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ORIGINAL CONTRIBUTION

Renovation of Waste *Chrysanthemum morifolium* (Marigold) into Valuable Biochar: A Study on the Utilization of Solid Waste by Pyrolysis

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Abstract Flowers have become a part of our daily life, and wind up joining the waste stream, after little use and almost practically no reuse. Disposal of the flowers despite playing an eloquent role in religious faith, rituals, wedding ceremony, etc., finishes up mostly at open dumping or in water bodies. An endeavor has been made to valorize the flower waste generated from the flower market in Puducherry. White Marigold *Chrysanthemum morifolium* was retrieved in bulk from the market waste stream and was thermally treated into valuable low-cost biochar. The products were synthesized by pyrolysis in slow thermal carbonization conditions at 350 °C and 500 °C. Before treating thermally, the collected waste marigold flowers were analyzed for decomposition behavior via TGA. The biochar products were further characterized, analyzed, and compared, for various physiochemical parameters, namely pH, conductivity, moisture content, ash content, and bulk density. Specific physical and chemical properties and the effect of slow pyrolysis at two different temperatures were further investigated by performing FTIR, SEM, XRD, and BET. Biochar pyrolyzed at 500 °C possessed better characteristics for environmental applications than biochar derived at 350 °C.

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Keywords Marigold flower waste · Biochar · Pyrolysis · Low-cost · Characterization of biochar

Introduction

With an estimated population of about 1.38 billion, India is the second-most populous country with daily solid waste generation amounts up to 145,626 MT/Day [1]. Manifold characteristics reflected in environmental degradation due to improper disposal of solid waste are demanding specific handling and appropriate disposal. Rapid urbanization with an elevating trend of open dumping around the cities has furthermore degraded the environmental quality harshly [2]. Solid wastes characteristics are very diverse, depending mainly on the lifestyle and culture of the inhabitants [3]. Almost 80% of municipal solid waste being dumped into landfills, with a maximum load of organic matter, is generated as a result of diverse cultural, social, religious, dietary, and day-to-day life habits [4]. Toward proper management and processing of biodegradable waste biomass, many attempts have been made, which includes both biochemical and thermochemical routes [5]. The thermochemical pathways for biomass conversion are achieved mainly by combustion pyrolysis, gasification, and liquefication. The maximum temperature and pressure conditions, use of catalysts, and any pretreatment make those processes unique.

Floral refuse has also been a challenge in Indian cities, and thus needs specific attention. Flowers are mostly used in temples, churches, and dargahs as offerings, used in homes as a part of religious faith, marriages for decoration, and some are leftover after the sales in flower markets, etc. [6]. Due to lack of facilities and legislation, the apt environmentally friendly disposal of floral waste has been a major concern. With a high microbial decomposition, time compared to other organic wastes like kitchen waste, the treatment of floral wastes needs a precise and novel approach [7] (Fig. 1).

The value-added products from the waste flower have been of keen interest for researchers, with many attempts being made to extract, manufacture, and utilize the various derived products mostly organic dyes and pigments [8–10]. Also, the floral waste has been used in many other ways, which include conversion to biofertilizers via composting and vermicomposting [11–13], bioethanol [14], biogas and methane enrichment [15, 16], essential oils [17] incense sticks, and handmade soaps other daily use products [18].

Biochar is known for its carbon content (Fig. 1); the biomass is decomposed at a high temperature by the process called pyrolysis [19, 20]. Biochar has been studied extensively by researchers [21]. Biochar has been used for so long since it mitigates climate change, sequestrating carbon, and used for sustainable waste management [22–24]. Organic waste-based biochar has been used as carbon sequestration and soil amendment [19, 25], additive to composting [26], for sorption of heavy metals and dyes [24] and thereby reducing the amount of waste to be sent to the landfill. The processing conditions such as duration, heating temperature, and the feedstock nature regulate the biochar's physicochemical properties [19, 27].

Puducherry is one of the union territories of India, politically covers around 492 km^2, and its geographical location spread in different regions, namely Puducherry, Karaikal, Mahe, and Yanam. The latitude and longitude of the Puducherry region are 11° 56′ N and 79° 53′ E, respectively, and is comprised of 55.9% of Puducherry U.T. The Puducherry region is the capital town and its urban landscape ornated by the shore of Bay of Bengal. Puducherry administration provinces consist of 5 commune panchayats and 2 municipalities (Puducherry & Olgaret). The amalgamation of Indo-French culture and several other unique features of Puducherry attracts visitors globally. Tourism-based revenue generation is a significant source of benefits to both public and private agencies, and the maintenance of the aesthetic aspect is very important. The entire solid waste generated in the region is managed by the local urban and rural administration departments. With a daily waste generation of 495 metric tons, the city is subjected to severe environmental hazards [1]. As of 31.12.2018, the authorities handled nearly around 350 MT/day of solid waste generated in 122 wards, and 10% of the total waste collected was processed [28]. The major single-point sources of municipal solid waste in Puducherry are from various markets from municipalities and commune panchayats. Puducherry has numerous markets and "Ulavar santhai" (A direct market for farmers). The waste generated from the market varies from unsold flowers, rotten broken vegetables & fruits, packing materials like cardboard, plastics bags, paper, and jute bags, fish and meat, etc. The flower waste generation significantly varies over the season, festival days, and types of flowers and demands. An attempt has been made for the thermochemical transformation of flower waste into biochar.

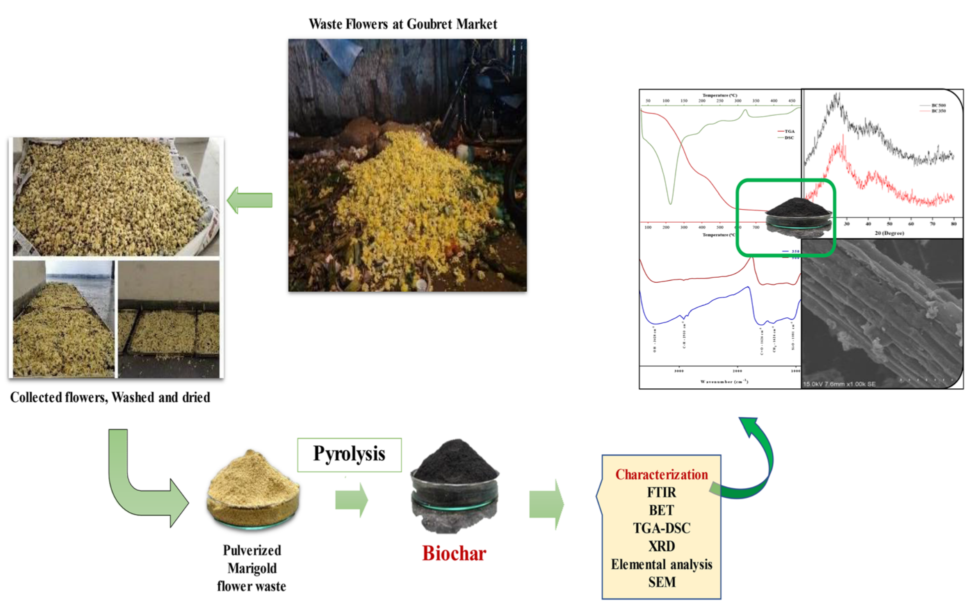

Fig. 1 Pyrolysis mechanism

Materials and Methods

Preliminary Process

The discarded flowers from the Goubert Market, Puducherry, India (Fig. 2), were collected from market premises, directly from the sellers before they discard them to the dump. The flower waste compromised 90% White Marigold *Chrysanthemum morifolium* which is locally called "Saamanthi" and was thus chosen as the raw material for making biochar. The flower waste collected from the market was first sorted from paper and plastics and other impurities and washed with distilled water. After washing, the flower waste was sun-dried for 72 h and then pulverized. The pulverized powder hereafter termed FP and the same was used as natural resources for biochar production.

Pyrolysis of floral waste

The pulverized white marigold flower powder (FP) was dehydrated by the hot air oven for six hours, and the

Fig. 2 **a** Goubert market, **b** flowers thrown as waste outside the Goubert market

temperature was maintained at 72 ± 5°C. The FP was then sieved through a 2 mm sieve, and 50 g was loaded to a ceramic container, that was heated in a muffle furnace, at two different operating temperature of 350 °C and 500 °C with a hold duration of 30 min and the rate of heating is 10 °C min^{-1}, leading to slow pyrolysis conditions [38]. The products thus obtained were further cleaned using distilled water (pH 7), dehydrated by a hot air oven at 110 ± 5 °C to a constant weight [6]. The biochar products hereafter referred to as BC350 and BC500 for biochar obtained at 300 °C and 500 °C, respectively, were stored in airtight glassware.

Moisture, Volatile, and Ash Content Determination

ASTM D1762-84 recommendation was followed for the determination of moisture, ash, and volatile content. For the gravimetric determination of moisture content, the residues of the crucible should be removed by heating the crucible for 10 min at 750 °C; then, 1 g of biochar sample was taken in the crucible and heated at 105 °C for 2 h [29].

Further calculations were made using the following equations;

$$\text{Moisture},\ \% = \frac{(A-B)}{A} \times 100$$

$$\text{Volatile matter},\ \% = \frac{(B-C)}{C} \times 100$$

$$\text{Ash},\ \% = \frac{D}{B} \times 100$$

where A air-dried sample used in grams, B sample after drying at 105 °C, in grams, C sample after drying at 950 °C, in grams, D residue in grams.

Determination of Yield and Bulk Density

Bulk density and yield of biochar were measured as per the equation below;

$$\text{Yield},\ \% = \frac{W_c}{W_o} \times 100$$

$$\text{Bulk density} = \frac{\text{Mass of dry sample (g)}}{\text{Total volume used (ml)}}$$

where W_c is the dry weight (g) of the biochar and W_o is the dry weight (g) of the dry biomass (PFP).

Characterization

For assessing the decomposition behavior of PFP, thermogravimetric analysis (TGA) was performed on a simultaneous thermal analyzer (TA- Instrument TGA, Q500) from 30 to 900 °C and in airflow 50 mL min^{-1} with a normal pressure. The particle size distribution of biochar has a significant role in biochar physicochemical properties. Biochar was sieved through a sieve set of different sizes, ranging (0.5, 0.25, 0.1, and 0.075) mm. The elemental analyzer was used to evaluate the composition of carbon, hydrogen, nitrogen, and sulfur elements (Thermo Fisher Scientific Inc., MA). Brunauer–Emmett–Teller (BET) finalized the pore dimension and surface area. In the BET method, the surface area is calculated by the adsorption rate of liquid N_2 over the surface at a minimum temperature of 77 K [30]. BET surface area analyzer from "Micromeritics Instrument Corporation made Gemini VII 2390" was used. Hitachi S-3400 N with 10 kV accelerating voltage is utilized to take samples of scanning electron micrographs. Biochars were held on sticky carbon tape over the aluminum stub followed by sputter coating with graphite before viewing. The Perkin Elmer Spectrum 1, FT-IR instrument was used for determining the FTIR spectra of the biochar sample. Pre-dried and pulverized biochar samples were dispersed in spectroscopic-grade KBr, and pellets were made. After baseline correction, signal averaging, signal enhancement, and other spectral manipulation, the spectrum was obtained over a region of 450–4000 cm^{-1}. Crystalline C and additional materials in the biochar were determined by the XRD application. The degree of crystallinity of biochar samples was analyzed by X-ray diffractions patterns XRD (PANalytical X'Pert).

Results and Discussion

Generation of Flower Waste from the Goubert Market

Goubert market is the main market of Pondicherry city, the market zone is a center of attraction for tourists, with two narrow pathways, and about 60 shops, selling flowers on both sides of the pathway. The flower market opens at around 5 am and closes late by 10 pm. The flowers from the flower cultivators come from nearby villages. Flowers are sold mainly as cut flowers or in the form of garlands. Marigold (*C. morifolium)* having the maximum sales followed by Jasmine (*Jasminum polyanthum and Jasminum auriculatum),* Rose (*Tabernaemontana divaricata, Rosa),* and others. The market has a total of 12 wholesale shopkeepers that sell flowers to most of the shops in the city. The market generates copiously segregated flower waste, at a varying rate, depending on the demand, season, and rate.

TGA-DTG Analysis

Pulverized marigold flowers (FP) were exposed to thermogravimetric analysis. The decomposition of cellulosic and hemicellulosic segments of biomass has been found to begin from 250 °C and occurred up to 350 °C and further loss in weight due to degradation of lignin components above to 500 °C [31]. Figure 3 shows the mass yield during the pyrolysis process and the data resembles typical biochar conduct. The results of TGA analysis with air are shown in Fig. 3 and the sample exhibits different characteristics, distinct weight loss curves with varying temperatures are observed; at the temperature of 150 °C, a weight loss of about 10% represented the first curve and hinted the moisture removal [32]. When the temperature is further raised from 150 to 400 °C, the curve indicates weight loss of up to 51% which can be linked to capillary water holding in the lignin matrix arrangements of the biomass [33], and then, till 410 °C almost a total 59.79% of weight loss was noted, this may have arisen due to the total decomposition of lignin structures. The curve shows a further weight loss of 30% till a temperature of 600 °C, and the curve was smooth thereafter to a maximum temperature of 950 °C. The fastest mass loss was seen near to 350 °C and in between 490 and 530 °C; hence, 350 °C and 500 °C were chosen as two pyrolytic temperatures.

Physiochemical Parameters

The results of the various physiochemical measurements are given in Table 1. The biochar yield obtained at 350 °C and 500 °C was 40% and 31%, respectively. With the increase in temperature, there has been a reported decrease in yield [34]. Both the biochars were found in basic nature. BC350 was found to have a pH of 7.8 and BC500 with a pH of 8.7. The latter was found to have a more basic pH possibly because of the high pyrolytic temperature. Increased biochar pH and ash content have also been

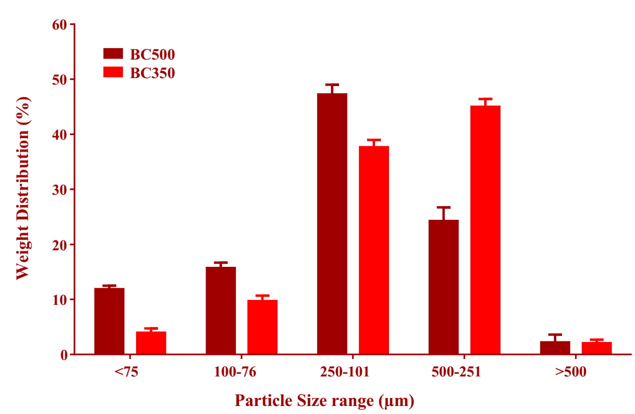

Fig. 3 Particle size distribution of biochars

Table 1 Physicochemical parameters of BC350 and BC500

Parameter	BC350	BC500
Yield (%)	37	29
pH	7.8	8.7
EC at 25 °C (S/m)	0.33	0.15
Bulk density (g/cm^3)	0.42	0.53
Moisture content (%)	6.3	8.9
Volatile matter (%)	21.9	16.7
Ash content (%)	6.7	8

Table 2 Elemental analysis of BC350 and BC500

Sample	C %	H %	N %	O %	S %	*C/H* ratio	*C/N* ratio
BC350	60.02	4.32	3.36	25.24	0.07	14.05	18.06
BC500	62.94	2.05	3.07	24.31	0.01	30.18	20.14

related to feedstock particle size [35]. The increased bulk density in BC500 can be due to the smaller particle size. The BC500 has almost 11% of 75 µm which has resulted in more bulk density than BC350. BC500 was found with higher moisture content and volatile matter. Ash content was found higher in BC350. The elemental composition of the biochar is given in Table 2. Negligible traces of sulfur were found.

Particle Size Distribution of Biochar

Figure 4 displays the particle size distribution (PSD), concerning weight fraction; the PSD of the biochar was observed mostly between 250 and 100 µm for both BC350 and BC500 and represents 50% and 39%, respectively. Also, for BC350, about 46% was in the range of 500–250 µm. Biochar having a particle size of less than 75 µm was found almost three times more in BC500 than in BC350. The significant difference in particle size of two biochar can be contributed to the reduction of size due to higher temperature for BC500. Pyrolysis temperature and particle size are strongly dependent on each other; also using slow pyrolysis has also resulted in a wide range of particle size in biochar [35].

FT-IR Results

At 350 °C and 500 °C in FTIR spectra, prepared biochar displayed numerous peaks at diverse wave numbers. In the structure of dried Marigold flower waste and biochar, stretching of the phenolic group and hydroxyl (O–H) groups as moisture was represented at peak 3400 cm^{-1} succeeded by a broad spectrum, whereas carboxylate bonds

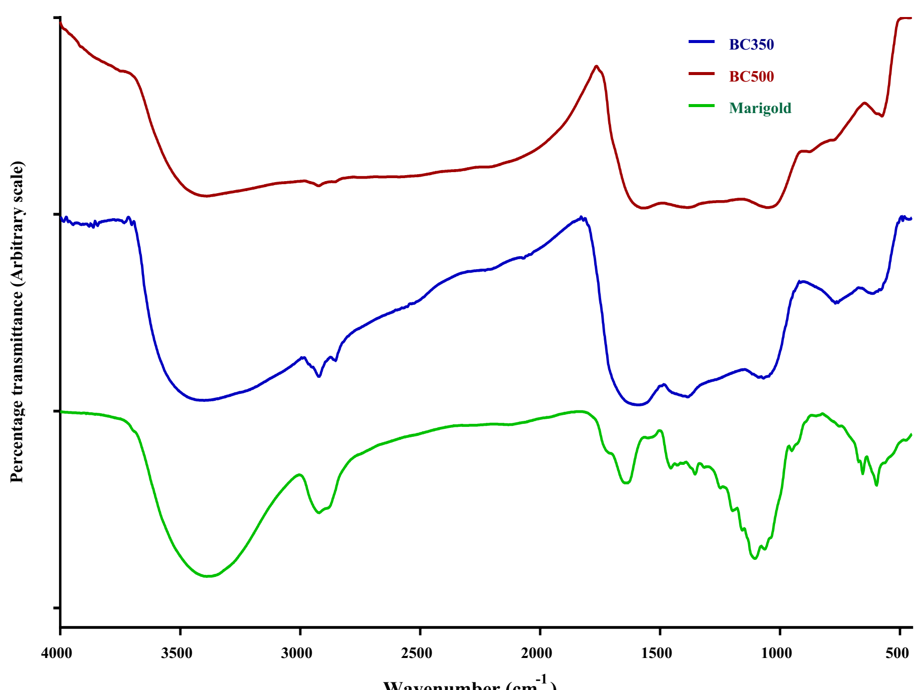

Fig. 4 FTIR Spectra of the Marigold, BC350 and BC500

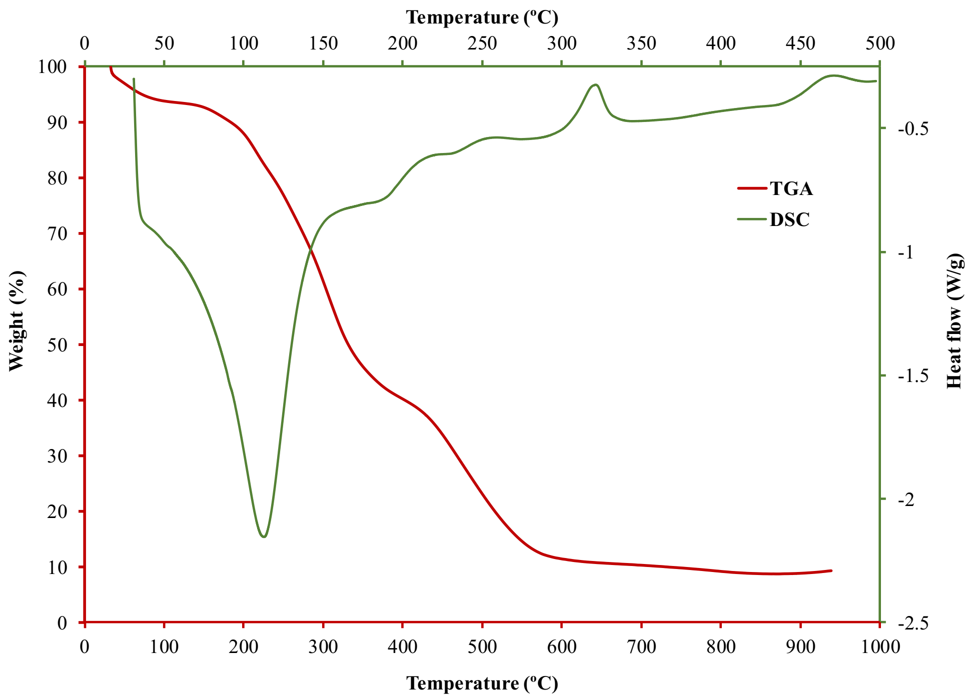

Fig. 5 TGA and derivative thermogravimetric graphs of marigold flower powder

such as C=C and C=O and its presence were shown by the stretching of bonds at peak 1596 cm^{-1}. The broad band following signifies, at 3400–3410 cm^{-1}, alcohols, organic acids, and phenols with H-bonded O–H stretching vibrations of hydroxyl groups; at 2850–2950 cm^{-1}, alkyl structures with C–H stretching of alkyl structures; at 1620–1650 cm^{-1}, asymmetric stretching of COO–; at 1460 cm^{-1}, C–H deformation of CH_3 group; at 1280–1270 cm^{-1}, phenolic compounds with O–H stretching; the bands nearby 460, 800, and 1000–1100 cm^{-1}, bending of Si–O stretching [35, 36]. The reduction in the intensity of the peak around 3400 cm^{-1} may be due to dehydration of the biomass during the pyrolysis (Fig. 5). The disappearance of the peak at 2944 cm^{-1} may be due to the removal of volatiles present in the biomass (Marigold).

Scanning Electronic Microscope Results

SEM images of the biochar revealed the evident morphological difference between BC350 and BC500. The details of mesopores and micropores and the arrangements of the pore can be studied from SEM images [37]. The images of FP showed a smooth integrated surface and absence of pores, compared to the biochar, where the surface was rough and pores were visible.

By witnessing the numbers and arrangement of the pores, the biochar surface has exposed several morphological differences. The SEM image of BC350 showed the appearance of fusing of biomass into vesicles, with predominant melting (Fig. 6). The morphology of BC500 showed well-structured pores, while certain portions appeared brittle (Fig. 7). The morphological differences are attributed to the release of volatiles accompanied by the temperature rise, which resulted in the formation of the pores, and the decomposition of biomass resulted in cracks and fractures in the biochar [37].

BET Analysis

The surface area and pore volume of biochar BC350 and BC500 were acquired by the adsorption of nitrogen as shown in Table 3. The specific surface area values are of various extents. As a result of increasing the pyrolysis temperature from 350 to 500 °C has increased in the S_{BET} from 5.09 $m^2\ g^{-1}$ to 8.60 $m^2\ g^{-1}$ and in the V_T from 2.3×10^{-3}·cm^3·g^{-1} to 4.1×10^{-3}·cm^3·g^{-1}. Similarly, S_{mic} was negligible in BC350, showing the absence of cylindrical micropores; also V_{mic} in BC500 was about 1.05×10^{-3}·cm^3·g^{-1}. During the process of pyrolysis, the progressive degradation of organic materials present in the flower waste constitutes hemicellulose, cellulose, and lignin indicated by the formation of the vascular bundles and channel structures or cylindrical pores. As a result of degradation, the surface area and total pore volume are increased [36].

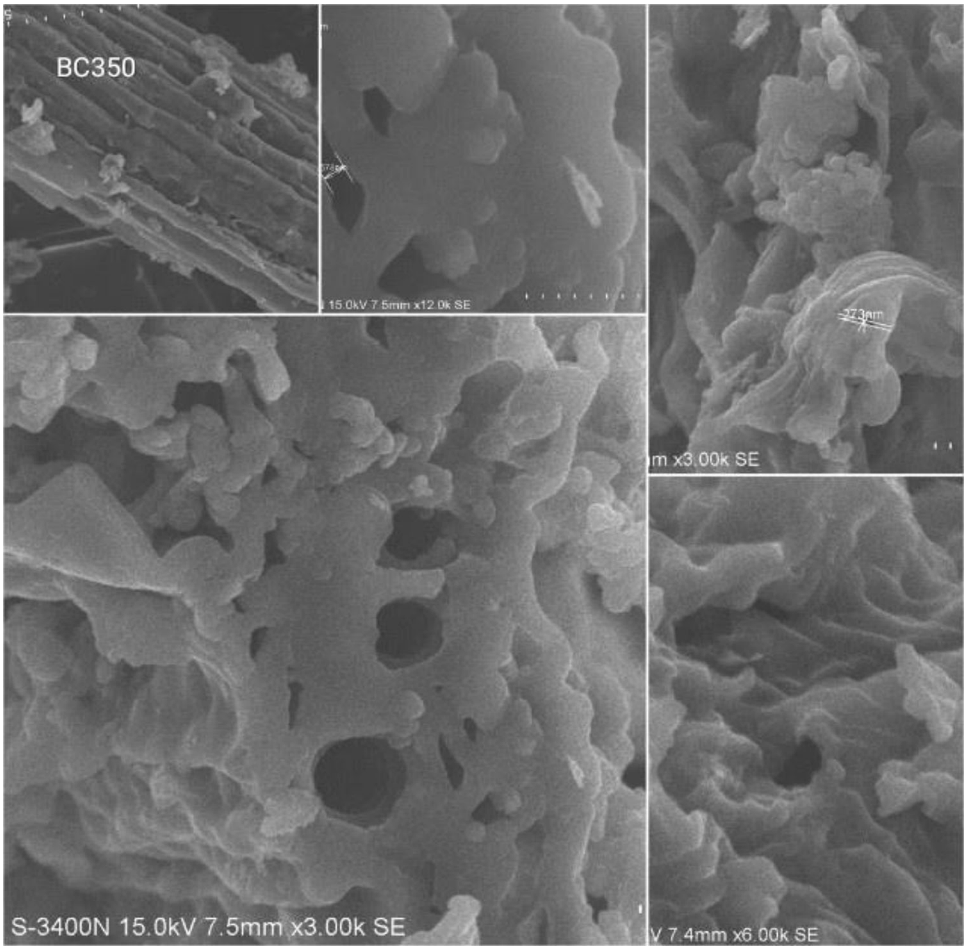

Fig. 6 SEM micrographs of BC350

X-ray Diffraction (XRD)

"XRD is utilized for creation investigation of biochar and the assurance of crystalline C and the insights regarding organics and inorganics [36]. Different diffraction tops at 2 h that are 19°, 22°, 28°, 31°, 43°, 47°, 51°, and 66° were fixed by the examples (Fig. 8). A wide mound was seen from 20 to 30° for the two examples, uncovering the crystallographic planes of cellulosic and silica. A few little and sharp tops in the XRD design were seen with pyrolysis temperature increment, imply the existence of cellulose ($2\theta = 19°$), and minerals like quartz or SiO_2 ($2\theta = 28°$) and calcite ($2\theta = 31°$) [38].

Effect of Aqueous Extract of Flower Waste-Derived Biochar on the Germination of *Brassica juncea*

The existence of different minerals upgraded the estimation of the chosen biochar as a capable soil changing material [39, 40]. In this study, for providing the basic information for effective crop production, we examined the effect of aqueous biochar extract on the seed germination and seedling growth of *Brassica juncea*.

Brassica juncea as a testing plant was chosen for the study. Indian mustard is a sensitive leafy vegetable and is commonly consumed worldwide [41]. Three grams of oven-dried, 2 mm sieved powder of different biochar samples were taken in a 300-mL Erlenmeyer flask, and then, 100 mL of distilled water was added. After stirring for 2 h at 200 rpm on a magnetic stirrer in dark, the mixtures were filtered through Whatman No. 42 filter paper. The extract obtained from biochar, pyrolyzed at 350 °C and 500 °C, is hereafter denoted as E350 and E500. The effect of the aqueous extracts in this study was examined at three concentrations of 10%, 30%, and 100% (v/v). The different aqueous extracts were added into a 100 mm × 15 mm size Petri dish with a moisturized blotting filter paper and fourth seeds were placed in each Petri dish, all in triplicates in an environmental test chamber (T08, Technico®) at 28 ± 2 °C maintained at a relative humidity of 62 ± 3% [42]. The seeds were incubated in dark for the first 48 h, followed by a 16-h light and 8-h dark cycle with

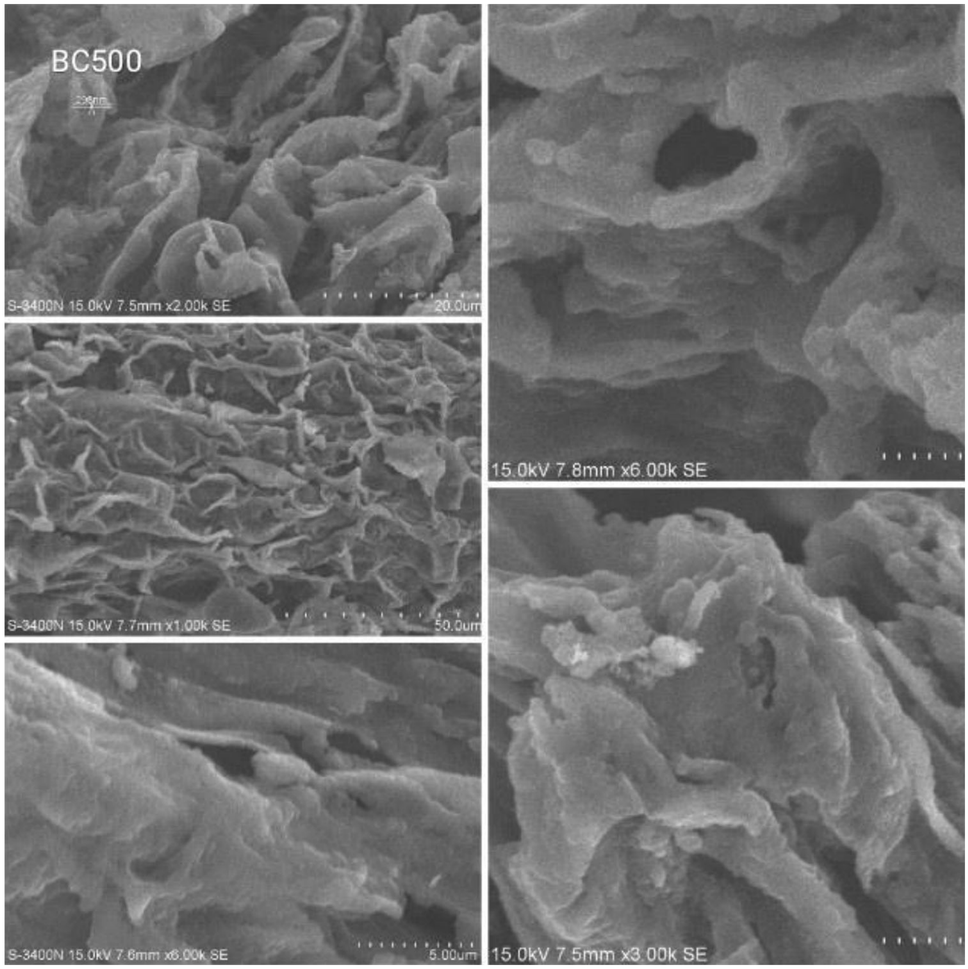

Fig. 7 SEM micrographs of BC500

Table 3 Results from BET analysis

Sample	BC350	BC500
S_{BET} (m^2 g^{-1})	5.09	8.61
S_{mic} (m^2 g^{-1})	***	2.01
V_T (10^{-3}·cm^3·g^{-1})	2.34	4.15
V_{mic} (10^{-3}·cm^2·g^{-1})	***	1.05

S_{BET}, S_{mic}, V_T, and V_{mic} were the BET surface area, micropore surface area, total pore volume, and micropore volume, respectively

***Pores could not be located by *t* plot

a light intensity of 4500 ± 300 lx. Germination count was done every 24 h; lengths of both hypocotyl and radicale of mustard seedlings were measured after 120 h of culture. Germination percentage was determined by the formula of [43] formula [44]:

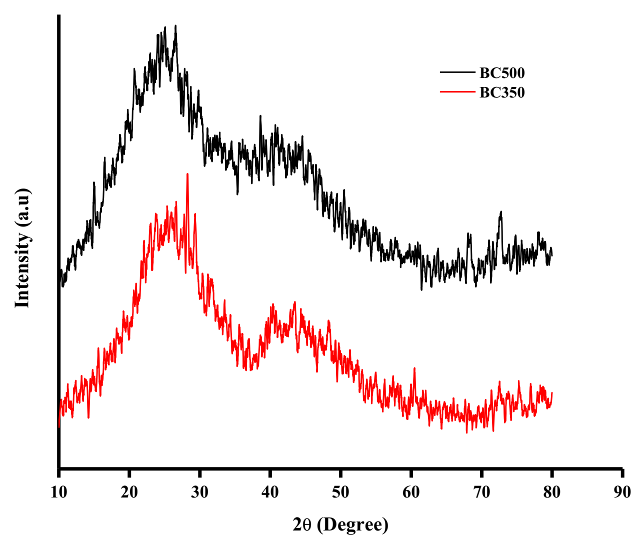

Fig. 8 XRD graph of BC350 (blue) and BC500 (green) (color figure online)

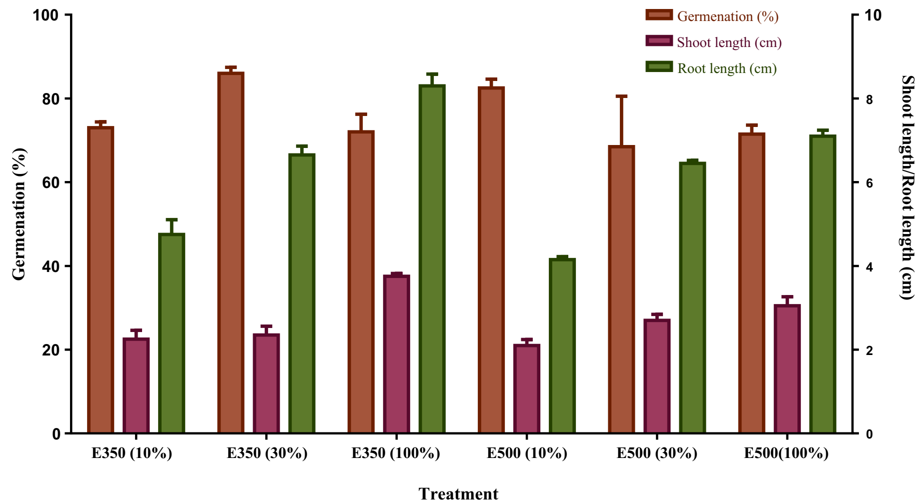

Fig. 9 Effect of aqueous extract of biochar on germination percentage, shoot and root length

$$\text{Germination }\% = \frac{\text{Number of seeds generated during the time interval}}{\text{Total number of seeds sown}}$$

Aqueous extracts of biochar showed varied effects on the germination, shoot and root length of the mustard seedlings [45], as shown in Fig. 9. The results showed that E350 and E500 with different concentrations of 10%, 30%, and 100% resulted in similar or promoted the germination percentage, shoot and root growth of Indian mustard compared to the control. The biochar synthesized at higher temperatures has a high concentration of PAHs in their aqueous extracts [46]; these characteristics of extract influence the germination as well as the development of shoot and root [47], considered as reliable indicators for the sensitivity of plant to phototoxic compounds [48]. The germination percentage was seen higher in treatment E350 for 30% concentration. Seed germination is a critical phase in a plant's life cycle and is highly responsive to the environment surrounding it [49]. The influence of the pyrolysis temperature and concentration of biochar extract showed varying effects on seedling development. Treatment E350 showed maximum seedling development, with mean root and shoot length up to 8 cm and 4 cm, respectively, at 100% concentration. Biochar synthesized at higher temperatures shows a high risk in plant germination and seedling development [50]; the same was ascertained by our study. However, studies regarding inhibition and nonsignificant impact have also been observed [51, 52]. This study provides evidence of simulation in seedling growth, and thus, biochar synthesized from marigold can utilize as a soil amendment or fertilizer.

Conclusion

The present work toward waste minimization through the preparation of biochar from waste flowers by direct pyrolysis process and with no compound enactment evinces to be an accomplishable strategy. The physicochemical parameters of biochars were analyzed. The impact of pyrolyzing temperature on the properties of biochar was obvious. The biochar BC500 was made permeable and genuinely better than BC350 in numerous regards. The BC500 demonstrated a vastly improved surface area than the BC350, great carbon sequestration, and higher moisture holding uncovering its potential for different ecological advantages. The valorization of flower waste through thermal treatment sounds is attainable, and it very well may be considered as an eco-friendly alternative. The constructive utilization of flower waste from the market lessens the waste entering into the landfill site and value addition to the flower waste. Expanding the use of biochar in various applications like carbon sequestration, diverse contaminant removal, and enhancement of physical–chemical property of soil ensures the sustaining demand for biochar, especially from the inexpensive feedstock. The current study suggests that the pyrolysis of flower waste to yield biochar is a feasible option for revenue generation and the knowledge on the application of biochar to various processes is essential for the commercial extension. Identifying the specific and unique application for flower-based biochar among other feedstock is the design of the future scope of this study.

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Declarations

Consent for Publication All authors agreed to publish their data before biochar and have not submitted the data in any form to any journal.

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