



## Innovative bio-pyrolytic method for efficient biochar production from maize and pigeonpea stalks and their characterization

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### ABSTRACT

Agricultural residues in excess of livestock fodder are garnering global attention and stern concerns owing to their accountable share in environmental hazards due to the lack of effective disposal mechanisms and indiscriminate burning. Recycling these residues for biochar production using pyrolysis is a cost-effective and locally feasible technique which offers a twin-prong solution addressing both climate and soil health issues. This research work compares a portable kiln prototype that is affordable and easy to use, with a muffle furnace at three distinct pyrolytic temperatures (400 °C, 500 °C, and 600 °C) to produce biochar from the stalks of maize and pigeonpea. The biochar properties were characterized using Electron Microscopy-Electron Dispersive X-ray (SEM-EDX), X-ray Diffraction (XRD), Fourier Transmission Infrared Spectroscopy (FTIR), and Thermogravimetric Analysis (TGA). The findings indicate significant variations in biochar properties based on raw material source, pyrolytic method, and varied temperatures. Higher pyrolysis temperatures were found to reduce the amorphous organic phase and alter the ultrastructure of biochar, as evidenced by XRD analysis. SEM imaging showed macropores in oval and round shapes with crystalline deposits. The carbon content, as per EDX, decreased with increasing temperature, aligning with changes in functional groups. Edinburgh's stability test revealed that kiln biochar has more stable carbon content compared to biochar produced using muffle furnace and the stable carbon increased with the rise in temperature. A comparative analysis demonstrated that biochar quality at 400–500 °C in a muffle furnace was on par with that produced in the portable kiln at 400 °C. Therefore, considering the kiln's portability, efficiency, cost-effectiveness, and scalability, it is a promising decentralized method for biochar production, offering a cutting-edge solution for agricultural waste management and soil carbon enhancement.

### 1. Introduction

Soil organic carbon (SOC) is the important factor and cornerstone, governing soil health comprising chemical, physical, and biological properties of soil (Soderstrom et al., 2014). The unsustainable practice of crop residue burning and on-farm agricultural waste mismanagement, on one hand, leads to the loss of soil organic carbon and on the other hand contributes to climate change (IPCC Climate Change, 2002; Raza

et al., 2022; Pravalie et al., 2021). It was estimated that globally SOC depletion and land degradation are leading to an economic loss of US\$ 6.3 to US\$ 10.6 annually (UNCCD, 2017). Therefore, it is crucial to identify sustainable solutions that address the disposal of agricultural residues and the enhancement of soil quality for farming. Amongst the hardy crop residues, pigeonpea makes up around 15% of India's overall crop of pulses, and the country is responsible for 70% of the world's pigeonpea production. The production of pigeonpea in India is around

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28.95 million tonnes/year (FAO, 2018) and the estimated woody pigeonpea stalk is equivalent to 10.33 million tonnes/year (Cardoen et al., 2015). India produced about 33.62 million tonnes in an area of 10.04 million hectares in 2021–22 (Acharya N.G. Ranga Agricultural University, 2022). In India's rural parts, these wastes are mainly used for cooking purposes and for making carbon for gunpowder, in a very incompetent and jumbled manner (Cardoen et al., 2015). However, these crop residues hold immense potential to build SOC through recycling.

Conservation agricultural practices like mulching with crop residue, incorporation of organic waste into soil, and compost application are known to improve SOC and build the resilience of soil through the improvement of physical, chemical, and biological properties. In recent times, biochar has emerged as a crucial, strategic, scalable, and economical solution for addressing global challenges like land degradation, climate change, and greenhouse gas emissions. Various thermochemical methods like, pyrolysis, hydrothermal, gasification, and liquefaction are often employed to transform biomass into fuel, with biochar being a common byproduct in these techniques (Ozcimen & Meriçboyu, 2010; Kim et al., 2012).

As a soil amendment, biochar can be used to improve soil quality and promote structural aggregation, microbial activity, increased water holding capacity, and increased nutrient bioavailability for plants (Das et al., 2021; Doran and Zeiss, 2000; Tan et al., 2022) and therefore, expected to reduce the utilization of chemical fertilizers (Tsolis et al., 2023). Apart from pyrolysis conditions, the biochar characteristics depend on raw material used, moisture, and nutritional content (Tag et al., 2016). The temperature of pyrolysis is a known factor that affects specific surface area, pH, and functional groups (Tomczyk et al., 2020).

Recent advances in biochar research and technology have significantly contributed to its development and focused on optimizing the conversion of agricultural residues into high-quality biochar, considering factors such as pyrolysis temperature, duration, and feedstock type (Mukherjee et al., 2013; Kumar et al., 2023; Venkatesh et al., 2013; Kumar et al., 2024). Pyrolysis, fast pyrolysis, gasification, torrefaction, and flash carbonization are known methods of biochar production (Yaashikaa et al., 2020), however, a decentralized low-cost technology is required to scale up the benefits of biochar in farmers' fields. The introduction of portable kiln prototypes represents a significant leap in biochar production technology. Advancements in portable and decentralized biochar production technologies are particularly noteworthy, offering cost-effective and scalable solutions for smallholder farmers (Venkatesh et al., 2013; Nataraja et al., 2021).

In light of these advancements, our study aims to characterize biochar derived from leguminous and non-leguminous crop residues (pigeonpea and maize stalks) using various production methods. This includes redesigning and developing a vertical portable kiln based on the principle of direct updraft. Although earlier research has emphasized on proximate and elemental analysis of biochar produced in kilns, an in-depth characterization was lacking. In the present study, the characterization of biochar using advanced techniques like SEM, EDX, XRD, and FTIR has enabled a deeper understanding of its properties besides estimating stable carbon and heavy metal contamination. The authors have attempted to contribute to the basic research on biochar, which also delves into providing practical insights for its economical and decentralized production using portable kilns for sustainable agriculture.

## 2. Materials and methods

### 2.1. Material collection and re-sizing

The maize (M) and pigeonpea stalks (PP) were collected from ICRI-SAT, Patancheru campus farm (17° 15' N latitude and 77° 35' E longitude). The collected raw materials were subjected to re-sizing using tractor-mounted chopper and shredder machine having a size control

of 5–10 cm. The chaffed material was stored under shade until pyrolysis.

### 2.2. Pyrolysis and sample preparation

Both maize and pigeonpea stalk materials were subjected to pyrolysis under varying temperatures using biochar kiln prototype and a table-top muffle furnace (Thermo Scientific, model: F30400). The size-reduced feedstocks were subjected to pyrolysis in a muffle furnace at varying temperatures of 400 °C, 500 °C, and 600 °C.

### 2.3. Redesigning biochar kiln for efficient biopyrolytic process

A cost-effective prototype of a biochar kiln was designed and manufactured at ICRI-SAT's farm and engineering section using iron barrels. The barrel was given openings from the bottom and a provision for a lid (31 × 31 cm) on top, to design a kiln for ideal pyrolysis. The length of the kiln is 88 cm, the circumference is 81 cm, and the diameter is 58 cm. The bottom surface of the kiln was given about 20 holes with a diameter of  $3.2 \pm 0.1$  cm to facilitate the incinerating process, however, the existing popular models have 40 holes of 2 cm diameter each (Venkatesh et al., 2015). The increase in hole size would allow effective pyrolysis along with an increase in incineration rate.

The pictorial representations of the pyrolysis process using a biochar kiln and muffle furnace are given in Figs. 1 and 2. The pigeonpea and maize stalk samples are abbreviated as pigeonpea (PP) and Maize (M). The pigeonpea biochar samples prepared using a kiln and muffle furnace (MF) at 400 °C, 500 °C, and 600 °C are abbreviated as PB Kiln, 400 PPB MF (pigeonpea biochar prepared in a muffle furnace at 400 °C), 500 PPB MF (pigeonpea biochar prepared in a muffle furnace at 500 °C), 600 PPB MF (pigeonpea biochar prepared in muffle furnace at 500 °C) respectively. The maize biochars prepared using kiln and muffle furnace (MF) are abbreviated as MB Kiln, 400 MB MF (maize biochar prepared in muffle furnace at 400 °C), 500 MB MF (maize biochar prepared in muffle furnace at 500 °C), 600 MB MF (maize biochar prepared in muffle furnace at 600 °C), respectively.

### 2.4. X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis was performed by directing a single beam of X-ray onto a crystal at a specific angle and the beam will be reflected on the sample being tested. The X-ray diffractometer was equipped with Ni-filtered Cu-K $\alpha$  radiation ( $k = 1.5406$  Å) at an accelerating voltage of 40 kV and an emission current of 40 mA to direct X-ray beam on the sample. Crystalline constituents of biochar samples were determined by XRD analysis using an X-ray diffractometer (Make: Bruker D8 Advance) at a diffraction angle of 10–80° for about 30 min at a speed of 2.5°/min. (Meili et al., 2019).

### 2.5. Fourier transmission infrared spectroscopy (FTIR)

The surface functionalities of biochar samples were determined using Fourier transmission infrared spectroscopy (FT-IR, Shimadzu, Japan). KBr pellet method was followed for recording fifty scans per sample per each of the spectrum with an infrared spectra range of 4000  $\text{cm}^{-1}$  to 400  $\text{cm}^{-1}$ . (Liu et al., 2015).

### 2.6. Scanning electron microscopy and electron dispersive X-ray (SEM-EDX)

Scanning electron microscopy and EDX using Quanta FEG 250 with minimum resolution range capability of 1 nm copy (SEM, specifically the Quanta FEG 250 model, Eindhoven, Netherlands) was conducted for both maize and pigeonpea biochar samples. Surface morphology and pore size of the biochar samples were studied using the mentioned instrument. The SEM micrographs were captured at 4  $\mu\text{m}$ , 10  $\mu\text{m}$ , 40  $\mu\text{m}$  and 100  $\mu\text{m}$ .

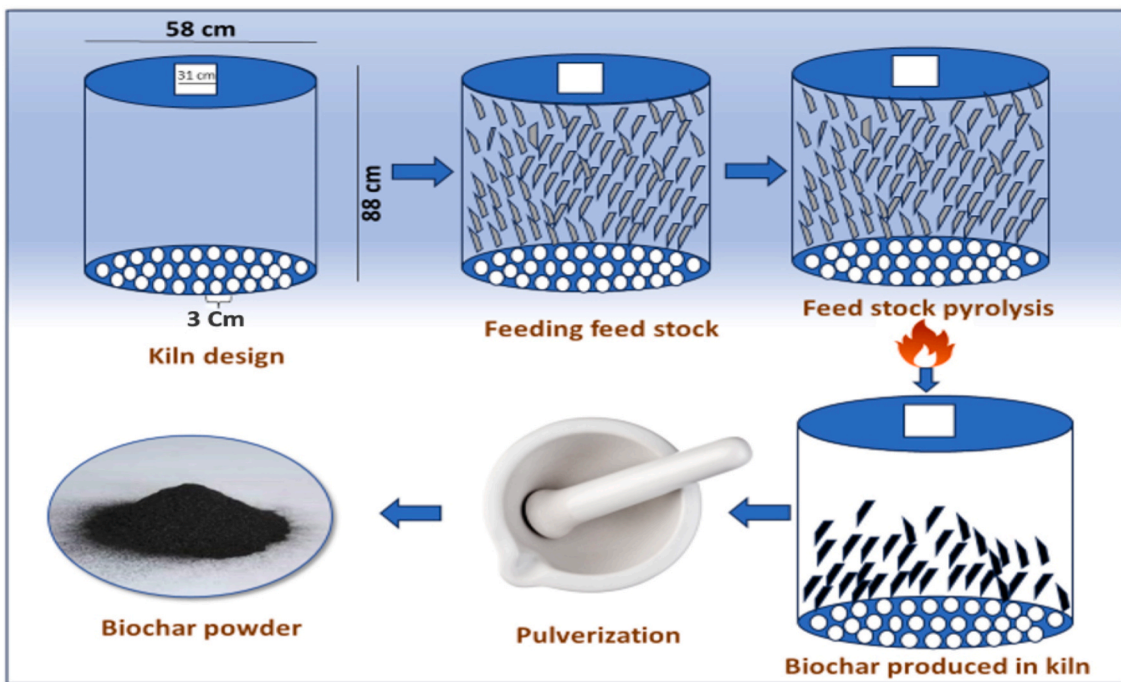


Fig. 1. Pictorial representation of pyrolysis by kiln.



Fig. 2. Pictorial representation of pyrolysis by muffle furnace.

2.7. Thermogravimetric analysis (TGA)

Thermogravimetric analysis was performed for maize and pigeonpea biochar samples using a Thermogravimetric instrument (TGA-DSC, Model: Discovery SDT 650, TA Instruments –Waters India Pvt. Ltd). TGA analysis was conducted as per the protocol followed by Kumar et al.

(2019).

2.8. Examination of biochar stability

The biochar samples were tested for stability by the chemical oxidation method (Edinburgh stability tool) as per Jindo & Sonoki

(2019). The carbon content in the dried residue was estimated following dumas dry combustion method using TCN analyser (Skalar, Primas SNC 100). The stable carbon was calculated using the following equation.

$$\text{Stable carbon (\%)} = \left\{ \frac{[\text{Carbon (g) after 5\% H}_2\text{O}_2 \text{ treatment}]}{[\text{Carbon (g) before 5\% H}_2\text{O}_2]} \right\} \times 100$$

### 2.9. Examination of heavy metals

The presence of heavy metals has been tested by using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES).

## 3. Results and discussion

### 3.1. X-ray diffraction (XRD)

The purpose of the XRD analysis was to identify the specific crystalline minerals that affect the properties of biochar for its intended applications. The existence of various inorganic components in biochar was shown by the peaks found in the XRD examination (Figs. 3 and 4).

The detection of peaks ranging from 28.3 to 28.44° was predominantly linked to crystalline configurations of calcite (CaCO<sub>3</sub>) and sylvite (KCl) (Devi and Saroha, 2013; Pariyar et al., 2020). Peaks within the 50–75° range suggested the existence of silicates and quartz associated with Mg, Ca, and Mn. XRD analysis indicated various inorganic constituents in the biochar. When comparing the biochar derived from maize stalks to those derived from pigeonpea stalks at the same pyrolysis temperatures, the intensity of the peak in the maize stalks biochar indicated the presence of higher potassium (K) and Chloride (Cl). Upon pyrolyzing at approximately 400 °C, the biochar displays structures that are similar to those of graphite. But unlike the XRD pattern seen in biochar produced in a kiln, the material loses its crystallinity when heated over 400 °C in a muffle furnace. This may be attributed to the amorphous nature with limited crystallinity, obtained due to the

elevated pyrolytic temperatures. The outcomes of the present study were in accordance with the results found by Sultana et al. (2023) who showed that non-edible rubber seed cake (RSC) is a novel bio-oil matrix (obtained through pyrolysis technique) which is amorphous comprising lower crystallinity indices in both RSC and its biochar product. As the pyrolysis temperature increased, the amorphous organic phase decreased, and ultrastructure is altered in both the types of biochar. This phenomenon aligns with the findings of Pariyar et al. (2020), who evaluated the changes in physicochemical properties, of biochars produced from various feedstocks at different pyrolysis temperatures and observed that the sharpness and intensity of the peak was noticed to increase with the pyrolysis temperature due to the decrease in the amorphous organic phase and ultrastructure changes in pine sawdust. As previously noted, the number of peak values increases as the pyrolysis temperature rises, suggesting that as the temperature rises, more elements present in the biochar sample are being detected Zhang et al. (2015). Applying a high temperature to the sample causes the cellulose and lignin to break down, exposing and enabling the equipment to identify other elements and minerals contained within the sample.

### 3.2. Fourier transmission infrared spectroscopy (FTIR)

The FTIR spectra of biochar derived from pigeonpea and maize stalks were analyzed to understand the molecular transformations taking place during pyrolysis at varying temperatures and methodologies. As presented in Figs. 4 and 5, the FTIR spectrum showcases the characteristic functional groups of the biochar, consistent with findings from prior literature (Talari et al., 2017; Ozcimen & Ersoy-Mericboyu, 2010; Sahoo et al., 2021). The distinct peaks observed in the FTIR spectra provide insights into the functional groups present in the biochar, which in turn reveal the chemical characteristics and potential applications of these biochars (see Fig. 6).

#### 3.2.1. Hydroxyl group (O–H stretching ~3300 cm<sup>-1</sup>)

Both pigeonpea and maize biochars exhibited a broad peak around 3300 cm<sup>-1</sup>, suggesting the presence of hydroxyl groups. While this was

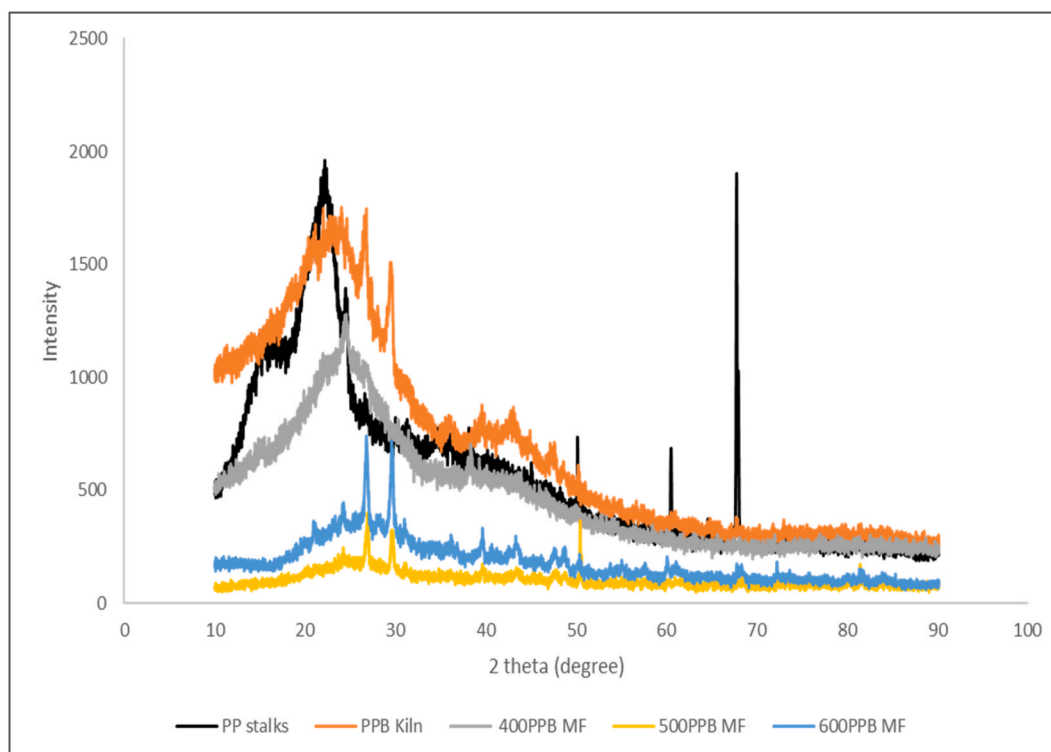


Fig. 3. XRD of biochar samples prepared from pigeonpea stalks.

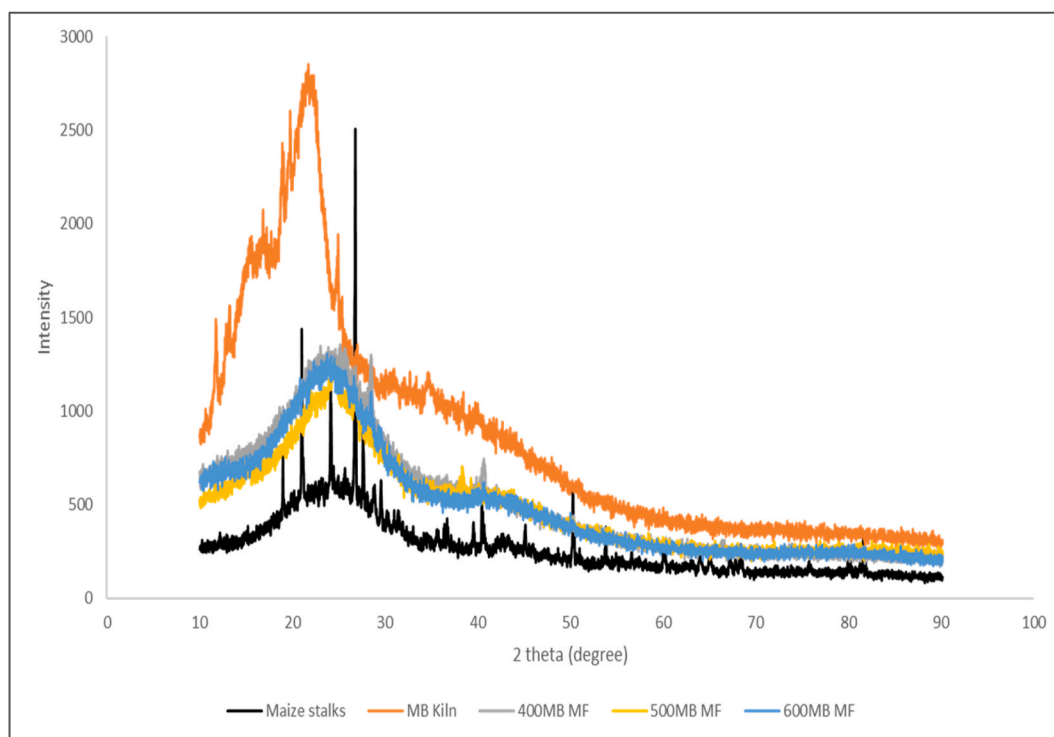


Fig. 4. XRD of biochar samples prepared from maize stalks.

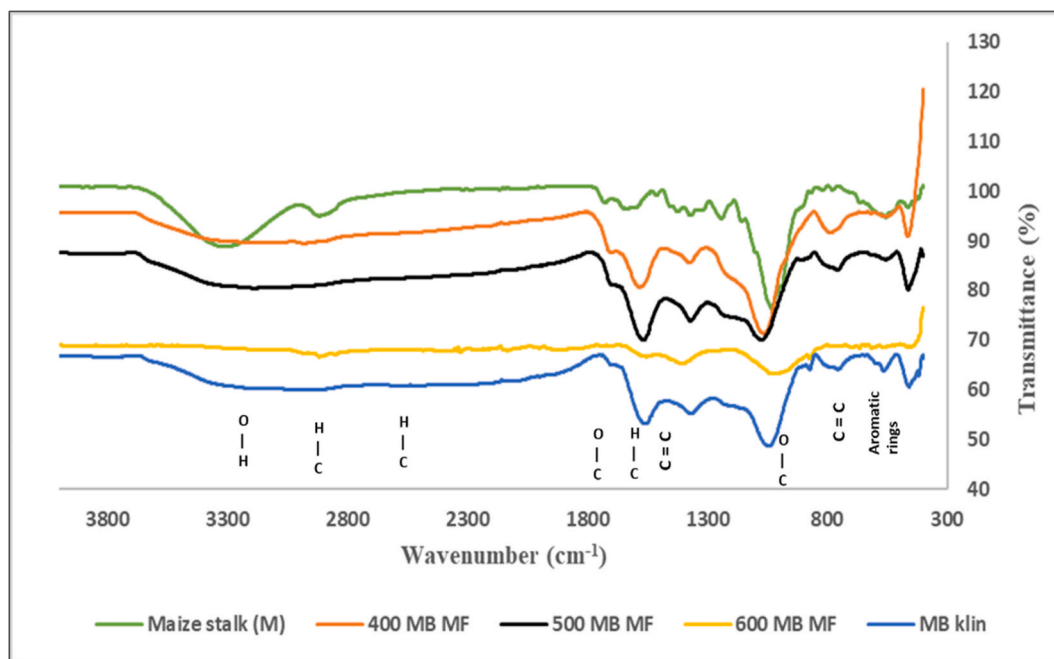


Fig. 5. Fourier-transform infrared spectra (FT-IR) of Maize derived biochar samples at different pyrolysis temperatures (400 °C, 500 °C, and 600 °C).

pronounced in the raw pigeonpea and maize stalk samples, there was a clear decline in this peak's intensity with increasing pyrolysis temperature, indicating dehydration or moisture loss. Similar results were also reported by Sahoo et al. (2021), that the pyrolysis temperature had a remarkable impact on the functional groups of biochar of pigeonpea stalk, bamboo biomass and derived biochars. The decline in hydroxyl group intensity with increased pyrolysis temperature could be attributed to the thermal degradation of cellulose, hemicellulose, and lignin components present in the stalks. As temperature rises, water content is

released due to the cleavage of these hydroxyl bonds. The results align with the pyrolysis research on bamboo by Zhang et al. (2017), Hadey et al. (2022) and Qin et al. (2020). The results obtained in the present study were similar to those reported by Sahoo et al., in (2023) regarding slow pyrolysis. They discovered that the peaks indicating O-H functional groups, such as alcoholic and phenolic groups, as well as water, at 3399 cm<sup>-1</sup> for pigeonpea stalk and 3410 cm<sup>-1</sup> for bamboo, decreased with an increase in the pyrolysis temperature.

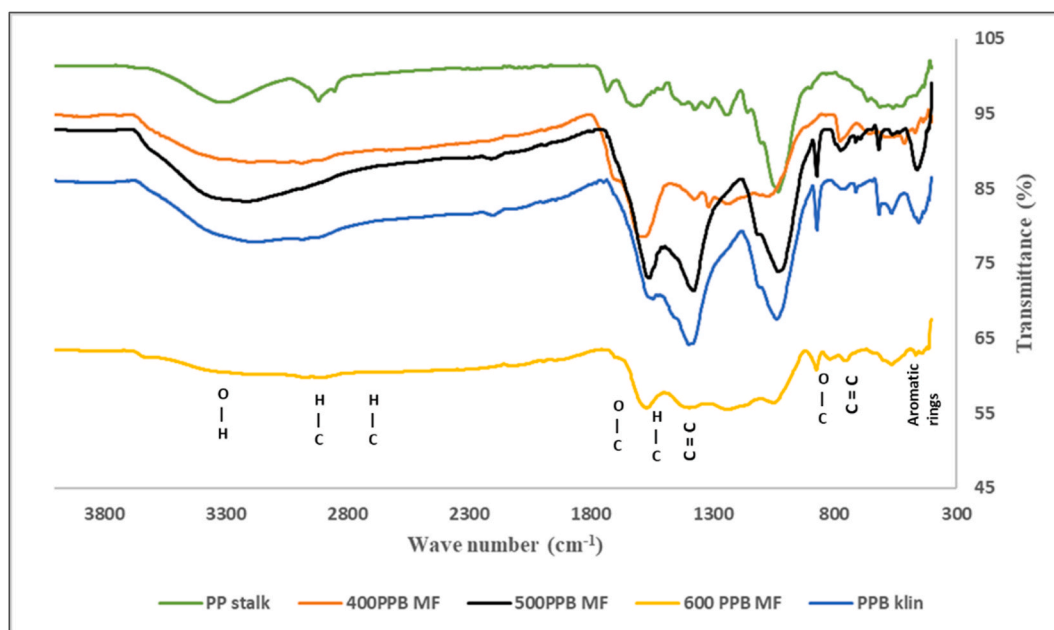


Fig. 6. Fourier-transform infrared spectra (FT-IR) of pigeonpea derived biochar samples at different pyrolysis temperatures (400 °C, 500 °C, and 600 °C).

### 3.2.2. Aliphatic hydrocarbons (C–H stretching $\sim 2800\text{--}3000\text{ cm}^{-1}$ )

Characteristic peaks for C–H stretching, indicating the presence of aliphatic hydrocarbons, were visible in biochars derived from maize and pigeonpea stalks. However, these peaks were more pronounced in the maize stalk biochar, suggesting a richer aliphatic content than the pigeonpea stalk biochar. Maize may have a higher lipid content than pigeonpea, due to its biochemical composition. Plant lipids are mostly aliphatic, which could explain the prominent peaks observed in maize biochar. The findings of the present study align with the investigation conducted by Zhang et al. (2020) on the production of liquid oils from plastic waste using heat carrier in a rotary kiln. They found that the presence of C–H stretch of  $\text{CH}_2$  or  $\text{CH}_3$  in the liquid oils corresponded to the absorbance peak in the range of  $3000\text{--}2755\text{ cm}^{-1}$ .

### 3.2.3. Carbonyl groups (C=O stretching $\sim 1800\text{ cm}^{-1}$ )

Maize and pigeonpea stalk biochars displayed peaks around this region, implying the presence of carbonyl groups or carboxylic acids. The intensity and sharpness of these peaks were more pronounced in the pigeonpea stalk biochar, indicating its richer carbonyl content. The carbonyl groups' prominence in the pigeonpea biochar might suggest the degradation of cellulose and hemicellulose into simpler fragments, leading to the formation of volatile compounds and functional groups like carbonyl. Similar results were also reported by Sahoo et al. (2021) and Hadey et al. (2022).

### 3.2.4. Aromatic rings (C=C stretching $\sim 1300\text{--}800\text{ cm}^{-1}$ )

The spectra revealed several peaks between  $1300$  and  $800\text{ cm}^{-1}$ , suggesting the presence of aromatic rings. Biochars derived from maize and pigeonpea stalks exhibited increased aromaticity with rising temperatures, but the maize biochar displayed a more pronounced aromatic nature at higher temperatures, especially at  $600\text{ }^\circ\text{C}$ . A similar phenomenon was observed by Yang et al. (2007), Hadey et al. (2022), and Popescu et al. (2018). The increase in aromaticity with temperature can be attributed to the gradual condensation and polymerization of smaller volatile fragments, leading to the formation of larger, more stable aromatic structures. Furthermore, the more pronounced aromatic nature of maize biochar might be related to the feedstock's original lignin content, which tends to produce more aromatic residues upon pyrolysis (Ozciimen & Ersoy-Mericboyu, 2010; Popescu et al., 2018; Reza et al., 2019). The findings are consistent with the results of a study conducted by Liu

et al. (2015) which compared the production of biochar from various agricultural by-products using FTIR spectroscopy. The study found that at higher temperatures ranging from  $650$  to  $800\text{ }^\circ\text{C}$ , the spectra showed a gradual loss of aromatic groups until the dominance of graphitic carbon.

It was observed that the transmittance percentages were relatively elevated for biochar produced at  $400\text{ }^\circ\text{C}$ , but these percentages reduced with an increase in temperature, particularly when reaching  $600\text{ }^\circ\text{C}$ . This was in conformity with the findings of Uchimiya et al. (2011) and Ahmad et al. (2014). The kiln method, when compared to the muffle furnace method, demonstrated similar degrees of carbonization at comparable temperatures. This was evident from the reduced hydroxyl peaks and increased aromatic ring peaks, suggesting that the kiln method could achieve similar or even better carbonization levels at potentially reduced energy expenditures (Traore et al., 2015; Popescu et al., 2018).

### 3.3. Surface morphology by scanning electron microscopy

The different pyrolysis methods and temperatures resulted in varying surface morphology and pore sizes of the pigeonpea and maize biochar samples due to varied cellulose, hemicellulose, and lignin content (Tanquilut et al., 2019; Wozniak et al., 2021). As per international union of pore and applied chemistry, the biochars obtained using kiln and muffle furnace from pigeonpea and maize stalks (Figs. 7 and 8) are under the category of macropores as the pore size is more than  $50\text{ nm}$  (Chen et al., 2018). It was observed that the maize stalks have comparatively more pores than pigeonpea stalks. The pore shape of raw material was observed to be oval, and the biochars have round to oval-shaped pores. The pigeonpea stalks are filamentous in shape under SEM view, the fibers are observed as bundles of fibrils bound together by hemicellulose and lignin and when subjected to pyrolysis, their surface morphology changed. The maize stalks were sheet like structures rather than filamentous like pigeonpea stalks. The morphology of the maize biochar prepared in kiln (MB kiln) is like a mycelial network. MB kiln and 400 MB MF has tubular pores, 500 MB MF and 600 MB MF have smooth surfaces and pores were observed on the surface. It was observed that the macropore number increased with an increase in pyrolysis temperature, which is an indication of increased surface area. These findings align with the previous studies (Lua et al., 2006) pertaining to the influence of pyrolysis conditions on the development of pores in activated

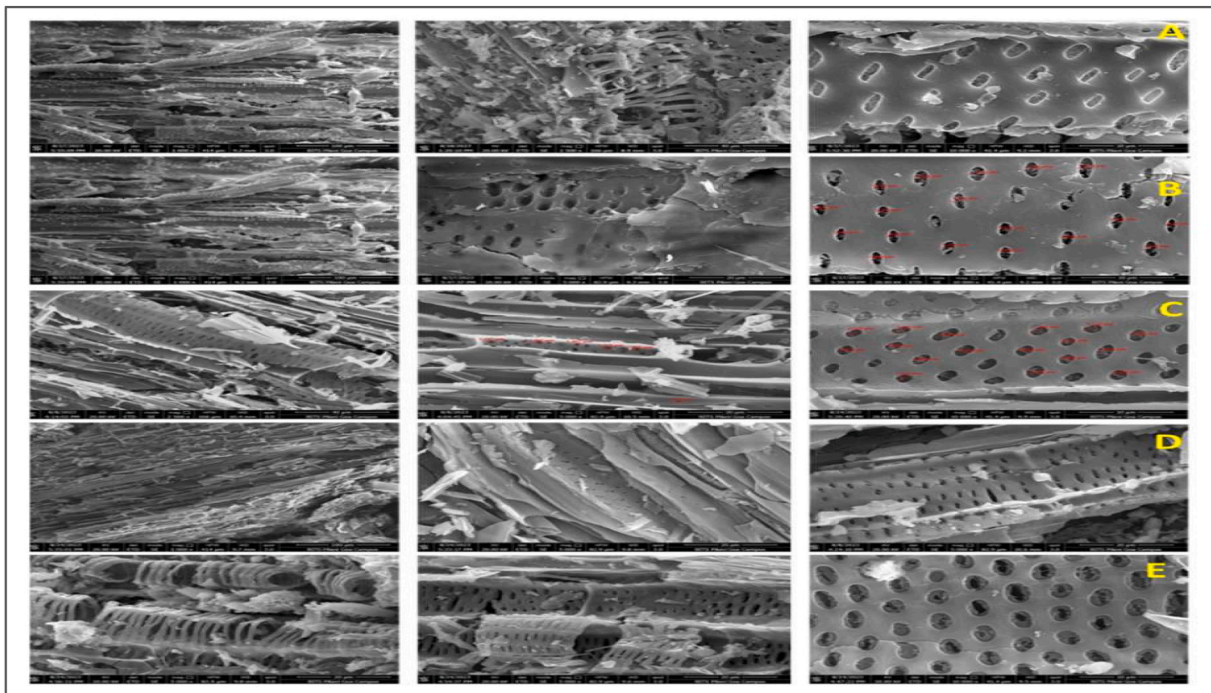


Fig. 7. Scanning electron microscopy of A) Pigeonpea stalks, B) PPB Kiln C) 400 PPB MF, D) 500 PPB MF, E) 600 PPB MF.

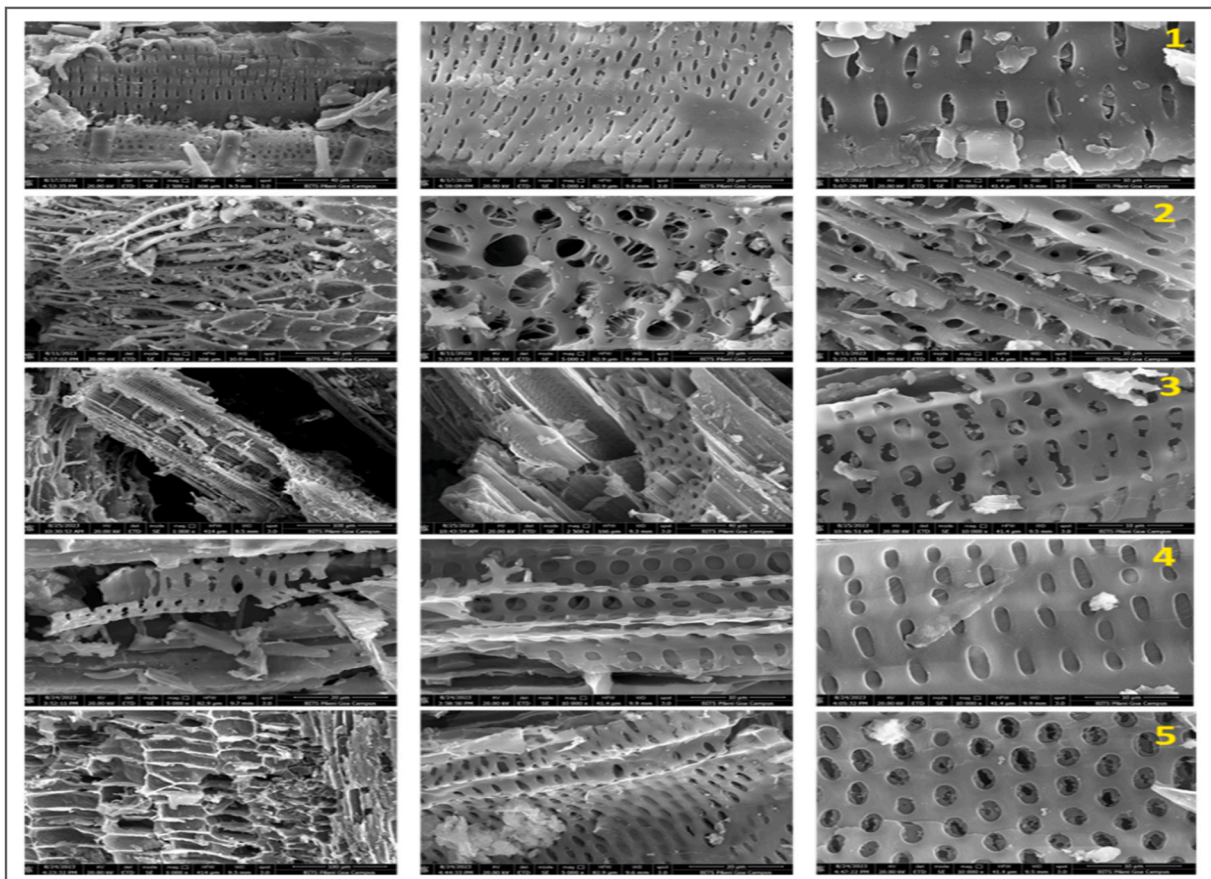


Fig. 8. Scanning electron microscopy of 1) Maize stalks, 2) MB Kiln 3) 400 MB MF, 4) 500 MB MF, 5) 600 MB MF..

carbons made from oil palm shells revealed an increase in BET surface area, micropore surface area, and micropore volume at a pyrolysis temperature of 600 °C compared to 400 and 500 °C. The micropore size of biochar has a negative correlation with increasing temperature (Ma et al., 2016). However, such a phenomenon was not observed in this study. The macropores of pigeonpea biochar are more uniform and regular compared to maize biochar when pyrolyzed in the kiln. It was observed that crystalline deposits were present inside and surroundings of the pores of biochar samples derived from both pigeonpea and maize stalks.

### 3.4. Electron dispersive X-ray (EDX)

Electron dispersive X-ray analysis of the biochar samples was conducted to estimate the elemental weight and atomic percentages. The EDX analysis results are given in Table 1. Carbon (C), Oxygen (O), Silicate (Si), Potassium (K), and Calcium (Ca) are considered important minerals to be present in biochar for its application as soil amendment (Zaitun et al., 2022). The EDX results (Fig. 9) indicated that carbon was the dominant element, followed by oxygen (O) and potassium (K) except for the biochar produced at 600 °C in the muffle furnace, which has comparatively low carbon content (Table 1). The results are in line with a recent study conducted by Mujtaba et al. (2021) on the physiochemical properties of biochar (BC), compost (C), and co-composted biochar (BCC) derived from green waste and found that all organic materials had high levels of C, O, K, Al, Si, and Ca nutrients, while BC, C, and BCC had lower levels of P, Mg, S, and Fe. A steep drop in the carbon content is witnessed at 600 °C, irrespective of the methods and materials used for biochar production. Nigam et al. (2017) found similar results while studying the pyrolysis of *Mentha arvensis*. The SEM-EDX analysis of *Mentha arvensis* biochar samples highlighted a predominant presence of carbon, followed by oxygen. Pigeonpea biochar showed higher carbon weight and atomic percentage than maize biochar, which might be attributed to the high lignin content present in pigeonpea stalks. Similar observations were reported by Gaskin et al. (2008) and Srinivasan et al. (2015).

It has been reported that higher lignin concentrations in plant biomass increase carbonization, resulting in elevated carbon and ash levels in biochar (Sohi et al., 2010; Wang et al., 2015). With rising pyrolysis temperatures, there was a decline in the weight and atomic percentages of minerals such as C, O, and K. Similar results were observed during research on three *Gramineae* plants, where it was depicted that an increase in pyrolysis temperature led to a reduction in the mineral concentration in biochar samples (Liu et al., 2016). It was found that the biochar derived from kiln and muffle furnace have similar mineral content. The traces of calcium and magnesium were found in biochar produced using muffle furnace at 500 °C and 600 °C, however, these elements were under below detection limits in biochar samples produced at 400 °C using kiln and muffle furnace.

**Table 1**  
EDX analysis of biochar.

Biochar sample	Carbon (C)		Oxygen		Potassium (K)		Magnesium (Mg)		Silicate (Si)		Calcium (Ca)	
	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
Pigeonpea stalks	48.9	56.6	49.3	42.8	1.7	0.6						
PPB Kiln	63.7	74.3	23.1	20.2	4.4	1.6	–	–	5.0	2.5	3.9	1.3
400PPB MF	65.3	71.8	33.6	27.8	1.2	0.4	–	–	–	–	–	–
500PPB MF	57.3	67.0	34.0	29.9	8.8	3.1	–	–	–	–	–	–
600PPB MF	5.0	11.9	18.9	33.0	38.7	28.1	–	–	–	–	37.4	26.5
Maize stalks	41.9	50.3	51.8	46.7	1.5	0.6	–	–	4.8	2.4	–	–
MB Kiln	60.1	66.9	24.0	20.0	2.7	0.9	1.0	0.6	–	–	–	–
400 MB MF	57.9	67.9	32.3	28.5	9.2	3.3	0.6	0.3	–	–	–	–
500 MB MF	44.3	58.6	31.0	30.8	17.5	7.1	2.1	1.2	2.1	1.2	5.1	2.3
600 MB MF	14.3	27.5	25.7	37.1	30.8	18.2	–	–	–	–	28.4	16.4

### 3.5. Thermogravimetric analysis (TGA)

Maize and pigeonpea stalks, and their biochars derived using a portable kiln and muffle furnace were subjected to TGA analysis, which revealed that raw choppings of maize and pigeonpea stalks were found to be less stable with increasing temperature compared to their biochar samples. This phenomenon is justified as the raw materials did not undergo the pyrolysis process and when subjected to higher temperatures more weight loss is observed owing to the charring effect and loss of moisture. The high thermal stability of biochar is due to its high stable carbon content and low volatile matter (Minugu et al., 2021).

It was observed that the loss of weight in maize stalks is 4.024 mg (78.66 % of weight loss) and pigeonpea stalks is 4.132 mg (76.83 % of weight loss), when subjected to increased temperature up to 1200 °C. Biochar derived at 600 °C has less weight loss as compared to the biochar derived at 400 °C and 500 °C. The thermal degradation temperature range was found to be different for the stalks and biochar derived at different temperatures. TGA of maize stalks and the biochar samples revealed that the thermal degradation temperature ranges of maize stalks and maize biochar derived at 400 °C, 500 °C and 600 °C were 188.51–348.65 °C, 351.18 °C–639.26 °C, 364.30–676.05 °C and 374.80 °C–701.44 °C respectively (Fig. 10.). The thermal degradation temperature ranges of pigeonpea stalks, and pigeonpea biochar derived at 400 °C, 500 °C, and 600 °C were 247.43 °C–341.52 °C, 387.83 °C–688.85 °C, 372.14 °C–669.42 °C and 364.64 °C–672.25 °C respectively (Fig. 11). Similar results were observed by Das et al. (2023) while characterizing the biochar samples produced from maize stalks. The mass loss (wt.%) was observed to be higher for biochar produced at low temperature (400 °C) compared to high temperature, whereas the thermal stability of biochar produced at 600 °C was higher compared to 400 °C. The findings of the study are in conformity with Qurat-ul-Ain et al. (2021). The enhanced thermal resilience of biochar sourced from pigeonpea stalks, compared to that from maize biomass, can be attributed to the high lignin content in pigeonpea stalks relative to maize stalks which is in conformity with the findings of Kumar et al. (2019). The authors observed that within a temperature range of 200 °C–480 °C, the main components of lignocellulosic biomasses get thermochemically degraded. Maximum cellulose and hemicellulose breakdown occurs between 200 °C and 350 °C, while a portion of cellulose continues to break down up to 500 °C. Beyond 500 °C, lignin degradation continues up to 1000 °C.

### 3.6. Carbon stability

The results indicate a clear variance in stable carbon percentage across biochar produced using kiln and muffle furnace at different temperatures, notably all the samples have more than 77% of stable carbon. As stable carbon compounds are less likely to be released into the atmosphere as greenhouse gases (Ren et al., 2018), the biochar from both pigeonpea and maize stalks could be of great significance,



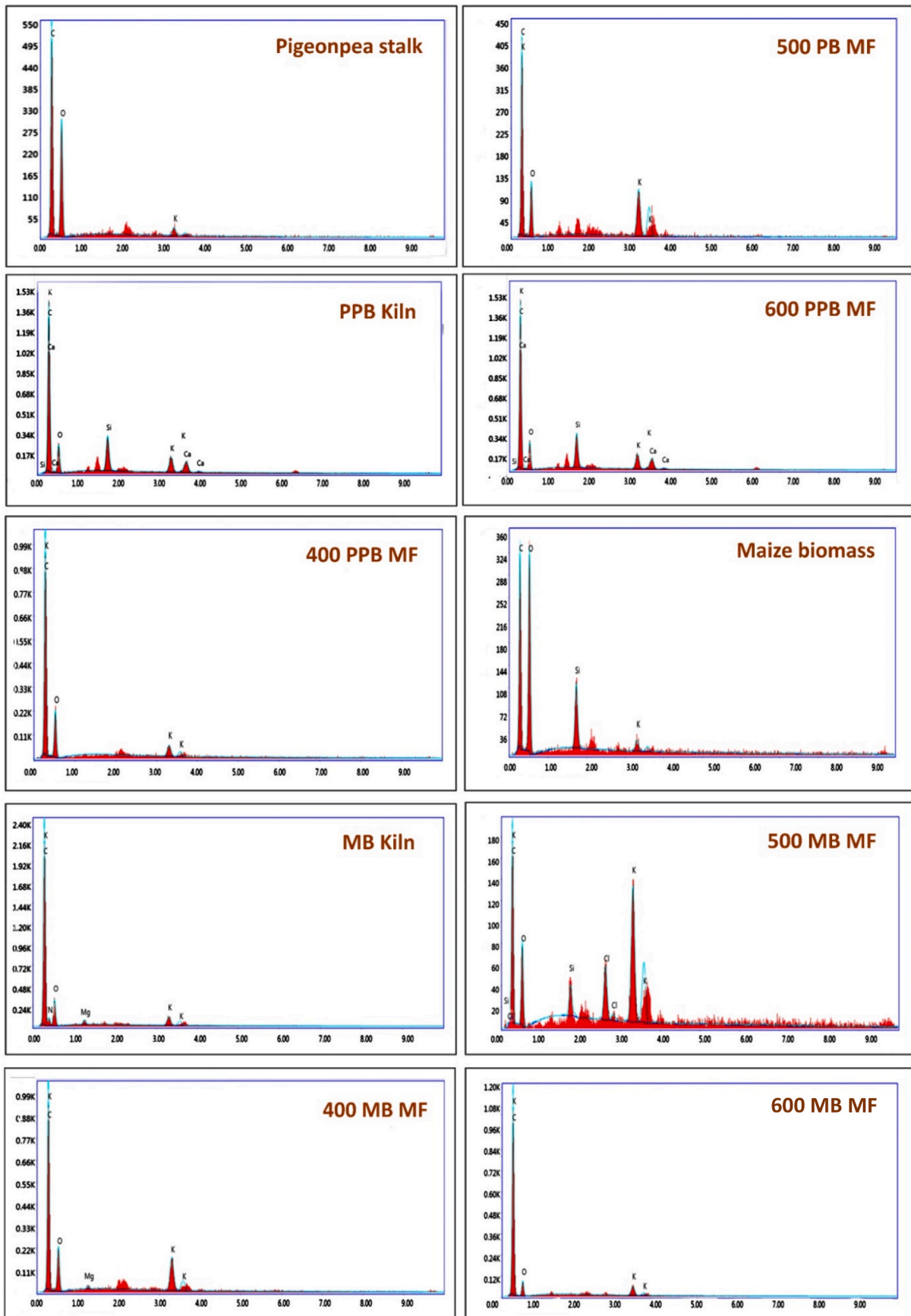


Fig. 9. Electron dispersive X-ray (EDX) analysis of pigeonpea, maize stalks and biochar samples.

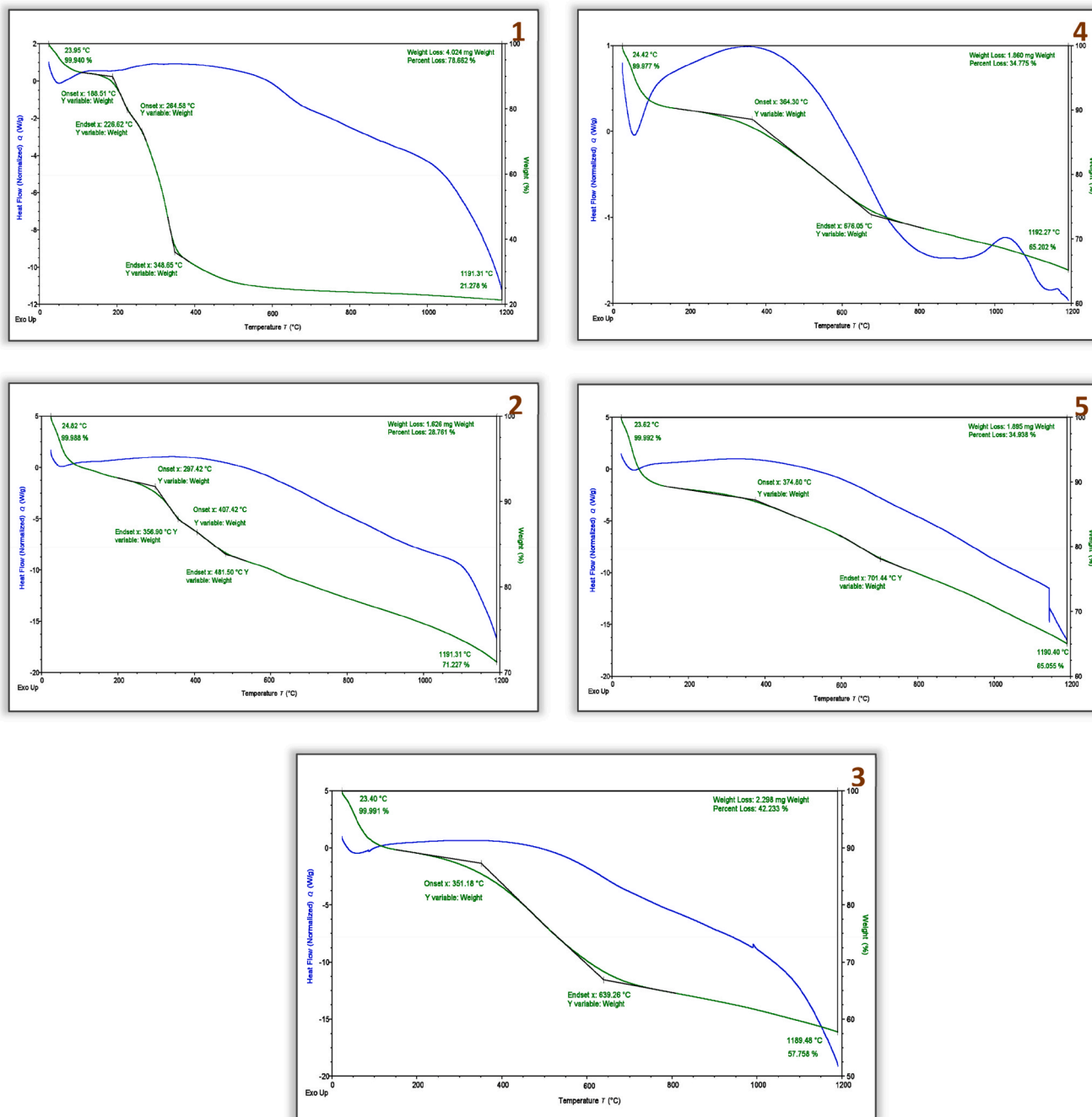


Fig. 10. TGA of biochar samples produced from Maize, 1. Maize: Maize stalks. 2. MB Kiln: Biochar prepared in Kiln, 3. 400 MB MF: @400 °C in MF, 4. 500 MB MF: @ 500 °C in MF, 5. 600 MB MF: @ 600 °C in MF.

particularly in the context of climate change. The stable carbon content increased as the temperature rises from 400 to 600 °C while producing biochar from pigeonpea stalks in muffle furnace (Fig. 12). The kiln method at 400 °C proved to be effective for enhancing stable carbon content in pigeonpea biochar (98.68%) and maize biochar (94.49%) compared to muffle furnace. The design and operation of kilns can be more thermally efficient with uniform heat distribution, retaining better heat and requiring less energy to maintain high temperatures over time, which can lead to an efficient pyrolysis and greater stable carbon content (Sakthivel et al., 2023).

### 3.7. Heavy metal contamination

All biochar samples observed to have heavy metal concentrations below International critical limits (International Biochar Initiative., 2023). Therefore, the permissible levels of potentially toxic elements (Table 2) suggest that the biochar produced using pigeonpea and maize stalks could be used as soil amendments.

### 3.8. Economics of biochar production

The production economics of biochar from kiln is given in Table 3. The cost of kiln is about 1500 INR which is inclusive of the drum, perforations, and handle fitting. With the conversion efficiency of 28 % and

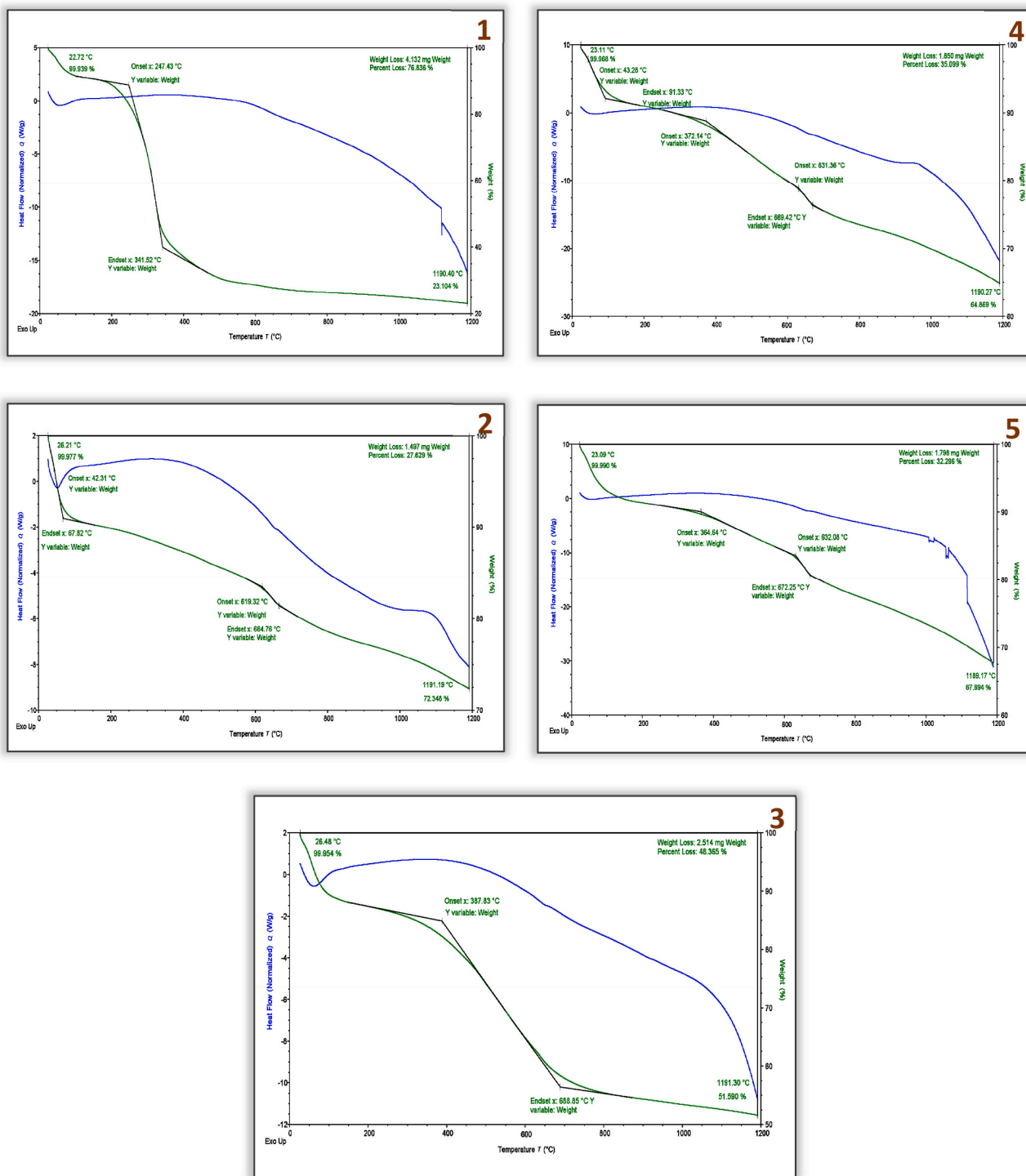


Fig. 11. TGA of biochar samples produced from Pigeonpea, 1. Pigeonpea: Pigeonpea stalks. 2. PPB Kiln: Biochar prepared in Kiln, 3. 400 PPB MF: @400 °C in MF, 4. 500 PPB MF: @ 500 °C in MF, 5. 600 PPB MF: @ 600 °C in MF.

23 %, a 100 kg biomass generates 28 and 23 kgs of biochar from pigeonpea and maize stalks, respectively. The operational costs towards the production of pigeonpea and maize biochar are 750 and 800 INR with B:C ratios of 1.60 and 1.31, respectively. The results revealed that the production of biochar from pigeonpea stalks is more economical than from maize stalks due to the high conversion ratio of pigeonpea stalks. Similarly, the low capital and production cost of the portable kiln with high monetary returns proves this technology as a scalable and viable option at the farmers' fields.

#### 4. Conclusion

The present study stands unique as it deals with redesigning scalable low-cost kiln prototype for biochar production from different sources and its comprehensive qualitative characterization in comparison with muffle furnace at varying temperatures. Biochar produced at varying pyrolysis temperatures showed a substantial disparity in the physico-chemical characteristics of biochar, which might be attributed to differences in the lignin, cellulose, and hemicellulose content of the raw

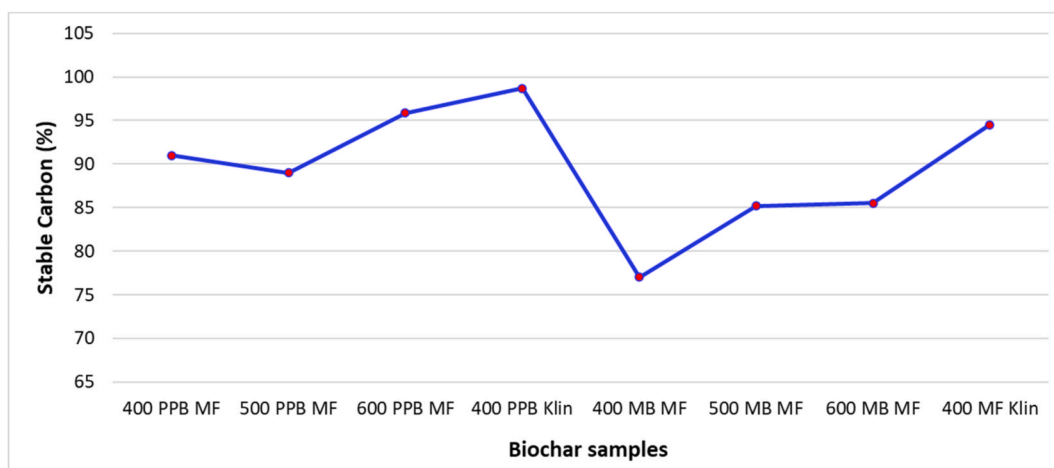


Fig. 12. Influence of pyrolysis temperature on the stable carbon content of Pigeonpea and Maize biochar samples.

**Table 2**  
Total concentrations of heavy metals (ppm) in Biochar samples.

Biochar	Total-Cu	Total-As	Total-Cd	Total-Cr	Total-Pb	Total-Ni
Critical limits as per International Biochar Initiative (2023)	6000	100	39	1200	300	420
400 PPB Klin	46.00	2.98	0	3.65	34.23	18.63
400 PPB MF	14.45	4.23	0	3.90	0	25.60
500 PPB MF	34.45	4.93	0	5.63	5.33	41.40
600 PPB MF	46.75	5.60	0	7.83	9.93	56.20
400 MB Klin	21.20	0	0	12.88	25.03	38.38
400 MB MF	1.83	0.40	0	2.33	0	13.65
500 MB MF	0.65	3.53	0	2.55	0	13.08
600 MB MF	0.18	4.98	0	2.25	0	13.18

**Table 3**  
Cost of producing biochar from Pigeonpea and Maize stalk using 100 kg biomass per day.

Capital investment per kiln	INR per kiln
Cylindrical metal drum inclusive of perforations, top lid and handle	1500
<b>Operational cost of biochar production per day</b>	
Kiln operation (2 man-days)	700
Cost of pigeonpea stalks @ INR 0.5/Kg	50
Cost of maize stalks @ INR 1/Kg	100
Total operational cost of pigeonpea biochar	750
Total operational cost of maize biochar	800
<b>Production economics</b>	
Conversion efficiency of Pigeonpea (%)	28
Conversion efficiency of Maize (%)	23
Market Price of biochar (INR)	40
Gross income- from Pigeonpea (INR)	1120
Gross income- from maize (INR)	920
BCR-Pigeonpea biochar	1.60
BCR-Maize biochar	1.31

materials. As the temperature increases from 400 to 600 °C, the thermal resilience and number of pores in biochar increased substantially. However, the strength of the functional groups (C–H, C=C, C–O), carbon content, and crystallinity decreased considerably as reflected from FTIR, SEM-EDX, and XRD results, respectively. A controlled anoxic condition at 400 °C temperature produced thermally stable, macroporous biochar with functional groups (C–H, C=C, C–O) using both the bio-pyrolytic methods. Comprehensive profiling of biochar revealed that the quality of biochar produced at 400–500 °C in muffle furnace is comparable with biochar prepared in the portable kiln at 400 °C. Thus,

keeping in view the cost efficiency, operability, and scalability, the modified portable kiln is a potential decentralized bio-pyrolytic method for efficient biochar production from maize and pigeonpea stalks, which also proved to produce biochar with higher stability. The prototype kiln was redesigned and developed at a pilot scale however, authors are curious to study the economic feasibility and societal perspective at a wider scale, given the farmers’ skepticism over the investment in biochar production.

#### 4.1. Way forward

The future scope of research on biochar delves with long-term studies on soil application, influence of biochar application on microbial diversity, and plant health. The economic and environmental impact involving mitigation of carbon footprints and greenhouse gas emissions along with optimization of pyrolytic conditions and the role of catalysts should be explored. Indian governance through the multiple incentivization programs for voluntary carbon markets has initiated schemes like Mission Life-Lifestyle for the Environment, one of the key components to qualify waste management based green credits. Therefore, biochar production would play an indispensable role in waste management per se and can be considered to qualify for the ecomark label for accruing green credits in the near future.

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#### Availability of data and materials

All data generated or analyzed during this study are included in this published article and its supplementary information files.

#### CRedit authorship contribution statement

**Rajesh Pasumarthi:** Conceptualization, Formal analysis, Investigation, Methodology, Writing – original draft, Writing – review & editing. **Gajanan Sawargaonkar:** Conceptualization, Methodology, Resources, Supervision, Writing – original draft, Writing – review & editing. **Santosh Kale:** Methodology, Validation, Writing – original draft, Writing – review & editing. **Nallagatla Vinod Kumar:** Methodology. **Pushpajet L. Choudhari:** Formal analysis, Methodology, Writing – review & editing. **Ramesh Singh:** Resources, Writing – review & editing. **Moses Shyam Davala:** Data curation, Writing – review & editing. **C. Sudha Rani:** Supervision, Visualization. **Srikanth Mutnuri:** Formal analysis, Investigation, Methodology. **M.L. Jat:** Methodology, Project

administration, Supervision, Visualization, Writing – review & editing.

## 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.

## Data availability

Data will be made available on request.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jclepro.2024.141573>.

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