical, heat and electricity. Integrated biorefinery has been extensively recorded as a most promising potential way for production of new bio-based fuels and chemicals by employing different combinations of feedstock and conversion technologies (Luo et al., 2017b; Ge et al., 2020). The remarkable feedstocks utilized in integrated biorefinery process includes a). Energy crops such as miscanthus, switchgrass, poplar, willow, and others (Ducey et al., 2013; Wilk and Magdziarz, 2017); b). forest, agricultural, industrial residues such as sawdust, straws, bagasse, shells, stovers and others (Wang et al., 2018a; Behera et al., 2020); c). algae and other microorganisms (Hanif et al., 2016; Lee et al., 2020a).

Pyrolysis, a technology with potential significance within a biorefinery that facilitates the generation of sustainable biofuels from different biomass feedstocks (Ahmad et al., 2017; Kostas et al., 2020). Microwave assisted pyrolysis (MAP), one of the eco-friendly means of waste disposal that is an imperative strategy of circular economy. It has been successfully utilized for biochar production and biofuels because of its volumetric and selective heating rather than direct heating associated with conventional pyrolysis process. MAP has been extensively reviewed by many authors in perspective of principle mechanisms, pyrolysis conditions and effect of different variables on product distribution, reaction kinetics, comparative analysis with traditional pyrolysis and challenges with scaling up the process and comprehend it as a cost and energy efficient process (Li et al., 2016a; Benoso et al., 2017;

Haeldermans et al., 2020). The performance of catalytic MAP has also been discussed comprehensively highlighting the significance of different catalyst to enhance energy efficiency of process and product selective distribution (Morgan et al., 2017; Liu et al., 2020b). Zhang et al. (2020) discussed about features involved in fast microwave-assisted pyrolysis used for biofuel production compared with conventional heating and concluded with yield range for biofuels, however, the comparative process energy requirements were not highlighted. Wang et al. (2020a) summarized the recent advancements in production of biochar and its modifications relating the characteristics and remediation purposes along with the feasibility of applying the novel strategies for soil remediation, whereas the detailed studies on characteristic and application of microwave assisted produced biochar for soil conditioner and plant productivity has not been elucidated. Foong et al. (2020) detailed the modes and types of pyrolysis and enlisted the aspects of microwave-assisted pyrolysis for biochar production and its utilization in environmental applications but the characteristics of microwave assisted activation and the application of biochar for soil remediation has not been included in the review. Kwon et al. (2020) reviewed the recent trends in development and application of engineered biochar from biomass, industrial wastes and its uses especially in electrochemical and biosensing applications without much description of its soil remediation and agricultural potential. Recent review by Foong et al. (2021) has elaborated the thermal conversion pathways like steam, catalytic, carbon dioxide reforming, and super-adiabatic combustion for production of hydrogen. The authors had summarized the advanced pyrolysis method including microwave pyrolysis where the effect of catalyst on the yield and properties on product, pre/post treatment mediated by microwave for product enhancement has not been discussed.

Although abundant literatures have been reviewed on MAP, sufficient information on characteristics of product composition and chemical structure influenced by microwave heating and the potential of MAP based biochar to be used as soil amendment and in contaminant immobilization for soil remediation has been scattered and discounted. Furthermore, most of the studies done so far has not accounted the energy difference between the microwave pyrolysis and the conventional pyrolysis. This review focusses on pyrolysis with its modes; reactors utilized and elucidates the advanced pyrolysis techniques with special emphasis on the features of MAP highlighting its potential advantages compared to conventional pyrolysis technologies. The effect of microwave-assisted carbonization and activation on biofuel quality has been discussed. The role of engineered biochar to act as soil conditioner in enhancing crop productivity and immobilization of soil contaminants has also been discussed to understand its importance in enhancing sustainable agriculture. Overall, this paper presents the current state of art related to production, characterization and utilization of microwave biochar to sort out the common issues concerning environment and energy nexus.

2. Pyrolysis and its economic significance

Pyrolysis, a thermolysis process wherein biomass is thermally degraded under inert atmosphere or low concentration of oxygen. It results in energy products in the form of synthesis gas (syngas), liquid bio-oil (BO), solid biochar (BC) and various value-added chemicals that could be used in environmental remediation (Chu et al., 2017; Lyu et al., 2020), catalysis, fuel source, waste management, agronomy growth, greenhouse gas (GHG) reduction and mitigate climate change by carbon sequestration (Nanda et al., 2016; Bolan et al., 2021).

Pyrolytic by-products make the process feasible in terms of co-generation of fuels for economic and environmental benefits. The study by Shabangu et al. (2014) implicates that 30 % of revenue of the plant was obtained from marketing methanol and 70 % from biochar, both being produced at 300 °C and conversely the rate when production temperature is at 450 °C. Overall energy, GHG emissions and economic

balance of pyrolysis system depend on the feedstock, production and fabrication of ancillary equipment, optimized process performance, pre and post-treatments, decommissioning of unit and the value of products. For instance, the value of biochar can be defined by Eq. (1) as given in [Roberts et al. \(2010\)](#).

$$\begin{aligned} BC = & p_p q_{C_p} + p_K q_{C_K} + \alpha \delta (p_p q_{Base_p} + p_K q_{Base_K} + p_N q_{Base_N}) \\ & + p_{GHG} q_{GHG} \end{aligned} \quad (1)$$

where the parameters describe about the price (p) and quantity of minerals like phosphorus (P), potassium (K), nitrogen (N) present in the biochar respectively; quantity of greenhouse gas (GHG) emission reductions; price and quantities of respective fertilizers applied to soil under base conditions. α is a conversion factor and δ is the fertilizer uptake efficiency difference between biochar amended and non-amended soils.

The economic value of bio-oil can be evaluated considering the least valuable crude oil (Fuel Oil No. 6) to be traded at 70 % of crude oil. Thus, the economic value of bio-oil (BO) in \$ kg⁻¹ can be represented as 70 % of the currently existing price of crude oil (\$ kg⁻¹) and density (kg barrel⁻¹ of oil) as shown in Eq. (2) as provided in the study by [Phusunti and Cheirsilp \(2020\)](#).

$$BO = ((0.7 * \text{Current market price of crude oil}) / \rho) \quad (2)$$

The economic value of syngas (SG) in \$ kg⁻¹ can be obtained using Eq. (3) ([Yao et al., 2018](#))

$$\text{Syngas} = f_g * R_g * \text{HHV}(g) \quad (3)$$

where, f_g represents the unit price (\$) of syngas per MJ, R_g represents production rate of syngas (\$ N⁻¹ m⁻³) and HHV(g) represents higher heating value of syngas (MJ N⁻¹ m⁻³).

The pyrolysis process comprehends to be a promising technology for industrial application since it utilizes the agricultural and forest residues in a potential way and reforms them into useful products.

2.1. Modes of pyrolysis

The pyrolysis of biomass is divided into several modes based on the process conditions for desired product. The main influencing parameters that vary the product yield and composition are reaction temperature, process heating rate, atmosphere, residence time, catalyst, feedstock type and its composition ([Mushtaq et al., 2015](#); [Tan et al., 2018](#)). Effective optimization of these parameters using the different optimization techniques often results in abundant product yield.

2.1.1. Slow pyrolysis

Slow pyrolysis is the common conventional thermochemical technique that has been utilized for extensive period focusing on biochar as the dominant product. It is characterized by low temperature (400 °C–600 °C) and heating rate (0.1–10 °C s⁻¹) along with higher vapour residence time (10–100 min) ([Wilk and Magdziarz, 2017](#); [Yuan et al., 2020](#)). It is often performed using fixed bed, tubular, fluidized bed, auger, ablative, microwave reactors ([Ridout et al., 2016](#); [Vieira et al., 2020](#)). The continuous slow pyrolysis will result in biochar of larger size with higher yield and mineralisation rate than other techniques under similar conditions. Slow pyrolysis can increase the combustible characteristic with significant increase of carbon content in biochar compared with hydrothermal carbonization and torrefaction ([Wilk and Magdziarz, 2017](#)).

2.1.2. Fast pyrolysis

Fast pyrolysis has been widely practised for increasing the liquid yield at moderate temperature (10–1000 °C s⁻¹) and short residence time ([Alvarez et al., 2019](#)). The temperature should be sufficient for

thermal cracking and time should be minimum in order to prevent secondary char formation. The fast pyrolysis in lab, bench-scale fixed bed reactor, bench-scale rotary kiln, was compared by [Lee et al. \(2020a\)](#) and it was found that the char yield was same in both lab and bench-scale fixed bed reactors but different from rotary kiln (RK) reactor. Char foaming and agglomeration got suppressed in RK reactor facilitating the continuous lignin pyrolysis and also increased the organic phase of liquid oil. Fast pyrolysis also aids in production of bio-oil with reduced polyaromatic hydrocarbon (PAH) in lieu of reduced emission of hazardous particulate matters ([Lu et al., 2018](#); [Lee et al., 2020a](#)). It is obvious that the biochar yield is higher at slow pyrolysis rather than fast pyrolysis since, fast pyrolysis releases more volatile that inhibits the deposition of free radical on the biochar surface and lower its concentration. Rather, in slow pyrolysis, the free radicals in volatiles make deposition on biochar surface and thereby increasing the yield of biochar ([Yuan et al., 2020](#)).

2.1.3. Flash pyrolysis

Flash pyrolysis experiments are performed at high temperature and very short residence time (5–6 s) ([Saadi et al., 2019](#)). At high temperature conditions, the char surface smoothness has been increased which indicate plastic phase formation and particle softening ([Maliutina et al., 2017](#)). The biochar produced through flash pyrolysis was shown to improve biological soil quality and the yield however, possessed short term risk to invertebrates in laboratory studies ([Gruss et al. 2019](#)) and showed negative significance of the cumulative nitrous oxide (N₂O) emissions ([Thers et al., 2020](#)). The high temperature heating involved in flash pyrolysis is often used for syngas production and this prototype can trail over the difficulties associated with commercial pyrolysis techniques ([Palumbo et al., 2019](#)).

2.2. Types of pyrolysis reactors

The production system of biochar ranges from primitive trench mounds to modern biorefinery plants. The main benefit of using batch or continuous feed pyrolyzer over traditional kilns or retorts is of increased process efficiency and product yield. Based on the motion, the reactors are classified as mechanical reactors (auger, rotary, stirrer), pneumatic reactors (fixed bed, fluidized bed), and column type reactors based on gravity motion ([Lewandowski et al., 2019](#)).

2.2.1. Fixed bed reactor

Fixed bed reactors are often employed in studying the pyrolytic yield and biomass characteristics ([Lee et al., 2019](#); [Wang et al., 2020a](#)). Pyrolytic behaviour and competing reaction in cellulose pyrolysis was studied by [Gao et al. \(2019\)](#) using fixed bed reactor and reported that biomass thickness, carrier gas flow rate influenced the distribution of product and its yield. Inefficient heating and poor gas-solid contact are a few limitations of the fixed bed reactor system ([Omoriyekomwan et al., 2016](#)). In order to resolve this issue, infrared radiation heating technique has been proposed by [Zhu et al. \(2019\)](#) for efficient and fast energy transfer (heating rate up to 30 °C s⁻¹) for cedar fast pyrolysis which enhanced the bio-oil yield at temperature lower than other conventional studies reported and it also inhibits the secondary reactions of primary volatiles.

2.2.2. Fluidized bed reactor

Fluidized bed reactor is simple to build and operate with the uniform temperature distribution owing to efficient heat transfer because of highly concentrated solid particles and the feasible process for scaling to pilot plant ([Carvalho et al., 2017](#)). The fluidized bed system is often used for high-quality bio-oil production constituted with aromatic hydrocarbons, oxygenates, phenolic compounds and water for subsequent fuel use ([Lee et al., 2020b](#)). [Wang et al. \(2013\)](#) produced 31 % biochar, 53 % bio-oil, and 10 % gaseous matter from microalgae remnants using fluidized bed reactor with silica as a fluidizing agent. The presence of high

amount of acid and water contents reduced the bio-oil quality and hence mechanically fluidized reactor (MFR) was used by Villemont et al. (2019) for bio-oil production with low content of water (8 wt%) and high energy value and it can be used for energy densification. However, some improvement techniques in sequential condensation of volatile vapours are essential for high-quality bio-oil production. Large requirement of carrier gas for processing the fluidized bed leads to thermodynamic penalties.

2.2.3. Auger reactor

Auger reactor is an intermediate pyrolysis reactor explored for the optimization and production of bio-oil and biochar extensively. Auger reactor is considered advantageous, as residence time of biomass could be easily maintained by rotating the single or twin-screw that feed the biomass to the heating zone (Brassard et al., 2017). The production of organic biochar (80 % C content) through auger pyrolysis reactor resulted in most stable biochar with less micronutrients (Pawar and Panwar, 2020). Kelkar et al. (2015) used spent coffee ground for production of bio-oil (yield of 61 % at 500 °C in 32 s) by using screw conveyor auger reactor which contains six electrical heaters in three zones maintained at different temperature range. A research gap on effect of geometry of auger flighting and mixing conditions for high yield has been acknowledged and elucidated the emphasis on studies between interaction of physical transport phenomenon and chemical interactions (Campuzano et al., 2019).

2.2.4. Ablative pyrolyzer

Ablative pyrolyzer is a new kind of fast or flash pyrolysis whereby there is direct contact between the biomass and rotating disc or cone in high temperature (Wise et al., 2019). Ablative pyrolysis facilitates the use of larger particles since the heat transfer mechanism was enhanced by surface renewal of feedstock and it controls the reaction rates (Luo et al., 2017a; Gupta et al., 2018). The particle size can be larger but the low thermal conductivity of feedstock act as a limitation for this type.

2.3. Advanced pyrolysis techniques

In addition to conventional pyrolysis, there are new advanced pyrolysis techniques enabled to explore the possibilities of enhancing the quantity and quality of pyrolytic products with less energy input and consumption. Those techniques are a). Co-pyrolysis contains feedstock of two or more materials blended and subjected to thermal degradation, which often used for production of high quality oil with high calorific value (Alvarez et al., 2019); b). Catalytic pyrolysis involves catalyst use in pyrolysis reactions to enhance the process efficiency (Chandler and Resende, 2019) c). Hydro pyrolysis refers to decomposition of biomass at higher temperature in presence of hydrogen (H_2) and resulting in production of hydrocarbon rich liquids and maximum carbon efficiency (He et al., 2020); d). Microwave associated pyrolysis involves the decomposition of biomass under the influence of microwave radiation which result in products of high quality (Afzal et al., 2018). Combination of these techniques also favours the selectivity towards pyrolytic products. Microwave based pyrolysis along with other advanced pyrolysis techniques have been compared in terms of energy efficiency and product yield as shown in Table 1. The review by Foong et al. (2021) emphasized that microwave pyrolysis and solar pyrolysis are energy efficient methods among other advanced methods wherein the effect of catalyst in microwave pyrolysis, significance of activation methods mediated by microwave irradiation was limited which has been discussed elaborately in further sections below.

3. Microwave-assisted pyrolysis

Microwave pyrolysis is relatively more efficient than other pyrolysis methods in terms of product quality and energy efficiency. It has been well comprehended that microwave source of electromagnetic waves

enhances proficient thermal degradation of biomass by the means of selective, volumetric, rapid and uniform heating compared to conventional pyrolysis (Nair and Vinu, 2016; Dai et al., 2020). It has the potential to produce unique grade of products through selective thermal gradient throughout the process. Microwave pyrolysis are employed largely for production of high-quality products which can be used directly in biorefineries, and for catalysis, hydrogen production, metal reductions, soil conditioners for increasing process sustainability as described in Fig. 1 (Lestinsky et al., 2017; Shukla et al., 2019). The significant change in morphological structure, evolution of surface functional groups, crystal lattice has been reported in microwave heating over conventional pyrolysis (Huang et al., 2016a,b). The modified texture properties of the biochar obtained possess distinct potential bio-refinery application and thereby establishment of sustainable bioeconomy.

3.1. Mechanism of microwave heating

The commercialization of microwave technology for heating has been emerged during 1950 by an American Engineer Spencer, who confirmed the temperature rise of food under the influence of microwave radiation which later being adopted by companies for residential purposes (Spencer, 1950; Menendez et al., 2010). Microwaves are non-ionizing electromagnetic radiation in the mid of radio and infrared frequencies with the range of wavelength, 1 mm to 1 m with frequency from 300 GHz to 300 MHz respectively (Zhang et al., 2017; Kostas et al., 2020). Small microwave ovens are operated with wavelength of 12.2 cm and a frequency of 2.45 GHz because of precise penetration depth while industrial-scale reactors use 915 MHz frequency with a wavelength of 32.8 cm (Menendez et al., 2010; Undri et al., 2014). The lab-scale microwave reactor composed of microwave oven and transparent reactor made up of borosilicate (15.7 m), or quartz (75.73 m) contained with an absorbent bed having superior penetration capacity (Zhang et al., 2017).

The microwave heating termed as dielectric heating involves dissipation of energy from the dielectric material when exposed to alternating electromagnetic field. When microwave radiation penetrates the material of a certain volume, the ionic or molecular dipoles were subjected to translational motion by internal generated electric field. In such manner, the polar molecules that contain dipole moment will tend to rotate and align themselves with oscillating electromagnetic field. These molecules will reverse the direction when the field alternates; vibrates and collides with the adjacent molecules and dissipates heat energy to others. The heat distribution is non-homogeneous and it is greater than macroscopic temperature in certain zones. The non-homogeneous temperature and rapid oscillation cause disruption to the material structure (Ganesapillai et al., 2016).

In microwave, dipole molecules will be irradiated with microwave and thermal energy is radiated from inside to outside which facilitates efficient heating mechanism based on the dielectric property of the material (Binner et al., 2014; Dai et al., 2020). Since certain biomass is less efficient in absorbing microwave owing to low dielectric, microwave absorbers such as carbonaceous material are being used for efficient microwave heating. Carbonaceous material acquires the selective and targeted heating and converts it into heat energy within itself based on the microwave absorption capability, and intensifies with irradiation time. The rapid rate of oscillation (million times per second) between the molecules produces the heat energy and results in volatilization and carbonization (Lam et al., 2018; Ge et al., 2020). In a simple way to signify the mechanism in two steps, the energy is absorbed and conducted to biomass and the ensuing phase is that more carbon is produced from the raw material whilst the continuation of pyrolysis process where microwave instigates the reactions inside the material contributing to volumetric heating, unlike the conventional pyrolysis.

Table 1

Summary of biochar characteristics and energy efficiency of different pyrolysis process.

Pyrolysis types	Feedstock	Pyrolysis condition		Biomass characteristics		Maximum product yield (%)	Biochar characteristics		Energy recovery efficiency or EROI	Reference
		Reactor	Parameters	Elemental composition (%)	Heating value (MJ kg ⁻¹)		Elemental composition (%)	Heating value (MJ kg ⁻¹)		
Microwave-assisted pyrolysis	Rice straw	Single mode microwave oven	Load: 200 g Power: 0.5 kW Duration: 30 min <i>N₂</i> flow: 50 ml min ⁻¹	C-45.76; H-6.22; O-47.50; N-0.52	HHV-16.16	Solid – 20 Liquid– 44 Gas – 36	Carbon content – 62		EROI: 3.56	Lo et al. (2017)
	Woody biomass- <i>Leucaena leucocephala</i>	Single mode microwave oven	Load: 8g Power: 0.1-0.25 kW Duration: 15–30 min <i>N₂</i> flow: 35 ml min ⁻¹	C-39.36; H-5.4 O-31.43; N-5.92	HHV-18.34	Solid – 21.45–56.50	C-76.29; H-2.64; O-15.07; N-1.02;	HHV-29.65	EROI: 19-34	Huang et al. (2017b)
	Sludge	1.4 kW microwave oven	Load: 15g Power: 0.8 - 1.4 kW	C-33.14; H-5.58 O-55.75; N-4.85	LHV-11.54	Solid- 48.14 Liquid-14.59 Gas - 52.76	C-25.98; -2.93; N-0.03; S-0.83	LHV - 4.59	Energy recovery efficiency: 6.23%–18.79 %	Luo et al. (2020)
Wet pyrolysis	Rice straw	1 L Parr stirred pressure batch reactor	Load: 15g Biomass/ Water: 1:20 Temp.: 250°C Duration:	G-38.4; H-6.6; O-40.3; N-2.5	HHV-15.3	Solid – 27.22–28.10	C-58.4; H-5.75; O-26.5; N-3.0	HHV - 24.46	Energy recovery efficiency: 36.67%–45.03 %	Xu et al. (2019)
	Sewage sludge	Electrically heated 4 L Zipper clave pressure vessel	Load: 2 kg Temp.: 180 °C-250 °C Heating rate: 3 °C min ⁻¹ Duration: 60 min	G-32.7; H-4.9; O-15.4; N-5.1	HHV-14.9	Solid – 51.8–74.2	C: 32.6; H: 4.1; O: 10.0; N: 4.1	HHV:15.1	Energy recovery efficiency: 52.3 %	Marin-Batista et al. (2020)
	Cattle manure	0.5 L stainless steel stirred & pressurized batch reactor	Load: 20 g Biomass/ Water: 1:10 Temp.: 200 °C-300 °C Duration: 45 min	G-33.9; H-4.9; O-23.8; N-3.6	HHV-14.3	Solid – 24.4–45.4	C: 43–47; H: 4.9–5.1; O-14.7–26; N:3–3.3	HHV: 17–20.5	EROI: 1.4–2.8	Posmanik et al. (2020)
Co-pyrolysis	Cotton gin trash (CGT) + cow manure (CM) + microalgae (M)	Batch pressurized reactor	Load: 300g Temp.: 400 °C-600 °C Heating rate: 5 °C min ⁻¹ Duration: 30 min	HHV: CGT- 17.9 CM - 19.8 M – 23.3	Solid – 91 (400 °C) Liquid – 48 (551 °C) Syngas – 22 (600 °C)		HHV for char: 7.18 HHV for bio-oil: 7.3	Energy recovery efficiency: 75.6 %		Hanif et al. (2016)
	Waste tire + Pine bark	Fixed bed reactor	Load: 35 g Temp.: 900 °C	C: 58.24–73.98; H: 04.82–06.05; N: 01.63–01.68; O: 18.30–35.31;	LHV: 22.6–30.1	Solid: 29–36 %			Energy recovery efficiency: 42–47.3%	Wang et al. (2020c)
	Microalgae (M)+ Sewage sludge (SS)	Fixed bed reactor	Load: 2.5 g Temp.: 425 °C -500 °C <i>N₂</i> flow: 400 ml min ⁻¹	HHV: 16.5–20.0	Solid:57.55 %		HHV: 6.49–11.67	Energy recovery efficiency: 56.77–63.43 %		Wang et al. (2016)
Vacuum pyrolysis	Waste tires	Vacuum pyrolysis and upgradation					LHV: 39.76	Energy recovery efficiency: 13.15%–28.15 %		Karagoz et al. (2020)
	Paper waste sludge	Fixed bed reactor	Load: 20 g Temp.: 250 °C	Lower Ash PWS C: 49.2; H:	HHV: Lower Ash PWS:	Solid: ~16–20 %		Energy recovery		Ridout et al. (2016)

(continued on next page)

Table 1 (continued)

Pyrolysis types	Feedstock	Pyrolysis condition		Biomass characteristics		Maximum product yield (%)	Biochar characteristics		Energy recovery efficiency or EROI	Reference
		Reactor	Parameters	Elemental composition (%)	Heating value (MJ kg ⁻¹)		Elemental composition (%)	Heating value (MJ kg ⁻¹)		
			-600 °C Heating rate: 30 °C min ⁻¹ Duration: 30 min	5.9; O: 44.8; N: 0.08; Higher Ash PWS	17.8 Higher Ash PWS: C: 47.2; H: 6.7; O: 45.7; N: 0.41					efficiency: 58.7 %
Tires	Vacuum pyrolysis reactor		Load mass flow: 25 kg h ⁻¹ Temp. 600 °C Pressure: 20 kPa Time: 86 min			Solid: 38.3 % Liquid: 40.7 % Gas: 12.1 %	C-86.49 H-1.30; N-0.51; S-1.96		Energy recovery efficiency: 70 %	(Oliveira Neto et al., 2019)
Steam pyrolysis	Sewage sludge	Vertical pyrolysis reactor	Load: 50g Temp.: 600 °C N ₂ flow: 100 ml min ⁻¹ Time: 30 min	C: 40.93; H: 5.01; O: 49.33; N: 3.85	Liquid: 14.16%–24.03 %		HHV: 13.74		Energy recovery efficiency: 61.92%–88.85 %	Mei et al. (2020)
	<i>Eucalyptus occidentalis</i>	Test reactor (prismatic jacketed chamber)	Load: 6g Temp.: 414 °C – 694 °C Pressure: 500 kPa	C-46.8; H: 5.4; O-45.0; N: 0.0	HHV: 18.3	Solid: 18.3%–37.1 %	HHV of char: 22.9–28.0 HHV of gas: 1.8–8.8		Energy recovery efficiency of gas: 1.2%–28.9 % Char: 19.2%–42.6 %	(Giudicianni et al., 2017)
Hydro pyrolysis	Algae	Stainless steel stirred reactor	Water/ Biomass ratio: 6 Temperature: 150 °C–300 °C Pressure: 25 bar Time: 20 min		Liquid: 42 %				EROI: 1.3	(Choudhary et al., 2020)
	Woody biomass	Fluidized bed reactor	Pressure: 20–35 bar						EROI: Diesel-4.19; Gasoline-4.31	Zupko (2019)

*C-Carbon; H-Hydrogen; O-Oxygen; N-Nitrogen content.

*HHV- Higher heating value; LHV: Lower heating value.

EROI represents energy return on investment.

3.2. Products of microwave assisted pyrolysis

The modern analytical technology intervention enables the researchers to target the desired compound by stabilising the parameters to control the products formation among the hundreds of chemical reaction in the pyrolysis process. Biochar, bio-oil, and syngas are the three main pyrolytic product obtained from microwave pyrolysis using biomass of different origin. Feedstocks like rice husk (Shukla et al., 2019), corn cob (Lawas et al., 2019), bamboo sawdust (Dai et al., 2020), seaweeds (Kostas et al., 2019), food waste (Lin et al., 2020) and from various other source has been subjected to MAP to obtain pyrolytic product of better quality with less energy input. Significant number of research has focussed on high-quality bio-oil production which acts as a viable source platform for value-added chemicals (Beneroso et al., 2017; Kostas et al., 2020) and production of H₂ rich syngas that could be used as biofuel (Shi et al., 2020). Biochar obtained from MAP has been used in vast applications such as soil conditioner, catalyst, adsorption, energy storage device, etc.

3.2.1. Bio-oil

Bio-oil produced through MAP is potential source for generation of

pharmaceutical and fine chemicals. The high value of nitrogen-containing bio-oil can be prepared from microalgae which contain higher nitrogen content than other biomass through MAP with less time and cost (Chen et al., 2016; Yu et al., 2017). The microwave oven of 2 kW was used by Huang et al. (2017a) for the pyrolysis of *Chlorella* sp. and *Spirulina* sp. with activated carbon and ferric oxide (Fe₂O₃) as microwave receptors and enhanced the nitrogen-containing aromatic and aliphatic groups in bio-oil respectively. *Chlorella* sp. containing more amino acids with nitrogen-containing side chains results in higher nitrogen retention in biochar and activated carbon promoting higher nitrogen conversions during pyrolysis (Ren and Zhao, 2013). Effect of temperature and feed rate on product yield on continuous microwave fast pyrolysis was predicted using rice straw and *C. oleifera* shells. The highest bio-oil yield with increased phenols, alcohol, aldehydes is of 27.45 % at 200 g min⁻¹ feed rate at 400 °C and 31.86 % at 24 g min⁻¹ feed rate at 500 °C obtained from *C. oleifera* shells and rice straw respectively. The optimum temperature difference is attributed to thermochemical behaviour of biomass composition (Wang et al., 2018b). Compounds like methyl ester, 1-methyl-5-oxo-, L-Proline, were found to be in bio-oil obtained from pelletized *Laminaria digitate* through MAP where the infrequent composition leads to a unique product

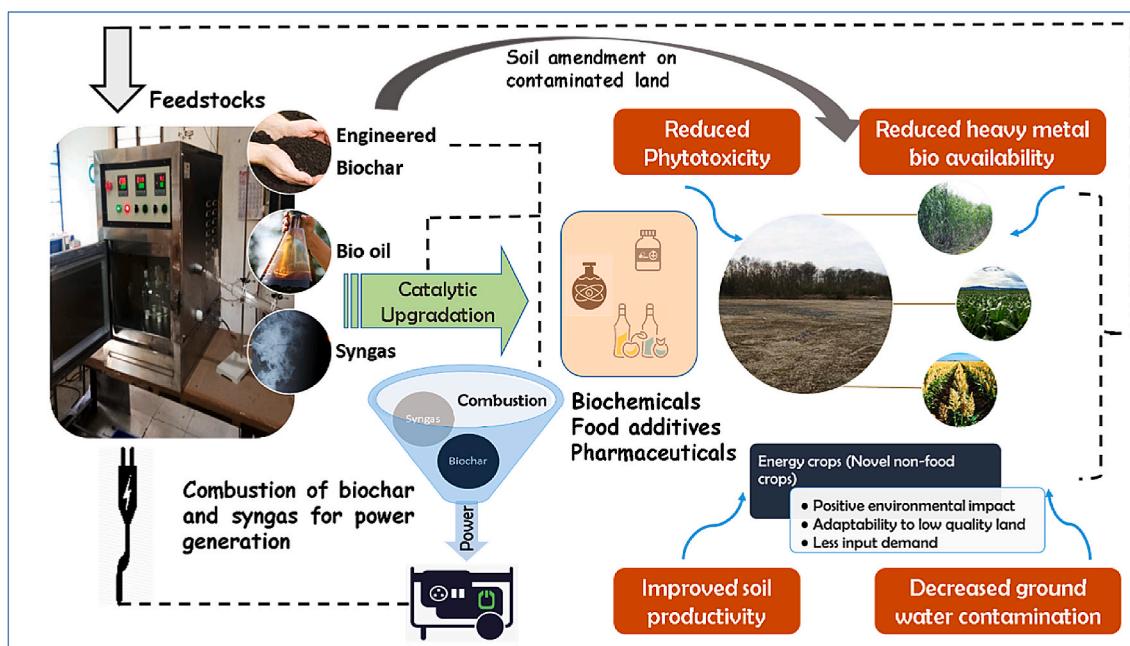


Fig. 1. The role of microwave pyrolysis as a sustainable approach for biofuel production.

(Kostas et al., 2019a). The palm empty fruit bunch (EFB) treated with microwave resulted in high yield with respect to power level. The EFB irradiated with 600 W of microwave resulted in higher light bio-oil which is favourable for performance of internal combustion engines (Sangjan et al., 2020).

3.2.2. Syngas

Hydrogen (H_2) and carbon dioxide (CO_2) are main compounds in gas products of microwave pyrolysis, which acts as a basis for chemical product synthesis like ammonia, alcohol and acetic acid which are potential source of energy (Valera-Medina et al., 2021). The syngas as a target product was accomplished by many microwave studies (Shi et al., 2017; Parvez et al., 2019a,b; Lin et al., 2020). Xin et al. (2019) attained goal of higher syngas yield of 55.70 % at 1 kW under CO_2 atmosphere using soybean straw in a microwave pyrolysis furnace. The endothermic processes like Boudouard reaction ($C + CO_2 \rightarrow CO$) and pyrolysis reaction was promoted greatly at higher power, heating rate and temperature and resulted in high syngas production. Thereby microwave technology is proving to be a very suitable alternative for syngas production under CO_2 atmosphere. The microwave-induced cracking removes the barrier of downstream processing of tar removal and catalyst deactivation associated with syngas production from gasification (Beneroso et al., 2015). Beneroso et al. (2016) obtained char from microwave pyrolysis of municipal solid waste organic fraction and used subsequently as absorbing bed material for cracking the tar at 800 °C and resulted in 0.49 m³ of syngas per kg of biowaste. The net energy consumption was 79.8 (60 + 19.8) Wh g⁻¹ of biowaste including MAP and tar cracking at 800 °C respectively. Luo et al. (2020) accomplished MAP of sludge by two modes, temperature and power control which paved the way to explore the heating and energy conversion mechanism and characteristic of biofuel from low power and high-energy utilization efficiency. It is noteworthy to mention that apart from H_2 and CO_2 , based on the feedstock composition, organically bound nitrogen during thermochemical conversion is converted into ammonia, which is an important component during syngas production. Chai et al. (2021a,b) identified syngas production technology from microalgae generating ammonia for achieving significant carbon reduction and further discussed the energy demands and government policies to drive its large-scale utilization. Another study by Chai et al. (2021a,b) reported

the utilization of H_2 and methane (CH_4) as a secondary fuel, to aid ammonia conversion, increase its burning velocity and reduce nitrous oxide (NO_x) emissions, promoting its sustainable real-time application as an energy vector. The role of key reaction under varying conditions with the scope of future perspectives and challenges on the usage and development of ammonia-based fuels were also emphasized. The ammonia production through microwave catalytic assisted process is gaining more attention nowadays with the aim to reduce the carbon footprint and the technical requirements like high temperature and pressure related to conventional Haber-Bosch (HB) process. Hu et al. (2020) reported that synthesis of ammonia in ambient pressure and low temperature under the influence of microwave irradiation leads to generation of carbon-free fuel with higher energy density and viable for energy storage and transportation. The utilization of microwave catalytic reaction for ammonia production is more effective and restrains the limitation related to economy of process scale of HB process.

3.2.3. Biochar

MAP relatively produces biochar in higher yield utilizing lower power even without the requirement of catalyst (Hossain et al., 2017) and found efficient utilization in recent applications as listed in Table 2. The significant influence of microwave parameters in biochar production was analysed through half-resolution factorial design. Effect of power and exposure time mostly influences product yield of cormcob under MAP. Low power enhances carbonization process in low time whereas high power facilitates syngas production but also enhances the quality of biochar, almost twice the percentage in terms of elemental carbon (Lawas et al., 2019). The MAP can be accomplished with low energy requirement of 3.6 kJ g⁻¹ and also enable the textural characteristics of biochar to act as an adsorbent for dyes or chemicals and the bio-oil composed of acetic acid, phenol and levoglucosan derivatives making it suitable for biorefinery applications (Kostas et al., 2020).

Self-purging pyrolysis is the cost-effective process in which the gaseous product within the reactor is trapped and being re-used as purging gas (Noor and Abdullah, 2018). Self-purging mode of pyrolysis has been employed for a range of biomass which offers biochar with good pore characteristics leading to reduced cost process than conventional pyrolysis, where external nitrogen purging is mandatory (Intani et al., 2016, 2018). The biochar production from waste palm shell in a

Table 2

Studies on microwave produced biochar utilization in recent applications.

Feedstock	Microwave parameters	Biochar characteristics	End application	Reference
Sugarcane bagasse	<i>Microwave-assisted KOH activation of biochar in a Teflon sealed reactor</i> Power: 700 W; CO_2 flow: $300 \text{ cm}^3 \text{ min}^{-1}$; KOH/biochar: 3	Surface area: $1019 \text{ m}^2 \text{ g}^{-1}$; $V_{\text{meso}}/V_{\text{total}}$: 56.7 %; Specific Capacitance –208 F g^{-1} ; Electrosorption capacity- 28.9 mg g^{-1}	Carbon electrodes for electrochemical desalination	Tang et al. (2020)
Electric arc furnace (EAF) dust	<i>Self-reduction of EAF with biochar in multimode microwave tube furnace</i> Biochar/EAF dust- 0.25 under N_2 atmosphere	Fixed carbon-79.87 % Volatile matter-6.02 %	Reduction efficiency for Zn-99.6 %; Pb-92.9 % Iron recovery of 87.36 % for magnetic concentrate Powered direct reduced iron product for steel making	Ye et al. (2020)
Wheat straw	<i>Microwave-assisted synthesis in microwave reactor</i> Precursor: Biochar, Ferrocene Power: 200 W Duration: 5 min	Surface area: $57.38 \text{ m}^2 \text{ g}^{-1}$; Pore volume: $0.001 \text{ cm}^3 \text{ g}^{-1}$ Hydrodynamic diameter: 400 nm Bandgap: 4.4 eV Specific surface area: $5.43 \text{ m}^2 \text{ g}^{-1}$	Synthesis of carbon nanotubes	Hildago-Oporto et al. (2019)
Oat hull	<i>Microwave-assisted sulfuric acid activation in microwave reactor</i> Temperature: 140°C ; Time: 30 min Sulfuric acid/biochar: 10:1	Pore size: 1.03 nm Total acidity: 7 meq g^{-1} Specific surface area: $1755 \text{ m}^2 \text{ g}^{-1}$	Production of biodiesel with yield of 89.97 % with 90 % FAME content	Gonzalez et al., 2017
Waste palm	<i>Microwave assisted activation KOH activation</i> Power: 700 W Time: 5 min	Pore volume: $0.942 \text{ cm}^3 \text{ g}^{-1}$ Specific capacitance: 164.8 F g^{-1}	Efficient supercapacitor with capacitance retention of 92.06 %	Liu et al. (2019)

modified microwave oven at 750°C through the self-purging mode was studied by Yek et al. (2017). It was used for activation using self-generated CO_2 produced during the pyrolysis and employed as an activating agent to generate waste palm shell activated carbon having surface area higher than $1200 \text{ m}^2 \text{ g}^{-1}$ and having adsorption efficiency of 440 mg g^{-1} for methylene blue dye. Self-purging microwave pyrolysis using oil palm shell was adopted for carbon-rich biochar production facilitating adsorption of methylene blue. Biochar yield of 40 % with 78.5 % of carbon has been obtained by Kong et al. (2019) at 700°C in 20 min with good pore characteristics and adsorption efficiency of 20 mg g^{-1} for methylene blue dye.

4. Catalytic microwave pyrolysis

As discussed earlier, the liquid product of microwave pyrolysis is a

combined organic compound that acts as reservoir for deriving value-added chemicals like phenols, aldehydes, levoglucosan, aromatic hydrocarbons. Nevertheless, the separation of these valuable products through current refinery is skeptical because of complexity of bio-oil composition and the adoption of biochar has been scarce in applications because of production cost. Hence, researchers have adopted efficient methods to control the pyrolysis reaction concerning more product selectivity without compromising the economic viability. Microwave catalytic pyrolysis is an emerging technique of using catalyst in microwave pyrolysis for increasing desired product yield and selectivity (Wang et al., 2018a; Mohamed et al., 2021). Utilization of catalyst or additive mixed either with biomass or as fixed bed promotes product selectivity and quality in microwave catalytic process with enriched valuable compounds. Catalytic microwave pyrolysis produced biochar assists the improvement of soil hydrological properties and it is more

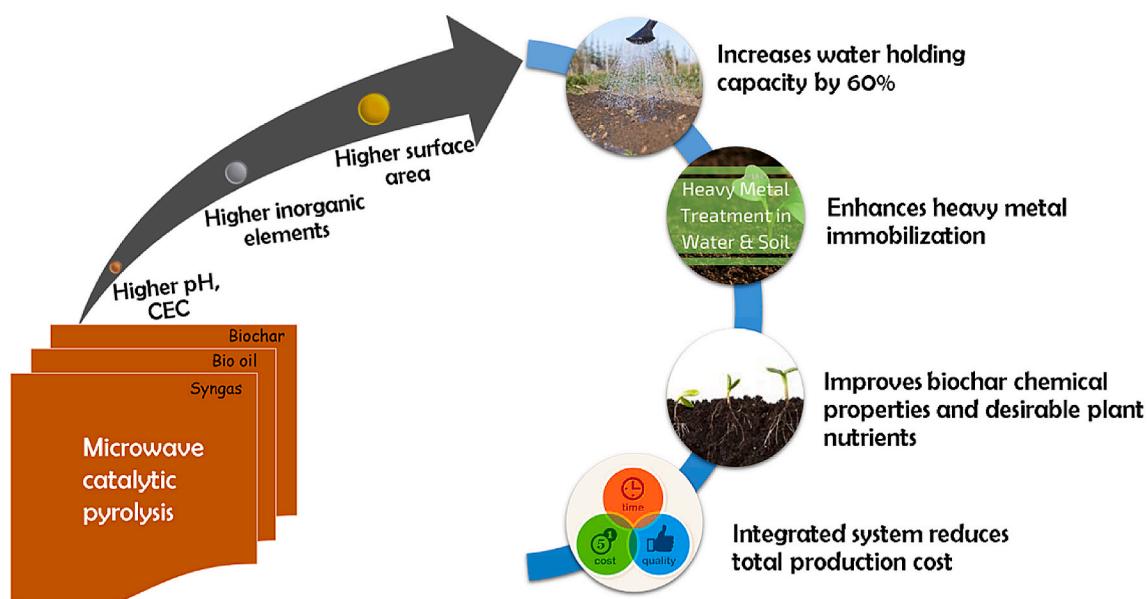


Fig. 2. Synergistic effect of biochar characteristics produced by microwave catalytic pyrolysis for soil remediation and crop productivity.

effective in sequestration of carbon as described in Fig. 2 (Mohamed et al., 2016b).

4.1. Ex-situ and in-situ catalytic microwave pyrolysis

The extensive literature survey implicates two types of catalytic microwave pyrolysis via *in-situ* and *ex-situ* method. In *in-situ* process, biomass and catalyst are mixed in certain ratios and fed into reaction chamber, or in other way, the microwave fixed bed with catalytic mixture is pre-heated and then biomass is fed into reactor (Yang et al., 2018). In *ex-situ* catalytic microwave pyrolysis, the biomass-derived products are carried by carrier gas to a catalytic bed which is separated externally (Wise et al., 2019). *Ex-situ* catalytic microwave pyrolysis is meant for processing of reaction through packed bed catalyst reactor and is often employed for production of bio-oil (Wang et al., 2014; Mamaeva et al., 2016). *Ex-situ* catalytic pyrolysis generates more aromatic hydrocarbon, and pyrolytic lignin than *in-situ* catalytic process owing to better reaction environment. The catalytic reactivity is compensated in *ex-situ* catalytic microwave pyrolysis since the pyrolytic vapours are expected to condense before coming in contact with catalyst (Li et al., 2016b). *In-situ* process facilitates the provision of microwave plasma and hotspots through the catalytic bed promoting the heterogeneous catalytic activity. *In-situ* catalytic process resulted in higher bio-oil yield than *ex-situ* catalysis at higher catalyst to biomass ratio. *Ex-situ* process has higher gaseous residence time than *in-situ* process and when large amount of catalyst is added, the time of contact between catalyst and the primary volatiles have been extended and increased the secondary cracking resulting in high yield of syngas (Wang et al., 2014; Luo and Resende, 2016; Fan et al., 2018).

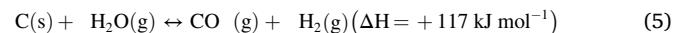
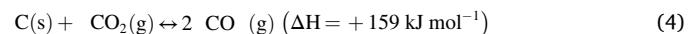
Metal oxides, zeolites, activated carbon are mostly used for the upgradation of products in microwave pyrolysis (Luo and Resende, 2016; Chang et al., 2018). Wang et al. (2018a) utilized catalysts like Cu/C, Pd/C, Pd-Ag, and HZSM-5 in pyrolysis of larch sawdust and found enhancement of phenol formation by these catalysts. The 36.94 % bio-oil yield with 75.88 % hydrocarbon content has been obtained by Jiang et al. (2020b) using ZSM-5 composite and the regenerated catalyst at 550 °C still produced 73.01 % of hydrocarbon products. The pores of catalyst ZSM-5 are diffused with primary vapours, while the aromatic hydrocarbons and small molecules are produced on active sites by catalytic cleavage of C-C and C-O bonds and conversion of intermediates (Fan et al., 2018; Liu et al., 2020b). The apparent activation energy is decreased by catalyst addition for decomposition of biochemical composition during pyrolysis or co-pyrolysis. The *ex-situ* catalysis using ZSM-5 or activated ZSM-5 promotes the deoxygenation reactions than *in-situ* mode while the lower molecular weight compounds were converted from oxygen-containing intermediates. Deoxygenation and excessive volatile cracking with catalyst increase the gas yield significantly with slight decrease in char yield and subsequent decrease in pyrolysis liquid yield (Wang et al., 2014; Shafaghat et al., 2019). Microwave pyrolysis combined with *ex-situ* catalytic reforming emphasized for production of bio-oil from soybean soapstock in tandem microwave pyrolysis system with feed-to-catalyst ratio of 2:1 at 550 °C (pyrolytic temperature) and 350 °C (catalytic temperature). The bio-oil yield decreased with increased ZSM-5 proportion in catalyst under microwave heating since it enhances the deoxygenation and aromatization reactions than conventional heating and resulted in generation of low oxygen-containing compounds (23.78 %) and higher aromatic hydrocarbons (75.88 %) (Jiang et al., 2020b). The zeolite-based catalyst having micropores hinders the diffusion of pyrolyzates derived from biomass having large kinetic diameter. Hence, a mesoporous based catalyst is a better solution for improving the mass transfer and increase the catalytic sites for the reactions (Kelkar et al., 2015b; Shafaghat et al., 2019). HZSM-5 catalyst treated with an organic base has developed mesoporous structure and exhibited higher selectivity towards monocyclic aromatic hydrocarbons, reduced the strong acidic sites on outer surface inhibiting formation of coke compared to parent catalyst by 4.6

% (Li et al., 2020b).

Huang et al. (2018) investigated the catalysis effect along with particle size and pre-treatment on the gaseous products from corn stover pyrolyzed under microwave. The substantial catalytic activity of aluminium oxide (Al_2O_3) was found to be significant after the pre-treatment using acid/steam. The size of catalyst also influences the gaseous yield where the relatively small size of catalyst hindered the secondary degradation of organic vapours released during pyrolysis, thereby reducing the catalytic activity. The adoption of *in-situ* catalytic pyrolysis is of low capital and operating costs because of single unit reactor configuration in comparison to *ex-situ* pyrolysis where the catalytic upgrading facilitated by separated zones are more beneficial for independent process optimization (Iisa et al., 2016; Shafaghat et al., 2019).

4.2. Feasibility of biochar as catalyst in microwave pyrolysis

The activated char or char obtained from pyrolysis process are being used as microwave absorber in heterogeneous reactions of microwave pyrolysis (Mushtaq et al., 2015). The dielectric loss tangent (ability of substance to be heated under microwave radiation) for the char is of 0.57–0.8 which is almost higher than biomass (0.1) and silicon carbide (0.25). When the pyrolytic vapours encounter the activated carbon, it enhances the reduction of oxygenated compounds in the volatiles indicated by lack or increase of certain functional groups. During the reforming process, the oxygen molecule was migrated to incondensable gas fraction from condensable bio-oil. Further, contribute to higher gaseous yield with subsequent lower bio-oil yield. The activation process for carbon catalyst's surface by microwave radiation catalyses and enhance the syngas yield is given by the following equations (Eq. (4) and Eq. (5)) (Hunt et al., 2013; Yek et al., 2020).



where, ΔH refers to the change in enthalpy. Char enhances these reactions through heterogeneous decomposition of organic gas molecules under relatively low temperature and ancillary cracking under the influence of microwave radiation (Lam et al., 2017a,b). This helps the activated carbon to serve as microwave absorber or catalyst for reforming of pyrolytic volatiles that is promoted under microwave heating than electric heating (Ferrera-Lorenzo et al., 2014).

4.2.1. Significance of char as catalyst for microwave pyrolysis

The catalyst, activated char functioning as microwave absorber developed hot spots under the influence of microwave heating. The gasification reactions are favoured by these hot spots, releases H_2 that promotes lignin decomposition and formation of phenolic compounds by further deoxygenation of lignin-derived monomers (Li et al., 2016b). The influence of biochar in catalytic microwave pyrolysis of same biomass source was examined by Dong et al. (2018). It was documented that biochar facilitated the production of gas at cost of bio-oil due to efficient cracking activity. The gaseous composition (H_2 and CO) was significantly higher under microwave heating (65.31 %) than conventional (60.05 %) with 20 % (wt.) of biochar dosage. The biochar was found to have more oxygenated groups that can react with high molecular hydrocarbon's side chains and resulting in the accumulation of $\text{C}_2\text{-C}_6$ compounds. The alkali content of the biochar showed positive effect on breakage and restructure of volatile heteroatoms and decomposition of high molecular hydrocarbons. The heating rate of the reaction increased with the content of biochar and is contributed by exothermic reactions during the process (Liu et al., 2014; Yek et al., 2020). The effect of catalyst to biomass (C:B) ratio and catalytic temperature using activated carbon via *in-situ* and *ex-situ* microwave catalytic pyrolysis emphasized for production of bio-oil to be enriched with

phenolic compounds has been studied by several researchers (Yang et al., 2018; Wang et al., 2020b). C:B ratio significantly affects the bio-oil yield in a negative manner where the lowest and highest yield was produced at same temperature (400 °C) with C:B ratio of 1.15 and 0.094 respectively. It was observed that high catalyst to biomass ratio (1:1) and low reaction temperature favoured bio-oil production with high phenolic content and activated carbon enhanced the catalytic conversion of guaiacols and lignin monomers into phenolic compounds (Yang et al., 2018). A pre-pyrolytic stage by means of conventional heating produced biochar was used as microwave absorber in subsequent microwave pyrolysis. Stepwise microwave synergistic pyrolysis enables short processing time, less consumption of energy, enhanced solidification of heavy metals via vitrification (Wang et al., 2020b). The pyrolysis of biomass is initiated with low temperature with the addition of carbon owing to decrease of activation energy based on the biomass species; facilitating efficient performance of microwave pyrolysis with low temperature (Salema et al., 2014). The microwave pyrolysis of spruce sawdust was aimed to increase the hydrogen or syngas yield using char derived from sawdust and or metal ions doped (Ni, Co, Fe) catalyst. The microwave pyrolyzed char was used as microwave absorber for *in-situ* catalytic upgradation of syngas. The char doped with Ni, Co enhanced the tar cracking and resulted in high yield of syngas with H₂ and reduced the liquid yield comparatively with non-doped char. Addition of some promoters to these metal-doped catalyst considering the reverse water shift gas reaction is necessary for efficient cracking and reforming (Lestinsky et al., 2017). The catalytic pyrolysis of corncob by iron modified biochar resulted in higher yields and the selectivity of cresol and phenol was observed by Dai et al. (2019). On contrary, certain biomass of high mineral constituents and lignin composition influence the reaction progression towards higher degree of reticulation to form a condensed polycyclic structure with the inhibition of macromolecular cracking to yield more solid char. Owing to high mineral content, the formed ash during the microwave pyrolysis inhibits the catalytic cracking process and decreases the gaseous yield (Collard and Blin, 2014).

4.3. Comparative study of microwave and conventional pyrolysis

Microwave heating has been opted as a potential heating source owing to elucidation of significant advantages over conventional pyrolysis. The accountable reasons include 1). Absence of heat transfer limitations within a customized reactor with limited residence time as it is an energy transfer process rather than simple heating (Chu et al., 2017); 2). Instantaneous reaction facilitating start-up of temperature and turn down (Haeldermans et al., 2020); 3). Reduction of pre-treatment process like decreasing the particle size to enhance the surface area to mass ratio, which facilitates easier downstream processing (Mohamed et al., 2021). The thermal and non-thermal effects correspond to unique nature of microwave irradiation to the polar molecules and that cannot be attained through conventional process (Arshanitsa et al., 2016; Liang et al., 2019b). Microwave pyrolysis has the ability for production of various grade or range of products because of linear temperature gradient caused by microwave radiation. The process efficiency and economics could be enhanced by microwave heating, while the same was least possible through conventional methods.

Many literatures have reported better product yield and enhanced physiochemical properties of the pyrolytic products generated through microwave in comparison with conventional heating in an electrical furnace/kilns. Huang et al. (2016) compared heating performance using single-mode microwave device and analytical pyrolysis using simultaneous differential scanning calorimetry-thermogravimetric analyzer using seven feedstocks. The heating rate of microwave pyrolysis (132 °C min⁻¹) is 42 % greater than the conventional (93 °C min⁻¹) pyrolysis and minimum time duration was needed to attain the highest temperature. The microwave pyrolysis heating performance is based on power

level, feedstock composition and it increased significantly with the combustible content. The time and required power input could reduce if the heating performance is increased by biomass containing high combustible content. The maximum temperature of microwave pyrolysis is lesser than conventional which comprehends that pyrolysis reactions occur at low temperatures when irradiated with microwave (Masek et al., 2013; Haeldermans et al., 2019).

The review by Kabir and Hameed (2017) has compared the merits of microwave heating over conventional heating and concluded that catalytic microwave pyrolysis could yield products from large particle size and higher moisture content compared to conventional pyrolysis. However, the energy efficiency of both process has not been detailed. Parvez et al., (2019a,b) used energy and exergy analysis as an evaluation tool for assessing the efficiency of the heating methods using gumwood biomass. The energy rate and the exergy rate increased with temperature and higher for microwave pyrolysis at each temperature. The pyrolysis system efficiency of conventional process is from 71.3 % to 73.6 % whereas, for microwave pyrolysis, efficiency ranges from 84.8 % to 89.9 %. The increase in efficiency corresponds to higher yield of gas and higher carbon content in biochar improving the combustion features. The energy and time consumption between microwave catalytic pyrolysis (MCP) and conventional catalytic fast pyrolysis (CFP) was studied using four different biomass by Fernandez and Menendez (2011). The microwave heating achieves lower heating time than conventional process, thereby leads to timesaving up to 60 % that increases with temperature and the energy saving up to 77 %. The specific power required to maintain the desired temperature for heating 50 ml of sample for 30 min is below 8 W g⁻¹ for MCP and over 10 W g⁻¹ for CFP. From the data reported, the energy consumption is 4 Wh and 5 Wh for MCP and CFP respectively. The energy conversion efficiency of conventional and microwave pyrolysis using activated carbon as catalyst was studied by Shi et al. (2020). Char and gaseous yields under microwave heating with reforming increased than conventional pyrolysis with reforming. This is mainly due to material selective heating nature and secondary cracking of non-condensable volatiles influenced under microwave heating which also mitigates potential corrosion difficulties associated with conventional pyrolysis. After the incorporation of catalytic reforming using activated carbon, the energy recovery efficiency has been increased from 5.56% to 6.91 % without reforming to 7.12%–8.49 % with reforming at 600 °C implying the significance of reaction enhancement. Zhang et al. (2019) executed a comparative study using pyrite and reported that microwave heating reduces the activation energy required for the process from 199.76 kJ mol⁻¹ to 153.72 kJ mol⁻¹ and there is uniform distribution of elements, many pore structures in lower temperature with a short residence time. Microwave heating catalyses the gas-carbon reactions by minimizing the Boudouard reaction's apparent enthalpy and activation energy compared to conventional methods (Hunt et al., 2013; Lam et al., 2018). The lignin isolated from softwood using microwave heating promoted the proportion of intact aromatic rings and intact side chains in lower proportions than conventional heating at same temperature. This facilitates the potential of lignin to be utilized as a source for low molecular weight aromatic compounds in higher yields in a cost-effective manner (Zhou et al., 2017). It was apparent that microwave pyrolysis characterized by high temperature improved the energy and exergy rate of pyrolysis rather than conventional pyrolysis.

Microwave catalytic pyrolysis (MCP) enhances more heat transfer than conventional catalytic fast pyrolysis (CFP). The difference in yield of bio-oil and composition obtained from MCP and CFP of kraft lignin was reported by Farag et al. (2016). Liquid product contains chemicals of more than 40 % and water less than 27 % compared with conventional fast pyrolysis. Microwave heating produced slightly higher liquid yield using char as microwave absorber, which enhances the heat conversion and promotes thermal decomposition of the lignin network. The unification of mass and heat transfer directions preserves aliphatic hydroxyl group and structure of lignin vapours, and the water content is

decreased in the liquid product in comparison with conventional CFP. Omoriyekomwan et al. (2016) performed the comparative study between MCP and fixed bed catalysis using lignite char and activated carbon and observed the increase of selectivity of phenol under microwave than fixed bed catalysis. The biochar underwent contraction and densification under conventional pyrolysis, whereas development of fluidity was observed on biochar surface under microwave heating.

MAP based biochar is more porous due to unique heating rate and better catalytic activity attributing to abundant functional groups on the surface than obtained under conventional heating (Mohamed et al., 2016b; Li et al., 2020a). The difference between proximate composition of biochar produced from conventional and MAP has been analysed and described through ternary plot (Fig. 3a). The data has been retrieved from specific literatures which has compared the proximate composition of MAP and conventional biochar. The biochar obtained through microwave pyrolysis contains more fixed carbon content than conventional pyrolysis, which makes the former process much more carbon negative, compared to the latter. The biochar's surface area obtained through microwave and conventional pyrolysis has been analysed (Fig. 3b). It was observed that microwave produced biochar possess greater surface area which enables its utilization for adsorption of contaminants, water retention and other environmental applications. These experimental entries resolved the limitation barrier associated with the conventional pyrolysis and explored the potential efficiency of microwave pyrolysis in enhancing the physicochemical properties of pyrolytic products with less energy input and favoured for environmental applications. Besides the advantages mentioned before, the few more includes efficient heating process by reduced heat loss; less production of side products; increased quality of bio-oil and higher gas yield fraction with high syngas (CO and H₂); controlling parameters to obtain products of good quality and quantity and it could be produced in a small size reactor that is cost-effective; equipment portability and process flexibility make it feasible for industrialization (Amoroso et al., 2020). Microwave assisted waste cooking oil transesterification showed the conversion of 96 % to biodiesel higher than conventionally heated reactor owing to synergistic effect of catalyst and microwave heating mechanism. Utilization of this waste cooking oil methyl ester reduces the emissions of particulate matters and NOx up to 21.7 % and 7.8 % respectively compared to

petrodiesel (Ali et al., 2020). Microwave assisted production of palm kernel oil biodiesel rapidly reduces the CO and HC and increases NOx in exhausts compared to conventionally produced fuel (Allami et al., 2019). From these observations, it could be interpreted that microwave irradiation influences the properties of fuel and reduces the exhaust emissions like NOx and particular matter compared to conventional method. This prospective tool in enhancement of the processing of different lignocellulosic materials efficiently can be taken into account that the knowledge obtained could be implemented not only for biofuel production but also for the development of improved or new stages of biorefinery-associated transformation of biomass or self-sufficient processes. The effective upgrading of pyrolytic products and subsequent reduction of undesired product formation could balance the energy consumption sufficiently and relative high cost in microwave systems that might progress the application of the microwave-based technique to industrial sectors.

5. Activation of biomass

The recalcitrance of biomass to bioprocess conditions depends on the intrinsic properties of biomass which includes cellulose crystallinity, degree of polymerization, arrangement of interfacial layer components, structural heterogeneity (Guerriero et al., 2016). Hence, pre-treatment conditions are needed which facilitates the improvement of quality through modifications or removal of functional groups and biomass surface structure which enhances the conversion and process optimization. The pre-treatment techniques signify 20 % of total cost of the process and it adds up to the input energy reduction for the process (Mafe et al., 2015; Seidl and Goulart, 2016). The current practice of pre-treatment methods includes physical (grinding, densification), chemical (acid, alkali, metal oxides), thermal (drying, torrefaction) and biological means. The activation process is the crucial stage in which high temperature and suitable activating agents are employed for optimum production of desired engineered biochar. Kołtowski et al. (2016) reported that activated carbon is observed to be more efficient than biochar in contaminant immobilization. Activation of biochar surface area can enhance the efficiency in reducing bioavailable contaminants concentration. This activation can be carried out using thermal

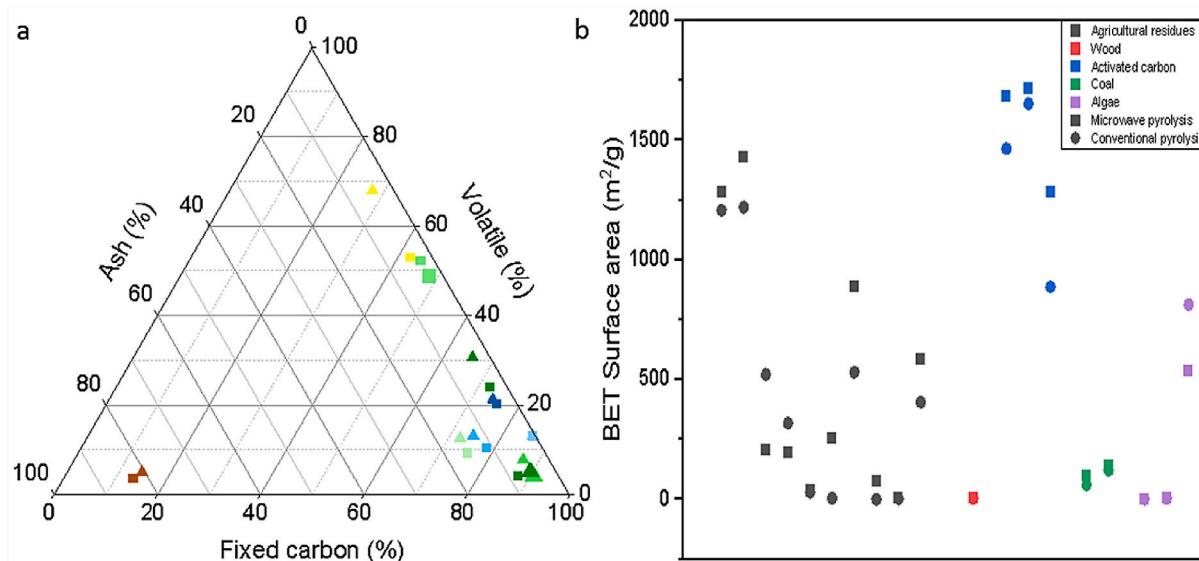


Fig. 3. a). Ternary plot of proximate composition of solid waste (brown), agriculture biomass (blue), wood (yellow), other waste (green) produced through microwave (square) and conventional (triangle) heating methods (Data has been retrieved from (Alslaibi et al., 2014; Dominguez et al., 2006; Haeldermans et al., 2019; Li et al., 2019; Liu et al., 2020a; Parvez et al., 2019a; Wu et al., 2015; Xin-Hui et al., 2011)). b). Comparison of the specific surface area of biochar produced from feedstocks under the influence of microwave and conventional heating (Data has been surveyed from (Abas and Ani, 2014; Abdelsayed et al., 2018; Chu et al., 2017; Duan et al., 2012; Ferrera-Lorenzo et al., 2014; Huang et al., 2011; Mohamed et al., 2016a; Mubarak et al., 2016)). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

(torrefaction), chemical (magnetic impregnation, acid or base) activation. (Yap et al., 2017; Yek et al., 2020).

Treatment methods are selected in a way considering the desired product output and quality (Lee et al., 2019). Methods like torrefaction with acid/salt treatment facilitates the removal of alkali and alkaline compounds which shows detrimental effect on bio-oil production, as these metals catalyse the decomposition and polycondensation reaction for biochar formation. Chen et al. (2021) has specified that pyrolysis bio-oil obtained from torrefied biomass is characterized by lower furfural, organic acids and aldehydes, whereas, aliphatic groups, aromatic anhydrous sugars, ketones, phenols and hydrocarbons are in higher amount. Several activating agents leave the particle core intact and some agents show large and deep cavities accessing the particle core resulting low structural integrity (Villota et al., 2019).

5.1. Physical activation

The physical treatment like grinding, densification minimizes the loss of material during the treatment process, as no washing steps are needed. The minimal mass loss is attributed to mechanical disruption associated with the stress caused during the treatment exposure, which modifies the surface topography of the biomass material. Apparently, the size reduction and increase in surface area can favour faster heat propagation and an increased pyrolytic conversion (Cherpozat et al., 2019). Energy yield obtained is often more than the mass yield indicating the improvement of heating value and energy densification (ratio of higher heating value of product to biomass). Physiochemical properties of torrefied biomass were found to be comparable with that of coal-like fuel and exhibits favourable combustion behaviour (Wilk and Magdziarz, 2017). The temperature needed for volatile release was reduced and the homogenous structure was favoured by enhancement of cellulose and lignin content. The pre-treatment using torrefaction has significant influence on kinetic and reaction flow parameters and reaction mechanism, however it was observed that more energy is needed by torrefied biomass to proceed with the pyrolysis process (Doddapaneni et al., 2016). The ultrasonic treatment enhances the surface cleaning of the biomass with open structured, which enhances the accessibility and particle surface erosion. But the scale-up process of ultrasonic treatment did not result in a significant improvement in surface characteristics and product yield (Cherpozat et al., 2019). The heat treatment of biomass results in high degree of aromatic condensation that leads to large size of aromatic clusters as like the chemical activation, which increases fractions of non-protonated carbon and aromatic carbon content. Subsequent chemical and heat treatment had resulted in formation of aromatic carbon of more condensed structures by volatile carbon fraction release and structure transformation by heat, leading to formation of highly aromatic solid products compared to pristine biochar (Nair and Vinu, 2016). The combination of physical pre-treatment enhances the fuel characteristics by removing the inorganic species to mitigate its associated problems based on product selectivity (Zhang et al., 2015).

5.2. Chemical activation

Feedstock material impregnated with dehydrating agent and followed by heat treatment resulted in porous carbon structure. Chemical activating agents commonly used include metal salts such as potassium carbonate (K_2CO_3), potassium hydroxide (KOH), zinc chloride ($ZnCl_2$), alkalis and acids (Ferrera-Lorenzo et al., 2014; Yap et al., 2017). Activation of biochar by means of alkali or carbonates based activating agent facilitates redox reactions with the carbon structure. Metal salt impregnated biomass promotes the reaction towards biochar formation by catalysing the repolymerization of volatiles during the pyrolysis process (Choi et al., 2019). The metallic atoms were found to pierce the biochar's inner structure through intercalation, thereby enlarging the pores that are existing and develop new pores further (Mao et al., 2015; Villota et al., 2019; Ge et al., 2020). Compared with physical activation

method, chemical activation consumes less energy and production cost and produces biochar with high surface area and product yield in low activation time. The added chemical may also act as catalyst in pyrolysis process and enhance the production of gas under CO_2 atmosphere (Cho et al., 2016). Lee et al. (2019) observed that addition of calcined seashell reduces the char agglomeration and increases quality of bio-oil via pyrolysis in fixed bed reactor. Acid pre-treatment declines the surface area with increase of concentration owing to decrease in number of pores which enhances the mass transfer resistance of organic matter, subsequently reducing tar yield and promoting char yield. It removes alkali and alkaline earth metals which promotes oxygen-containing gases (CO_2 , CO) and CH_4 , henceforth inhibits these compounds and increase H_2 production (Ye et al., 2017; Li et al., 2019). Metal impregnated biochar contained large amount of certain mineral elements, which could be regenerated and permitted to use as fertilizer. Mg loaded biochar enhanced the surface deposition of phosphorous and precipitation through chemical reaction with Mg particles and thus, increases its bioavailability facilitating the slow-release of nutrients from the fertilizer (Yao et al., 2013; Choi et al., 2019). Zhang et al. (2015) reported that water-washing treatment decreases the solid and gaseous pyrolyzates and increases the liquid yield obtained from microwave pyrolysis. However, essentially utilizing the washing process for excess chemical removal pertaining to surface is often costly and time-consuming and further generates large amount of wastewater.

5.3. Microwave-assisted activation

The microwave radiation is more recent approach of biomass pre-treatment compared to conventional process (Undri et al., 2014b). The microwave activation is being assisted with an activating agent like CO_2 , steam and chemical agents for improved activation. The activation mechanism of using CO_2 or steam under microwave radiation involves Boudouard reaction and gasification of steam as explained in (Eqs. (4) and (5)) respectively. These are endothermic reactions requiring more energy to maintain high temperature which is facilitated by beneficial utilization of microwave heating, resulting in better activation (Hunt et al., 2013; Yek et al., 2020). The microwave-assisted biomass activation can be implemented by a single step (simultaneous carbonization and activation) or two-step process (carbonization and activation are done at separate steps). The microwave pre-treatment produces numerous open cavities of various size and shapes over the surface and reduce the mass and heat transfer limitation thereby enhancing rate of conversion and product formation (Mao et al., 2015; Ganesapillai et al., 2016).

5.3.1. Single-step microwave carbonization and activation

The single step of simultaneous microwave heating and activation of biomass favours higher heating rate, steady increase of temperature profile, reduces heating energy consumption and time. The temperature profile of microwave-assisted activation involves four stages namely drying, more heating, devolatilisation and carbonization, activation with varying heating rates (Latiff et al., 2019; Lam et al., 2020). Single-step microwave pyrolysis with steam or CO_2 as activating agents was performed using orange peel waste. The biomass was irradiated with microwave power of 1 kW and gasifying agents were supplied from the bottom of microwave cavity when the temperature reached 100 °C. The activation step of either CO_2 or steam was reported to occur when the temperature reaches 500 °C. Since, steam activation requires lower energy for production of CO and H_2 (Eq. (5)), it showed higher heating rate than CO_2 (Yek et al., 2020). Single-step microwave pyrolysis combined activation by steam using waste palm shell was adopted to produce activated biochar to be used as adsorbent in hazardous landfill leachate. The carbon content and stability of activated biochar was increased as revealed through H/C (0.5) and O/C (0.1) ratios which indicates the degree of activation. The microwave activated biochar had chemical oxygen demand removal of 65 % and 595 mg g⁻¹ as maximum

adsorption efficiency as observed by Lam et al. (2020). The salacca peel was used as an adsorbent after microwave-assisted KOH activation and adsorb methylene blue of 375.9 mg g^{-1} of adsorbent (Klemantan et al., 2020) whereas, Chen et al. (2020) obtained porous carbon of super high surface area of $3065 \text{ m}^2 \text{ g}^{-1}$ employing inorganic salts in lignin as microwave absorber through microwave assisted KOH activation which showed high capacitance retention of 79.4 % with high current density of 50 Ag^{-1} .

5.3.2. Two-step microwave activation and carbonization

Two-step process involves the separate carbonization and activation process to occur subsequently. Ganesapillai et al. (2016) performed pre-treatment of crude glycerol – olive kernel in domestic microwave oven and carbonization in wire mesh type reactor and reported that chemical composition of sample was not significantly affected by microwave pre-treatment excluding partial drying. However, the treatment resulted in the enhancement of anhydro-sugars production attributing to lignin dissolution, ash removal and enrichment of crystalline cellulose. It presumably indicates efficient method of obtaining fermentable sugars from lignocellulose biomass by fast pyrolysis (Jiang et al., 2020a). Surface characteristics of activated biochar have also been found to vary drastically according to the treatment techniques. The hydrogen peroxide assisted microwave pyrolysis caused microscale explosions that resulted in micron and nano-sized pores. Higher microwave power enhanced the removal of oxygen-containing groups where its persistence weakens the dispersive force ($\pi-\pi$ bond interaction) between dye and the biochar aromatic units (Nair and Vinu, 2016). The impact of KOH and phosphoric acid (H_3PO_4) activation of cocoa pod husk (CPH) on modified textural properties of activated carbon under microwave heating was evaluated by Villota et al. (2019). H_3PO_4 -activated carbon was mesoporous (average pore diameter of 3.23 nm) whereas; KOH-activated carbon was microporous (average pore size of 2.19 nm). H_3PO_4 activated CPH had more desirable properties with higher yield and therefore it could be comprehended that H_3PO_4 is better activating agent in physio-sorptive characteristics and KOH is the better reactive activating agent since it caused more severity loss and low structural integrity of the product. Microwave assisted KOH activation of orange peel was performed by Lam et al. (2017b) and reported that it is one of efficient method to produce activated carbon of higher surface area and could be used for dye adsorption for wastewater treatment. The char obtained from microwave heating was steam activated and impregnated with metal and analysed for the microwave catalytic cracking and reforming of toluene for syngas production. The average conversion of toluene was achieved at 800°C for 120 min to 95.19 % through combined reforming by maintaining the stability of the activated char which lead to energy efficiency of 57.8 % (Li et al., 2020a). The microwave-assisted solvothermal pre-treatment for vacuum pyrolysis using ashe juniper waste increased the heavy and light bio-oil fractions and shifted the decomposition stages to higher temperatures attributed to lignin and extractable ash removal (Choi et al., 2019). The microwave activated biochar after washing contained higher surface area and pore volume because of rapid devolatilisation and resolidification of carbonaceous materials under microwave heating and the removal of alkali metals catalysed the secondary char formation (Zhang et al. 2015).

5.3.3. Effect of influential parameters on microwave-assisted activation

The efficiency of activation depends on operating conditions like impregnation ratio, carbonization temperature, time and biomass composition. These parameters are being optimized for production of activated biochar with defined textural properties and favour its utility in efficient removal of contaminant heavy metals, dyes, pharmaceuticals, potential electrode material in biosensor; energy storage application and soil conditioner (Ahmad et al., 2017; Saygili and Saygili, 2019). The interaction of microwave power, irradiation time and carbon-based materials influences the microwave heating on biochar modification.

The temperature of the process is significantly influenced by microwave irradiation time and range of microwave power (Lam et al., 2017a; Ge et al., 2020). Microwave irradiation time has dynamic effect on surface area and pore volume where longer exposure was reported to increase the original pore size and develop new pores whereas the extreme microwave heating period caused cracking and destruction of pores (Mubarak et al., 2016; Lam et al., 2018). Biochemical content of biomass like low moisture and ash content, higher volatile content facilitates the development of interconnected pores due to enhanced diffusion of activating agent into biochar (Lam et al., 2020). Effect of microwave pre-treatment on chemical composition of woody biomass revealed the non-promoting effect on the condensation process at 240°C – 280°C increasing anhydrous sugar production and cellulose crystallinity (Arshanitsa et al., 2016; Jiang et al., 2020a). Microwave steam activation was carried out after self-purging pyrolysis of waste palm shell and the correlation between the activation parameters has been obtained through optimization process. The highest yield of activated biochar was observed at low temperature in short time because of secondary cracking of gasification reactions between carbon and steam at high temperature. The loss rate of biochar was directly proportional to activation time where prolonged time of activation led to release of more volatiles components (Wang et al., 2014; Yek et al., 2019). Amount of activating agent to be coated on biochar was determined by impregnation ratio and it has to optimized for defined surface area as the thermal decomposition of hydrated agent results in blockage of pores and decrease in surface (Mubarak et al., 2016). The production of KOH activated biochar of pinewood chips and wheat straw was performed by Mao et al. (2015) through muffle furnace mediated carbonization and microwave-assisted activation with 600 W for 30 min to analyze the activation parameters on iodine number and the yield. The iodine number of activated biochar steadily increased with decrease in size of activated carbon and higher impregnation ratio. In large size particles, the restricted access of KOH leads to reduced activation reactions and the width of pore was broadened successfully with new micro and mesopores (Chen and Hashisho, 2012; Mao et al., 2015).

5.3.4. Analytical techniques to evaluate product characteristics during microwave activation

Microwave-assisted pre-treatment not only enhances the product yield but also improves its quality in terms of better physiological properties and chemical composition. Techniques like Pyrolysis–gas chromatography/mass spectrometry (Py-GC/MS) and the thermogravimetric analysis in combination with Fourier transform infrared spectroscopy (TG-FTIR) are commonly utilized to analyze the product composition as detailed in subsequent sections.

5.3.4.1. Pyrolysis–gas chromatography/mass spectrometry (Py-GC/MS) analysis.

Py-GC/MS is one of the characterization techniques used for analysis of composition and structure of materials. The sample is subjected to decomposition to generate smaller molecules and that is carried by gas chromatography and detected using mass spectrometry. Data acquisition from Py-GC/MS characterization for microwave treated softwood regarding carbohydrate-derived volatiles showed hemicellulose destruction and feasible recrystallization of amorphous region of cellulose. The result also revealed the existence of higher content of demethoxylated phenols in microwave irradiated samples indicating the higher availability of lignin sites for the reaction and achievement of more condensed lignin structure than under conventional heating (Arshanitsa et al., 2016). The microwave-assisted wet torrefaction of corns stalks with ammonia resulted in the decrease in peak area ratio for acetic acid, increase in furan content and approximately two-fold enhancement of glycolaldehyde and ketones which resulted in more formation of levoglucosan intermediate (Hu et al., 2019). The analysis of data by Ding et al. (2018) using Py-GC/MS for microwave pre-treated fast pyrolysis of pine sawdust and water hyacinth indicated the

product distribution under different levels of power and time with calcium oxide (CaO) as catalyst. Under microwave irradiation, CaO was found to enhance the neutralization of the acids into ketones resulting in high yield of phenols in case of pine sawdust, whereas the latter contained less phenol owing to low lignin. The microwave pre-treatment was found to reduce the activation energy offsetting the limitation of CaO addition (Liang et al., 2019a, 2019b, 2019b). The investigation on pyrolytic products enabled to identify the chemical structure of parent material since the presumption that in lower or higher degrees, the structural units of pyrolytic products embody the original structure of macromolecule. The analysis of low amount of lignin-derived components obtained during alkaline lignin pyrolysis suggested that lignin obtained under the influence of microwave treatment attained a condensed structure (Sequeiros and Labidi, 2017; Davila et al., 2019). The microwave-assisted isolation of lignin from softwood permitted less significant degradation and well-preserved structure. Based on the ratio of ion current peak, area between the biomass and microwave-assisted isolated lignin, the structural arrangement and the possible mechanism of phenolic compounds formation could be understood. And also it gave an obvious indication of cyclic aliphatic interlinkage formation between the monomer units which were in close proximity under the assistance of microwave radiation (Zhou et al., 2017).

5.3.4.2. Thermogravimetric analyzer with fourier transform infrared spectrometer (TG-FTIR). Thermogravimetric analyzer coupled with Fourier transform infrared spectrometer (TG-FTIR) enables a process assessment through the TG curve quantitatively and the decomposed products could be identified with the help of IR spectra of the evolved gases. The TG-FTIR pyrolysis data of biomass blends subjected to microwave irradiation revealed the presence of CO₂ (2358 cm⁻¹ and 669 cm⁻¹) and CO (2240–2400 cm⁻¹ and 650 -1000 cm⁻¹) gases respectively. Amount of CO₂ and CO decreased with increase in carbon percentage and its peak intensity is decreased with temperature increase which confirms a synergistic effect between carbon content and biomass pyrolysis (Tian et al., 2010; Salema et al., 2014). The microwave hydrothermal treatment employed for hemicellulose isolation from bamboo was subjected to TG-FTIR analysis by Luo et al. (2017b) indicating that pyrolysis behaviour is not affected significantly by microwave treatment at lower temperature (140 °C). The study on the synergistic effect of bitumen plasticization and devulcanization of ground tire rubber under microwave treatment was established by Zedler et al. (2018). It was reported that temperature required for 2 % weight loss is lesser for microwave treated samples than untreated because of reclaiming effect of microwave, which released higher amount of products that are sensitive for degradation at lower temperature. The amount of char was less after microwave treatment because of devulcanization that eliminated low molecular weight compounds. It was obvious from the 3D FTIR data that the major released products were aliphatic hydrocarbons exhibiting strong absorbance bands at 2800–3000 cm⁻¹ after slow pyrolysis. Other than TG-FTIR, Thermogravimetric analysis coupled with mass spectrometry analysis (TG-MS) was also used to analyze product characteristics. Li et al. (2020c) studied the thermal stability and depletion of char supported catalyst using TG-MS. The microwave activated char based catalyst is more stable even up to tar reforming temperatures (900 °C). The activated char showed less influence on the reverse water shift gas reactions, which spontaneously occurred with temperature increase (>700 °C) and demonstrated the stability of activated carbon for *ex-situ* hot gas conditions.

5.3.5. Benefits of microwave assisted activation and product application

The effect of microwave on the evolution of surface characteristic depends on the biomass characteristics. Carbonization and unsaturated development of matrix structure have been observed in softwood whereas in hardwood, only carbonization process has been promoted under the influence of microwave pre-treatment, these are associated

with the non-thermal effect of microwave radiation (Motasemi and Afzal, 2013; Arshanitsa et al., 2016). Chemical structure of matrix will be thermally stable with pores that are more consistent after microwave pre-treatment than conventional pre-treatment under similar temperatures. Latiff et al. (2019) reported that microwave treatment corrects the pore structure and functional groups at biochar surface to yield high performance adsorbents. The microwave activated samples facilitate higher initial sorption rate and could be successfully regenerated with stable sorption capacity (Liu et al., 2018; Shukla et al., 2019). Besides development of surface area and pore structure, significant difference in activation methods also influences the availability of nutrients that can be supplemented to soil by its application (Mohamed et al., 2016a, 2016b; Su et al., 2020). High activation temperature needed for CO₂ and H₂O assisted pre-treatment with longer residence time reduces the nutrients in biochar compared to microwave treatment. In microwave, the feedstock combustion is point-wise proceeding at lower temperatures thereby resulting in minimal nutrient loss (Koltowski et al., 2017a,b). The studies utilizing MAP of activated carbon (AC) for environmental applications have been listed in Table 3. The cost of AC production by microwave is sufficiently low compared to commercial activated carbon (Yek et al., 2019). More emphasis has to be implemented on optimization of the microwave-assisted activation methods to balance between the cost and performance of activated biochar production depending on the scenario of remediation.

6. Application of microwave produced biochar in agriculture

The energy-environment nexus represents an essential interweaving and interstate network, as these areas have been degraded heavily due to the anthropogenic activities over the past decade. Recent research has emphasized the application of microwave-based pyrolysis under the concept of biorefinery, to address the scarcity of water, fulfill the energy demands and to avert the environmental degradation issues.

MAP employing suitable additives/catalysts enhances the heating rate, reducing energy consumption and producing high-grade bio-oil and gaseous by-products that could be utilized as an alternative fuel. The biochar produced during MAP could be used for immobilization of heavy metals, and adsorption of dyes and contaminants from soil and water resources, thus acting as an environmental protective agent. Biochar also acts as a potential source of low-cost nutrients for soil and plant growth enrichment, reducing the utilization of chemical fertilizers, which have been found to degrade soil fertility and cause eutrophication. The enhancement of soil ability to withhold the water and nutrients is also considered one of the crucial needs for increasing the bioenergy plant growth in desert, arid and semi-arid lands. To retain bioenergy crop productivity in future, biochar application as a supplement to soil mitigates portended deficiencies in water supply that have resulted owing to climate change effects. The production and application cost of biochar to improve the soil health along with the irrigational costs could be offset by the benefits of nutrient supplementation and water retention aided by the char use. The use of biochar and its various mechanisms to establish a sustainable agroecosystem has been delineated in the subsequent section.

6.1. Potential insights of microwave produced biochar properties to act as soil conditioner

The biochar potential for the soil remediation is reliant on two main components such as carbonaceous matter and the entrained minerals (ash), which contribute to functions like adsorption and retention of water and organic pollutants; provision of nutrients releases and improvement in soil pH respectively (Qian et al., 2016) which are the main indicators for evaluating the biochar amended soil as suggested by He et al. (2021). Application of microwave absorbers enhanced the pyrolysis temperature, producing biochar rich in aromatic carbon content elucidated by solid-state cross polarization/magic angle spinning

Table 3

Microwave-assisted production of activated carbon with energy requirement and production cost.

Feedstock	Carbonization conditions	Activation conditions	Activated carbon recovery	Characteristics	Application	Microwave Energy consumption (kJ)	Cost of AC production (USD kg ⁻¹)	Reference
Waste palm shell Two-step Carbonization and activation	Power: 700 W Time: 25 min	Microwave Steam activation temp- 543 °C; 9.95 min	85.30 %	Surface area 570.8 m ² g ⁻¹ ; Pore volume: 0.262 cm ³ g ⁻¹ ; Pore diameter: 3.56 nm	Adsorption efficiency for methylene blue- 38.5 mg g ⁻¹	NA	0.68	Yek et al. (2019)
Banana peel Two-step Carbonization and Activation	Power: 700 W Time: 20 min N ₂ flow: 0.25 L min ⁻¹	KOH-NaOH IR: 1.0 IT: 24 h Power: 700 W for 10 min	24 %	C-75 %; N-1%; H-2%; O-22 %; Surface area: 1038 m ² g ⁻¹ ; Pore volume: 0.73 cm ³ g ⁻¹ ; Pore diameter: 25 nm	Adsorption efficiency for malachite green dye Removal efficiency of 90 %	1470	0.90	Liew et al. (2018b)
Palm kernel shell Two-step Carbonization and Activation	Microwave vacuum pyrolysis (MVP) 700 W, 25 min	NaOH-KOH mixture IR: 1.0 IT 24 h MVP for 10 min	79 %	C-85.2 %; N-0.5 %; H-3.8 %; O-10.5 % Surface area: 1320 m ² g ⁻¹ ; Pore volume: 0.70 cm ³ g ⁻¹ ; Pore diameter: 6.6 nm	Metallic AC -BOD removal-85 %, COD removal-89 %	1470	4-10.2	Liew et al. (2019)
Almond shell Two-step Carbonization and Activation	Electrical tubular furnace At 700 °C for 2 h N ₂ flow: 80 ml min ⁻¹	Microwave H ₂ O ₂ activation IR: 1:10 Power: 600 W for 12 min	33.5 %	Surface area: 1274 m ² g ⁻¹ ; Pore volume: 1.673 cm ³ g ⁻¹ ; Pore size: 2.82 nm	Adsorption capacity for sulfamethoxazole- 344.8 mg g ⁻¹	10,800	27.8	Zbair et al. (2018)
Durian seed Two-step Carbonization and Activation	Vertical tubular furnace 500 C; 1 h N ₂ flow: 150 cm ³ min ⁻¹	KOH IR: 0.83 IT: 24 h at 110 °C Power 440 W for 4 min	25.77 %	Surface area: 852.30 m ² g ⁻¹ ; Pore volume: 0.465 cm ³ g ⁻¹	Adsorption capacity for Methylene blue- 82.14 %	NA	NA	Ahmad et al. (2017)
Tannery sludge Single-step Activation and Carbonization	Power: 1200 W for 150 s - 4 cycles for 20 min N ₂ flow: 100 ml min ⁻¹	Zinc chloride (40 %); Lime (10 %); 6 M Hydrochloric acid	60 %	Surface area: 491 m ² g ⁻¹ ; Pore volume: 0.440 cm ³ g ⁻¹ ; Pore diameter: 4.69 nm	Adsorption capacity for Acid black 210-1108 mg g ⁻¹ Acid red 357-589.5 mg g ⁻¹	1440	32.43	Puchana-Rosero et al. (2016)
Prosopis juliflora Two-step Carbonization and Activation	Mixture of biochar produced IR: 1:15 Power: 280, 420, 560, 700 W Time: 6-15 min Susceptors used: Aluminum, graphite, silicon carbide, fly ash	Hydrogen peroxide IR: 1:15 IT: 24 h Power: 600 W for 10 min	50-70 %	C-75.13 %; N- 1.67 %; H-0.21 %; O-22.99 % Surface area: 357 m ² g ⁻¹ ; Pore volume: 0.13 cm ³ g ⁻¹	Adsorption capacity for Methylene blue- 91 mg g ⁻¹ Remazol Brilliant Blue R -83.3 mg g ⁻¹	NA	NA	Nair and Vinu (2016)
Coconut shell Two-step Activation and Carbonization	Power: 800 W Irradiation time: 20 min N ₂ flow: 0.2 ml min ⁻¹	Ferric chloride hexa hydrate (FeCl ₃ ·6H ₂ O) IR: 0.5 IT: 4 h at room temperature, dried at 100 °C	67.37-88.98%	Surface area- 834 m ² g ⁻¹	Adsorption capacity for Cadmium - 4.77 mg g ⁻¹ Lead-4.96 mg g ⁻¹ Saturation magnetization of 6 emu g ⁻¹ .	NA	NA	Yap et al. (2017)

C-Carbon; H-Hydrogen; O-Oxygen; N-Nitrogen contents; BOD – Biological oxygen demand; COD- Chemical oxygen demand; NA- Not available.

¹³C nuclear magnetic resonance spectroscopy (Haeldermans et al., 2019). The biochar produced from lower microwave power level/intermediate conventional pyrolysis (350 °C–450 °C) is less aromatic and can also be used as soil amendment (Kostas et al., 2020). It was also documented that MAP biochar has developed more finite pores and responsible for increased water holding capacity (WHC) compared to biochar produced from conventional method under similar conditions (Mohamed et al., 2016b). The biochar can be activated to enhance the physicochemical properties like surface area, pore volume under

microwave with superheated steam or carbon dioxide atmosphere to immobilize organic contaminants, reducing polycyclic aromatic hydrocarbon's exposure during its use as soil conditioner (Koltowski et al., 2017a,b). A significant positive correlation exists between biochar micropore area and soil WHC and cation exchange capacity (CEC) (Afzal et al., 2018). Potential benefits of improving germination rate, shoot and root length, nitrogen uptake and usage efficiency compared to chemical fertilizer has also been established in the study by Wen et al. (2017).

6.2. Featured applications of biochar for soil enrichment

Owing to high porosity, surface area and enhanced aromatic carbon and mineral content, the microwave produced biochar is suitable for soil remediation because of acidic soil neutralization, increased water retention and accommodation of nutrients (Yang et al., 2015; Mohamed et al., 2021).

Specific surface area and CEC of biochar presumably determine the soil's ability for retaining water and other nutrients improving soil fertility and nutritional efficiency. The higher surface area associated with increased microwave power produces numerous adsorption sites retains more nutrients and water and alleviate its deficiency during the young fruiting stage of the plant (Wen et al., 2017; Liew et al., 2018a). Microwave produced biochar contains higher CEC which aids in the covalent bond formation between the cations (K^+ , Ca^+ , etc.) and the lone pair of oxygen in water and thereby enhancing the biochar's ability to retain more water. The more stability of biochar structure with low O/C ratio consisting of stable pores formed due to microwave heating provides favourable habitat for bacteria and fungi that can promote growth of plants (Nam et al., 2018; Su et al., 2020).

The biochar supported growth of Oyster mushroom and showed an impressive growth rate with significant production yield of 27.5 g of oyster mushroom per gram of biochar after 35 days further concluded that biochar as plant growth-promoting agent owing to higher carbon content which is highly resistive for hydrolysis and chemical reactions (Nam et al., 2018). Biochar addition in the mushroom substrates retained moisture content in both unspawned and spawned substrates (Mahari et al., 2020). Catalytic microwave pyrolysis using sodium (Na) based catalysts in a microwave muffle reactor was found to enhance the carbon content and carbon sequestration rate, minimized nutrient decomposition rate significantly during soil amendment, thereby, enhancing its quality (Hossain et al., 2017). The combination of two additives on biochar production leads to synergistic effect on increased soil WHC as a consequence of enhanced microporosity of biochar owing to microwave heating (Mohamed et al., 2016a, 2016b, 2016b). The microwave-assisted steam activated biochar showed adsorption efficiency of 11 mg g^{-1} of 2,4-D herbicide within a shorter time-period comparable to activated carbon produced by other methods thereby

preventing contamination of surface water in agricultural land (Lam et al., 2018). The sustainable use of nutrient-loaded biochar can be supplemented for soil nourishment obtained from microwave pyrolysis (Shukla et al., 2019). Utilization of biochar as a biological carrier for the growth of lettuce in aquaponics was demonstrated by Su et al. (2020). Due to higher surface area and biofilm formation, the biochar supplementation facilitated nitrification in aquaponic effluent ($29.7 \text{ mg L}^{-1} \text{ week}^{-1}$) and supplied nutrients (110 mg of nitrogen per plant) promoting vegetative growth of lettuce (70 g).

6.3. Featured characteristic of biochar for heavy metal immobilization

The higher pH and CEC produced under microwave treatment enhances the metal precipitation and simultaneous reduction of its solubility. It enhances the exchange of cations and form co-precipitates or get complex with humic matters (Liu et al., 2018). Immobilization of heavy metals on biochar surface is regulated more importantly by ash composition of biochar. The higher heating rate of microwave produced biochar with higher ash content containing phosphates (PO_4^{3-}), carbonates (CO_3^{2-}) that increase the precipitation as metal phosphates and carbonates and also provide requisite nutrients (K, Ca, Mg, Al, Fe, Zn and P) to soil. The presence of functional groups like $-NH$, $-OH$, $C=C$, $C=O$, $Si-O-Si$ and $-PO$ facilitates the performance of metal immobilization process. The mechanism has been illustrated schematically in Fig. 4 (Yang et al., 2015; Qian et al., 2016). Catalyst assisted microwave pyrolysis of sewage sludge biochar increased heavy metals immobilization on the pores of biochar and thus reduced its bioavailability, which could otherwise deteriorate water and soil fertility. The heavy metal ions react with oxygen-containing groups at biochar surface and precipitate as metal hydroxides which then decomposes in presence of alkaline catalyst assisted microwave pyrolysis (Huang and Yuan, 2016; Sun et al., 2018; Hou et al., 2020). The microwave slow pyrolysis of sewage sludge prepared using pelletization and additives showed potential of immobilizing heavy metals on the structure of biochar presumably inhibiting its fixation, bioavailability for plants increasing the productivity (Racek et al., 2018). The oxygen-containing groups at surface of biochar are influenced by higher pH which deprotonates and enhances the adsorption efficiency. The microwave produced biochar at low temperature

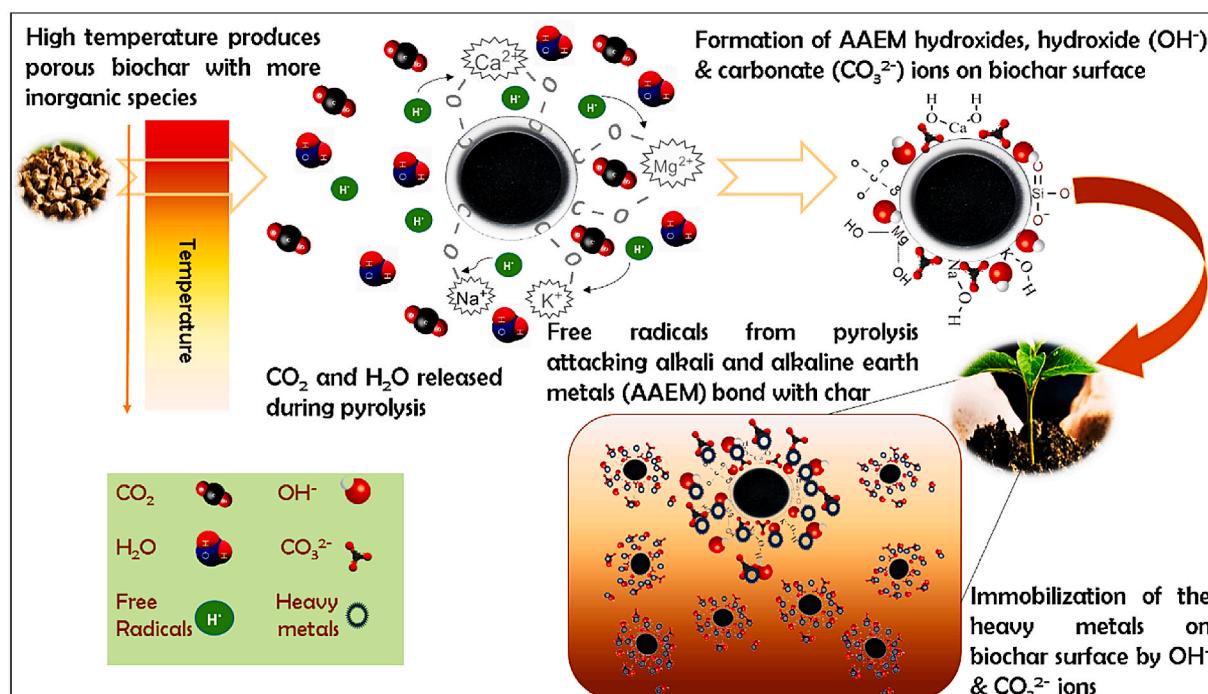


Fig. 4. Mechanism of microwave produced biochar in soil heavy metal immobilization.

(300 °C) reduced phytotoxicity and bioavailability of heavy metals and also increased rate of plant growth up to 145 % (Mohamed et al., 2017, 2021). Microwave irradiated biochar produced from slow pyrolysis beneficially reduced ecotoxicity and bioluminescence in soil and comprehended the microwave treated biochar is relatively more effective than other pre-treatment methods and non-activated biochars (Racek et al., 2018). Many literatures have stated that biochar is a suitable material with excellent biodegradation property for sustaining prominent environmental and agricultural benefits over fertilizers comprising long term carbon sink, enriching soil nutrient availability, microbial activity, fertility in turn leads to crop productivity (Ducey et al., 2013; Hossain et al., 2017; Liu et al., 2018; Lyu et al., 2020).

SWOT analysis of microwave-produced biochar for agricultural application has been presented in Table 4. Although advantages are well evident from the strengths and opportunities, it also emphasized that economic feasibility is a major weakness for adopting microwave-produced biochar in agriculture. Even though the quality of MAP biochar is superior than conventional biochar leading to 20 % increase in unit product price which is expected to generate higher revenues. However, due to higher capital expenditure (CAPEX) owing to higher installation cost, the discounted payback period is higher reducing the overall process feasibility (Haeldermans et al. 2020). It was observed that there are deficient studies in scaling up of prototype for the biochar production to be used in agriculture and more studies should be focussed on reducing the CAPEX. From the above entries, it can be concluded that biochar produced under microwave radiation is potent enough to adsorb the heavy metals, dyes, organic pollutants and helps in remediation of environment efficiently. The principle of carbon sequestration in biochar greatly enhances the mitigation of climate change. As evident in Table 1, the higher heating value of microwave produced biochar along with the upgraded bio-oil and gaseous by-products obtained makes the microwave assisted strategy desirable in terms of high energy recovery efficiency. Thus MAP biochar facilitates effective management of the nexus between the energy and environment and further, a multiscale sustainable approach should be proposed to offset the production and application cost with prospective economic benefits favouring energy production and environment remediation.

7. Summary and future perspectives

Biomass, a non-exhaustive energy resource via direct combustion is often associated with increased carbon emissions and global warming.

Table 4
SWOT analysis of microwave produced biochar in agriculture.

Strength	<ul style="list-style-type: none"> ➢ Great potential for higher porosity retains water and nutrients ➢ Symbiotic biological carrier for plant growth ➢ Relatively higher carbon content functions as soil amendment ➢ Proper additives facilitate higher sorption affinity and nutrient enrichment ➢ Improves soil hydrological properties and C sequestration rate
Weakness	<ul style="list-style-type: none"> ➢ Selection of proper additives or catalyst ➢ Scaling up of prototype with technical idea of superior biochar production ➢ Minimization of overall expenses to be lower than product market price ➢ Economic feasibility for biochar as soil conditioner
Opportunity	<ul style="list-style-type: none"> ➢ Sustainable biochar amendment for biomass growth to biofuel production ➢ Reclaim and remediate contaminated sites ➢ Multifunctional superior biochar production offsetting application and irrigation cost ➢ Tailoring biochar quality improves the soil physicochemical properties ➢ Feasible extrapolation of contaminant removal result from aqueous media to soil
Threats	<ul style="list-style-type: none"> ➢ Selection of contaminated feedstock risks safe practices and operational boundaries ➢ Interruption of indigenous microbial diversity

Pyrolytic conversion of biomass into high value-added and energy-based by-products under the influence of microwave radiation makes the process sustainable and reduces its negative impact on environment. Microwave pyrolysis is comprehended to be one of the potential energy-efficient thermochemical conversion of lignocellulosic biomass in terms of product selectivity, quality and yield. The process efficiency in terms of temperature rising characteristics, product composition and distribution is enhanced with less energy input and time under microwave heating than conventional. Besides, use of catalyst in microwave pyrolysis greatly affects the pyrolytic products and improves its end value by the effect of its influential parameters. Microwave pyrolysis technique has been found to produce high-grade bio-oil and gaseous products that act as renewable source of bioenergy. The microwave produced/activated biochar is a suitable amendment for improving soil fertility because of its surface characteristics, which facilitate enhanced water and nutrition retention and immobilization of heavy metals. The efficient utilization of microwave pyrolysis products for agronomic utility, production of energy, fine chemicals and environmental remediation facilitates renewable energy credit. Available literature evidences undoubtedly revealed its potential in the arena of energy and environment, but the natural and structural variation of properties often makes the expected impacts nebulous.

To bridge the existing gap associated with the optimized production of biochar from agro-industrial wastes and to aid the development of bio-based economy, the following perspectives are proposed to guide the future research directions:

- More studies should be emphasized on microwave pyrolysis of different biomass to explore the inherent biochemical variations exclusively for upgrading the bio-oil, and for superior biochar production to eliminate the different constraints associated with applications in soil fertility amendments
- Proper understanding of influential parameters under microwave heating with absorber materials for constructing the prototype for pilot-scale production of biochar to be employed as soil amendment agent is necessary
- To facilitate the better understanding of the potential areas of biochar application, consistent and repeatable characterization of the product quality under different operating conditions are essential
- Integrated process for higher return on investment should be formulated based on the biochar characteristics
- Engineered biochar produced through microwave pyrolysis with proper additives could be used as soil conditioner and the residual biochar could be utilized as catalyst in a biorefinery for the production of chemicals, pharmaceutical compounds, etc., improvising the process sustainability and economics
- Studies focussed on life cycle assessment along with monetary evaluation of energy and environmental impacts of microwave pyrolysis process are necessary to provide suitable indicators for promoting biochar based bioeconomy

Nevertheless, the analysis of the bibliometric trends along with the process system engineering can be effectively employed to map the potential loopholes in microwave pyrolysis process, to synchronize the biochar production and promotes its large-scale application to achieve a sustainable ecosystem.

Credit author statement

Mari Selvam S: Conception and design, Writing – original draft preparation, Final approval of the article Balasubramanian P: Conceptualization, Supervision, Writing- Reviewing and Editing, Final approval of the article.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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