



## Evaluating Biochar Properties Derived from Various Agro-residues Via Pyrolysis: A Comparative Study

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### ABSTRACT

This study was designed to analyze the physicochemical characteristics and functional groups of biochar obtained from four agro-residues-sugarcane bagasse (SB), rice husk (RH), cotton stalk (CS), and orange peel (OP). Pyrolysis of agro-residues was performed at 450°C for two hours. Elemental analysis indicated carbon concentrations between 19.39% to 66.30%, whereas oxygen levels decreased due to pyrolysis, signifying increased aromaticity and biochar stability. Structural characterisation revealed a peak cellulose content in OP (55.89%) and the maximum lignin concentration in RH (26.1%), both of which influence the biochar's adsorption capacity and nutrient retention potential. FTIR analysis verified essential functional groups, such as O-H, C-H, C=O, and aromatic C=C bonds, which augment biochar's reactivity and nutrient-binding potential. The alkaline pH (9.2–11.2) and diverse electrical conductivity values indicate the appropriateness of biochar for enhancing soil quality, especially in acidic soils. The findings suggest that the distinctive characteristics of biochars formed from agro-residues render them advantageous for sustainable agricultural uses, including nutrient provision and soil enhancement.

**Keywords:** Biochar, Pyrolysis, Agro-residues, Rice husk, Cotton stalk, Orange peel, and Sugarcane bagasse, Structural characterization, Sustainable agriculture.

### INTRODUCTION

Agriculture and its related sectors account for 17.32% of India's gross domestic product and support the livelihoods of around 60% of the population<sup>1</sup>. The use of agrochemicals, high-yielding variety seeds, and advanced machinery has led to a notable enhancement in agricultural production. As agricultural production rises, there is a corresponding increase in the pollution linked to this sector<sup>2,3</sup>. The

generation of agrochemicals and poorly managed agricultural waste significantly contribute to carbon emissions in the agriculture sector<sup>4</sup>. Moreover, the management of agricultural residues continues to be a significant issue in India<sup>5,6</sup>. The Indian Ministry of New and Renewable Energy reported that India generates 500 million tonnes of agro residue each year. A significant portion, ranging from 70% to 75%, of this waste is utilised in various industrial processes like fodder, fuel, and other applications. However,



there remains a surplus of 140 million tonnes that is incinerated, leading to environmental hazards<sup>7</sup>. The practice of burning crop residue emits harmful air pollutants, including particulate matter, ammonia, sulphur dioxide, nitrogen oxides, and carbon dioxide, which results in the depletion of essential nutrients such as nitrogen and organic carbon that would typically remain in the soil<sup>8,9,10</sup>. It has been reported that 1.41Mt of CO<sub>2</sub> is emitted for every Mt of crop residue that is burned<sup>11</sup>. In light of the present situation, it is clear that rigorous agricultural methods, alongside rising food requirements driven by population expansion, have exerted substantial strain on the agricultural sector, leading to a notable rise in waste and carbon emissions. This highlights the necessity for technological solutions to effectively manage agricultural waste for sustainable production within agroecosystems. Recently, biochar, which is a carbonised biomass obtained from different ligno-cellulosic materials like agricultural residue and wood, has gained significant interest as a cost-effective and environmentally friendly method for managing agricultural waste<sup>12,13,14</sup>. Biochar, with its extensive surface area, catalytic capabilities, and resistant characteristics, offers various environmental applications such as soil remediation<sup>15</sup>, wastewater treatment<sup>16,17</sup>, and carbon sequestration<sup>18,19</sup>. The majority of earlier investigations had concentrated on the production of biochar from sources such as wood, bamboo, and biosolids. The application of agro residue for biochar synthesis remains in the early stages of development. This study seeks to examine and assess the diverse potential of biochar derived from agro residues across various environmental matrices through a detailed analysis of their properties. Current study examined the various properties of biochar derived from four distinct agro-residues: Sugarcane bagasse (SB), Rice husk (RH), Cotton stalk (CS), and orange peel (OP).

## MATERIALS AND METHODS

### Biochar preparation

Biochar was produced from agro-residues (rice husk, cotton stalk, orange peel, and sugarcane bagasse). Agro-residues were firstly dried ensuring moisture content below 10%. All the residues were processed and sieved to maintained the size 2–10 mm. Equal amounts of each residue were used for biochar production to maintain uniformity. Samples

were placed in steel vessel to further minimize oxygen exposure. The process was carried out in a muffle furnace for 2 hours. The furnace was programmed to reach the target temperature (450°C) at a controlled heating rate (e.g., 10°C/min) to avoid thermal shocks. After 2 h, the furnace was allowed to cool naturally to room temperature before removing the samples to avoid thermal cracking.

### Proximate Analysis

Proximate Analysis determines the basic composition of biochar, including moisture content (MC) which Indicates the water content in biochar, measured by heating the sample at 105°C until a constant weight was achieved; volatile matter (VM) which was measured by heating the biochar at 550°C in a closed environment to quantify the gases released; ash content was determined by combusting the biochar at 850°C in an open environment to leave behind inorganic residue. Cellulose, hemicelluloses and lignin were also analysed as per Goering and Van Soest, 1970<sup>20</sup>.

Fixed Carbon (FC) which represents the stable carbon fraction was calculated using following equation:

$$\text{Fixed Carbon} = 100 - \{\text{Moisture (\%)} + \text{Volatile Matter (\%)} + \text{Ash (\%)}\}$$

### Ultimate Analysis

Ultimate Analysis measures the elemental composition of biochar. Following methods were used for ultimate analysis:

### Carbon (C), Hydrogen (H), Oxygen (O), Nitrogen (N) content

This indicated biochar's carbonization level, energy content, and potential for nutrient enrichment. Analysed using EDX (CARL ZEISS, SIGMA 360).

### Surface Area and Porosity

Surface Area and Porosity were analysed using BET (Brunauer-Emmett-Teller) Analysis (Quantochrome (Autosorb1Q-C-MP)).

### Surface Chemistry

Surface chemistry of biochar was analysed via (Fourier Transform Infrared Spectroscopy) (Perkin Elmer, Spectrum 100) in the wave number range of 4000-400 cm<sup>-1</sup>, which identified functional groups (e.g., -OH, -COOH, -CH<sub>3</sub>) on the biochar surface by

detecting characteristic infrared absorption bands.

## RESULTS

### pH and EC

pH and EC of the BC samples was measured using the HM Digital (pH-200) metre and EC metre (ELICO 183 EC-TDS ANALYSER) respectively in the ratio of 1:20.

### Surface Charge

Surface charge was analysed using a Zeta sizer (Malvern, Nano-ZS).

### Statistical analysis

All the experiments were carried out in triplicate and the results were expressed in average values.

### Proximate, ultimate and biochemical analysis of prepared biochar from agro-residues

There were noticeable changes in the agro-residues' physicochemical characteristics as presented in Fig. 1. Proximate analysis of the biochar of various materials was done for the following parameters: ash, volatile matter, fixed carbon and moisture content. Ultimate analysis of the biochar of various materials was done for the following parameters: C%, H%, N%, S%, O%, O/C, H/C, C/N, (O+N)/C. Physical, chemical and pore structural analysis of the biochar of various materials was done for the following parameters: pH, EC, CEC, SSA, TOC, HHV, and pore volume.

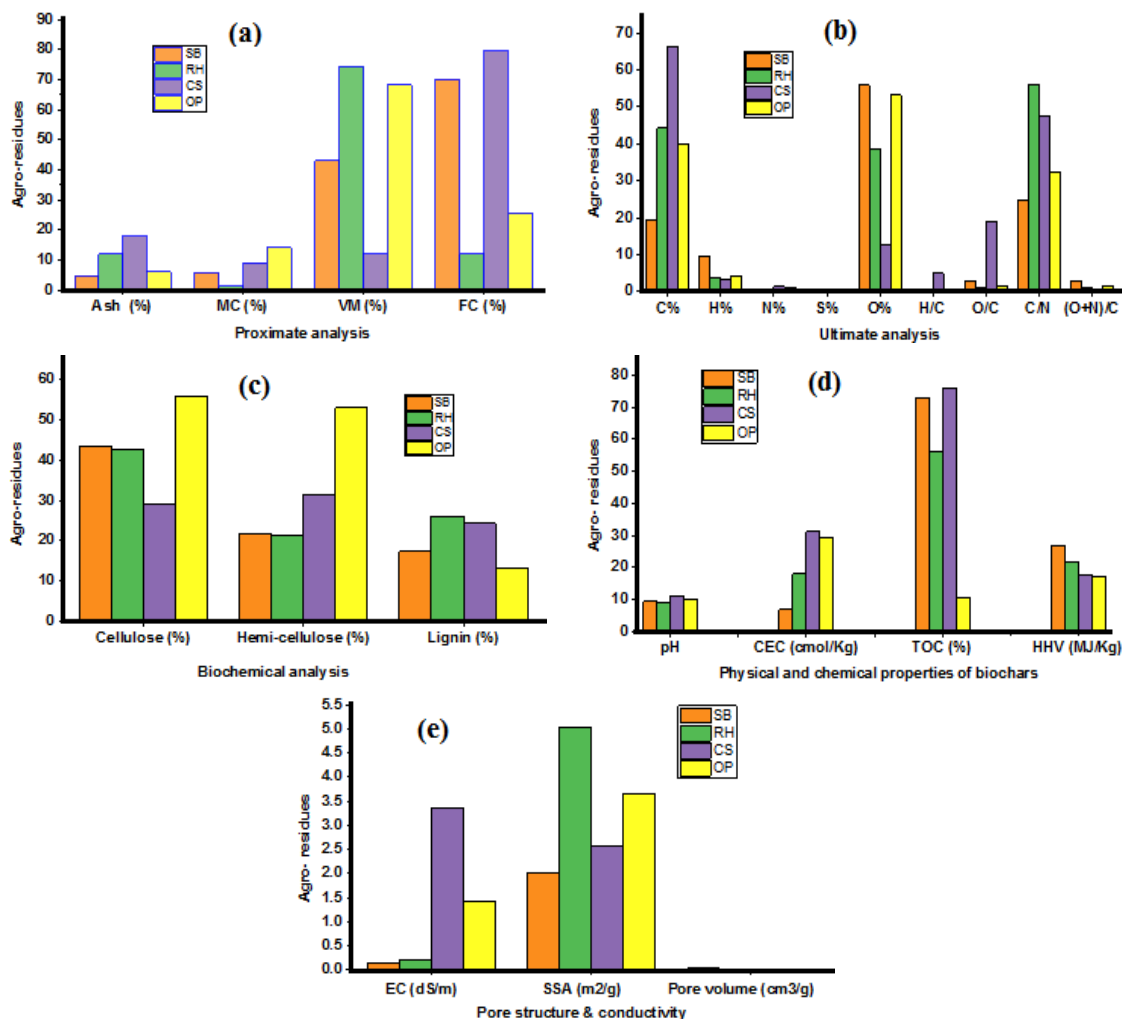


Fig. 1. Proximate and ultimate analysis of biochars prepared from all 4 agroresidues (a) ash%, volatile matter (VM), moisture content% (MC) & fixed carbon% (FC) (b) elemental analysis of biochars and their respective ratios, (c) biochemical analysis (cellulose, hemicellulose & lignin), (d) pH, CEC, TOC%, HHV, (e) pore structure and electrical conductivity (EC)

**Proximate analysis:** This study indicated significant variations in the composition of the four biomass materials examined sugarcane bagasse, rice husk, cotton stalk, and orange peel (Fig. 1a). The cotton stalk had the highest ash level at 18.10%, succeeded by rice husk at 11.98%, orange peel at 6.20%, and sugarcane bagasse at 4.82%. Regarding moisture content, orange peel had the greatest value at 14.20%, whereas rice husk displayed the lowest at 1.29%. Sugarcane bagasse and cotton stalk demonstrated intermediate moisture content at 5.82% and 9.08%, respectively. Rice husk exhibited the most volatile matter at 74.53%, followed by orange peel at 68.30%. Sugarcane bagasse and cotton stalk exhibited significantly lower values of 43.20% and 12.08%, respectively. In contrast, the fixed carbon content was greatest in cotton stalk (79.63%) and sugarcane bagasse (70.10%), while, orange peel (25.72%) and rice husk (12.11%) exhibited markedly reduced fixed carbon content. These variances indicate that each biomass material possesses distinct features that may affect its appropriateness for particular purposes.

**Ultimate analysis:** The comprehensive investigation of the four biomass materials revealed unique elemental compositions (Fig. 1b). The carbon content (C%) was highest in CS (66.70%), followed by RH(44.24%), OP(39.7%), and SB(19.39%). Hydrogen (H%) was comparatively higher in SB(9.68%) and lowest in CS(2.50%), moderately found in rice husk and orange peel (3.91% & 4.26% respectively). Nitrogen(N%) content varied, with the greatest value being CS(1.48%), followed by OP (1.23%), SB(0.79%), and RH(0.75%). Sulfur(S%) was only found in CS(0.28%) and OP(0.21%). The oxygen (O%) content was significantly higher in SB(55.88%) and OP(53.12%), moderate in RH (38.56%), and lowest in CS(10.80%). Due to its low hydrogen content, CS has a substantially higher H/C ratio (3.74) than others with minimum for RH (0.088), than orange peel (0.10) and sugarcane bagasse (0.49). Similarly, CS had the largest O/C ratio (16.19), indicating a high oxygen content relative to carbon, while RH had the lowest (0.87) followed by sugarcane bagasse (2.88) & orange peel(1.33). The C/N ratio showed that RH(55.98) and CS(45.06) were more carbon-rich than SB(24.54) and OP(32.27). The (O+N)/C ratio followed a similar pattern, with SB(2.92) the greatest and CS(0.18) the lowest. The results emphasize the unique

chemical profiles of each biomass, indicating their potential applicability for diverse uses, such energy generation, biochar formation, or material synthesis, based on their individual elemental composition.

**Physical, chemical-biochemical, and pore structural analysis:** The biochar produced from sugarcane bagasse, rice husk, cotton stalk, and orange peel exhibited significant variations in their physical and chemical properties (Fig. 1 d). Cotton stalk had the maximum electrical conductivity (EC) at 3.36 dS/m, succeeded by orange peel at 1.42 dS/m and rice husk at 0.22 dS/m, but sugarcane bagasse recorded the lowest EC at 0.15 dS/m. The pH varied from 9.2 to 11.20 {(rice husk (9.2), sugarcane bagasse (9.3), orange peel (10.09), cotton stalk (11.2)}, demonstrating that all biochars were alkaline. Total organic carbon (TOC) was highest in cotton stalk (76%) whereas it was lowest in orange peel (10.62%). Cellulose was the most abundant structural component in OP at 55.89% and the least abundant in CS at 28.98%. OP had the most hemicellulose at 52.9% and SB the least at 21.8%. The lignin content ranged from 13.3% in OP to 26.1% in RH (Fig. 1c). The specific surface area (SSA) was greatest in rice husk (5.02 m<sup>2</sup>/g), although the pore volume was marginally elevated in sugarcane bagasse (0.0358 cm<sup>3</sup>/g) relative to the other materials. Op had the greatest electrical conductivity (OP) at 1.42 mS/cm, while SB had the lowest at 0.15 mS/cm (Fig. 1e). The results underscore the variability in biochar characteristics, suggesting their distinct potential uses in soil enhancement, adsorption, or energy generation.

### Surface Properties

In order to understand the chemical composition and structural properties of the four agro residues-sugarcane bagasse (SB), rice husk (RH), cotton stalk (CS), and orange peel (OP)-the FTIR analysis showed that they all included different functional groups. In SB, O-H stretching vibrations linked to water, carboxylic acids, and phenols, as well as a relatively narrow range of 3340-3353 cm<sup>-1</sup>, were responsible for the large peak at 3500-3000 cm<sup>-1</sup>. The C-H stretching vibrations of lignin and polysaccharides were represented by the peaks at 2900-2800 cm<sup>-1</sup>. Deformation vibrations of C-H bonds were observed below 1500 cm<sup>-1</sup>, but aromatic C-C bonds in lignin are indicated by the peaks around 1642-1600 cm<sup>-1</sup>. C-O-C vibrations

linked to polysaccharides were detected at  $1160\text{ cm}^{-1}$ , while ester C-O-C vibrations were detected at  $1250\text{ cm}^{-1}$ . It was at  $1050\text{ cm}^{-1}$  that the C-OH groups were clearly visible. In RH, the peaks at  $3010$  and  $3040\text{ cm}^{-1}$  were associated with vibrations that strain the C-H bonds. Between  $1167$  and  $1170\text{ cm}^{-1}$ , secondary N-H<sub>3</sub> groups were detected. Peaks at  $1600\text{ cm}^{-1}$  correspond to C=O bonds in ketones and C=C bonds in quinines, while  $1244\text{-}1249\text{ cm}^{-1}$  was found to be aryl-O stretching. The vibrations of phenolic O-H bonds were detected at  $1375\text{ cm}^{-1}$ . While symmetric and asymmetric C-O bonds were detected at  $1050$  and  $1150\text{ cm}^{-1}$ , respectively, symmetric C-O stretching vibrations were seen at  $1100\text{ cm}^{-1}$ . In case of CS, the O-H stretching vibrations were seen in the  $3846$ ,  $3740$ , and  $3615\text{ cm}^{-1}$  region, along with additional peaks at  $1523(\text{C}=\text{C})$ ,  $1696(\text{ester, aryl ketone, ketone})$ ,  $1413\text{-}1739\text{ cm}^{-1}$  reflecting carboxylic acids (COOH), carbonyl (C=O), and amine (NH<sub>2</sub>) stretching vibrations. The aromatic C=C bonds were detected around  $1409\text{-}1463\text{ cm}^{-1}$ , while the C-OH, Arom-H ( $1000\text{-}1290$ ), C-C ( $1260\text{-}700$ ), C-N ( $1230\text{-}1020$ ), C-O ( $1250\text{-}1050$ ) bonds were seen around  $1052\text{ cm}^{-1}$ . The vibrations of C-H bonds were indicated by the peaks at  $780\text{-}874\text{ cm}^{-1}$ . OP exhibited O-H stretching vibrations between  $3500\text{-}3353\text{ cm}^{-1}$ , and C-H stretching vibrations (corresponding to methyl, methoxy, and methylene groups) were detected at  $2960\text{ cm}^{-1}$ . N-H stretching vibrations were thought to be responsible for the peaks at  $1592\text{ cm}^{-1}$ , while NO symmetric stretching was observed at  $1374\text{ cm}^{-1}$ . At  $780\text{ cm}^{-1}$ , vibrations of the aromatic C-H bonds were detected. The BET surface area of the residues increased significantly after treatment. RH exhibited the highest surface area ( $5.02\text{ m}^2/\text{g}$ ), while SB had the lowest ( $2.01\text{ m}^2/\text{g}$ ), indicating differences in porosity and thermal decomposition patterns. ZETA results showed that SB & CS had negative surface charge while RH & OP had positive surface charge.

## DISCUSSION

The results showed that the agro residues have different characteristics, which shows how useful they are in different contexts. The low ash content in SB makes it appropriate for biofuel applications, whereas the higher ash content in CS and RH implies possible use in mineral recovery. Energy generation is well-suited to RH and OP due to their high volatile matter

content, whereas CS's low VM content reflects its superior thermal stability. Soil amendments, especially in acidic soils, may be possible with OP and CS due to their high electrical conductivity and pH. Because of its higher cellulose and hemicellulose concentration, OP shows promise for bioethanol synthesis, according to the structural analysis. On the other hand, RH and CS are more suited for biochar or thermochemical applications, due to their high lignin content. Based on their composition and qualities, these findings highlighted the residues' appropriateness for various industrial, agricultural, or energy-related applications. Thermal treatment had diverse effects on the four agro residues, as seen by the differences in elemental composition and atomic ratios. Due to its higher carbon content, CS shows more carbonisation and may be more suited for uses involving biochar or carbon-based materials. RH's enhanced aromaticity and thermal stability, brought about by its low H/C and O/C ratios, make it an excellent candidate for carbon storage or adsorption applications that involve long-term exposure to carbon. However, SB exhibited poor pyrolysis efficiency due to the retention of several oxygenated functional groups, despite having the maximum oxygen content and (O+N)/C ratio. As a result, SB may become more reactive and useful as a soil amendment or in catalytic processes. OP may find usage in niche markets that demand sulphur-based functionalisation due to its moderate carbon concentration and 0.21% sulphur content. Taken together, these findings provide evidence that the treated residues have unique characteristics that might be useful in energy, environmental, or material science contexts, depending on the final goal. As a reflection of their unique compositions and their uses, the agro-residues' FTIR spectra show the presence of different functional groups. Sugarcane bagasse (SB) can be a potential feedstock for bioethanol production or products that need structural integrity because it showed strong O-H and C-H vibrations, which are signs of its polysaccharide and lignin content. The existence of phenolic and ester groups raises the possibility of chemical modification and functionalisation for adsorption or catalytic applications. As a result of enhanced thermal degradation of lignocellulose, RH exhibited peaks for C=C and C=O bonds in quinines and ketones. Biochar production for pollutant adsorption is one example of an application that could benefit from its reactive sites or antioxidant properties, both of which are enhanced by the presence of aryl-O stretching

groups. Considerable O-H, C=O, and NH<sub>2</sub> vibrations were seen in CS, indicating a larger concentration of polar functional groups. This finding may increase CS's potential use as a bioactive material precursor or soil amendment. Biochar produced from carbon sequestration or adsorptive processes is stable because of the aromatic C=C bonds and low C-H concentration, which imply increased aromaticity. The significant cellulose and hemicellulose content of OP was reflected in its diverse spectrum of functional groups, which included O-H, NO<sub>2</sub>, and methyl/methoxy groups. For these reasons, OP being considered as a possible biofuel or bioethanol source. It may find use as a polymeric material or in catalytic reactions due to the presence of aromatic C-H groups and N-H bonds. By and large, the FTIR results showed that these agro-residues contain a wide variety of functional groups, which, depending on the feedstock, may indicate that they have energy, environmental, or material science-specific applications.

### CONCLUSION

This work thoroughly assessed the physicochemical, structural, and functional group characteristics of biochars obtained from four agro-residues-sugarcane bagasse (SB), rice husk (RH), cotton stalk (CS), and orange peel (OP)-synthesized at 450°C for 2 hours. The findings revealed considerable differences in elemental composition, functional groups, and structural properties among the agro-residue biochars, indicating their varied potential for agricultural and environmental uses. Elemental analysis demonstrated carbon enrichment and oxygen depletion during pyrolysis, with the maximum carbon content recorded in CS(66.30%) and the minimum in SB(19.39%), signifying differences in carbonisation efficiency. The hydrogen-to-carbon (H/C) and oxygen-to-carbon (O/C) molar ratios were maximum in CS biochar, indicating enhanced aromaticity and thermal stability, thereby rendering it appropriate for prolonged soil amendment and carbon sequestration. The structural investigation revealed that OP had the most cellulose and hemicellulose content (55.89% & 52.9%), whereas RH had the highest lignin content (26.1%), influencing the biochar's porosity and adsorption ability. FTIR analysis revealed functional groups such as O-H, C-H, C=O, and aromatic C=C bonds

in all biochars, with SB and OP exhibiting elevated polysaccharide peaks, RH exhibiting significant phenolic and quinone functional groups, and CS displaying increased aromaticity and polar functional groups. These functional groups play an important role in nutrient binding and slow-release qualities, which are necessary for biochar to function in nutrient delivery systems and improve soil fertility. The alkaline pH (9.2-11.2) and moderate to high electrical conductivity (EC) values indicate that these biochars can improve acidic soils and provide essential nutrients. The enhanced lignin and aromatic content in RH and CS biochars improves their adsorption capacity, but the higher cellulose concentration in OP makes it suitable for biofuel applications. These biochars' versatility makes them useful as pollution adsorbents and carriers for slow-release fertilisers, so improving sustainable nutrient management and environmental conservation. In conclusion, biochars derived from agricultural leftovers have significant potential for use in sustainable agriculture and environmental management. Their various physicochemical properties enable specialised applications such as nutrient delivery, soil augmentation, pollutant adsorption, and carbon sequestration in conformity with global sustainable development goals. The use of cotton stalk biochar in the manufacturing of potassium slow-release fertilisers demonstrates its importance in boosting nutrient efficiency and supporting sustainable agricultural methods. Subsequent research could focus on optimising pyrolysis parameters and conducting field-scale studies to fully realise their potential for increasing agricultural productivity while reducing environmental impact.

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### Conflict of interest

The author declare that we have no conflict of interest.

## REFERENCES

1. GOI., *Economic Survey of India.*, 2020 2021., **2021**, 2(9), 319–345.
2. Harizanova Bartos, H. and Stoyanova, Z., Impact of agriculture on air pollution., *CBU International Conference Proceedings.*, **2018**, 6, 1071–1076. [https://www.indiabudget.gov.in/budget2021\\_22/economicsurvey/doc/echapter\\_vol2.pdf](https://www.indiabudget.gov.in/budget2021_22/economicsurvey/doc/echapter_vol2.pdf).
3. Liu, Y.; Wang, P.; Gojenko, B.; Yu, J.; Wei, L.; Luo, D. and Xiao, T., *Environ. Pollut.*, **2021**, 291, 118209.
4. Kitamura, R.; Sugiyama, C.; Yasuda, K.; Nagatake, A.; Yuan, Y.; Du, J.; Yamaki, N.; Taira, K.; Kawai, M. and Hatano, R., *Front. Sustain. Food Syst.*, **2021**, 5, 649613.
5. Kumar, P., & Singh, R. K., *Environ., Dev. Sustain.*, **2021**, 23, 3696-3730.
6. Kumar, V.; Dev, M.; Janaagal, M.; Pooja, P.; Kumari, G.; Devendrappa, P. S.; Suresh, G.; Tallapragada, S. and Kthyalia, P., *Egypt. J. Soil Sci.*, **2025**, 65(1), 33-44.
7. Jain, R.; Naik, S.N. In: Baskar, C.; Ramakrishna, S.; Baskar, S.; Sharma, R.; Chinnappan, A.; Sehwat, R. (eds) Handbook of Solid Waste Management. **2022**, Springer, Singapore. [https://doi.org/10.1007/978-981-16-4230-2\\_26](https://doi.org/10.1007/978-981-16-4230-2_26)
8. Abdurrahman, M. I.; Chaki, S.; & Saini, G., *Environ. Adv.*, **2020**, 2, 100011.
9. Kingra, P.; Bora, S., & Singh, Y., *J. Agric. Phys.*, **2021**, 21(2), 316-331.
10. Kumar, R.; Kaur, A.; Sharma, S.; Bharti, H., & Kumar, R., *Educ. Admin. Theory Pract.*, **2024**, 30(5), 7253-7273.
11. Chandana, P.; Kumar Singh, P. and Shivashankar, K., *Int. J. Curr. Microbiol. Appl. Sci.*, **2020**, 9(11), 3077–3102.
12. Seow, Y. X.; Tan, Y. H.; Mubarak, N. M.; Kansedo, J.; Khalid, M.; Ibrahim, M. L., and Ghasemi, M., *J. Environ. Chem. Eng.*, **2022**, 10(1), 107017.
13. Amalina, F.; Abd Razak, A. S.; Krishnan, S.; Sulaiman, H.; Zularisam, A. W., & Nasrullah, M., *J. Hazard. Mater. Adv.*, **2022**, 7, 100134.
14. Malik, S.; Laura, J. S., & Khyalia, P., *Egypt. J. Soil Sci.*, **2024**, 64(4), 1479-1493.
15. El Naggar, A.; Ahmed, N.; Mosa, A.; Niazi, N. K.; Yousaf, B.; Sharma, A.; Sarkar, B.; Cai, Y. and Chang, S. X. Nickel in soil and water: Sources, biogeochemistry, and remediation using biochar., *J. Hazard. Mater.*, **2021**, 419, 126421.
16. Enaime, G.; Baçaoui, A.; Yaacoubi, A. and Lübken, M. Biochar for wastewater treatment: Conversion technologies and applications., *Appl. Sci.*, **2020**, 10(10), 3492.
17. Wang, X.; Guo, Z.; Hu, Z. and Zhang., *J. Peer.*, **2020**, J8, e9164.
18. Wijitkosum, S. and Jiwnok, P., *Appl. Sci.*, **2019**, 9(19), 3980.
19. Lefebvre, D.; Williams, A.; Meersmans, J.; Kirk, G. J. D.; Sohi, S.; Goglio, P. and Smith, P., *Sci. Rep.*, **2020**, 10(1), 19479.
20. Goering, H.K. and Van, S.P.J. Forage fibre analyses (apparatus, reagents, procedures and some applications). Agricultural Hand book, **1970**, United States Department of Agriculture.