Journal of Metals, Materials and Minerals, 37(1), e2639, 2027

Supplementary Information

Sustainable biochar production from pine needle waste by retort heating for value-added functional carbon materials

Arpita Roshni BEHERA[1], Prachi SINGHAL[1,*], and Sunita RATTAN[1]

[1] *Amity Institute of Applied Sciences, Amity University Uttar Pradesh, Noida, Uttar Pradesh, 201313, India*

***Corresponding author e-mail**: psinghal@amity.edu

Received date:
2 February 2026
Revised date:
24 June 2026
Accepted date:
29 September 2026

Keywords:
Pine needles;
Biomass;
Carbonization;
Biochar;
Graphitization

Abstract

Sustainable management of pine needles, as a major forest waste product, is a critical challenge due to their high flammability, potential to create fire and ability to restrict growth of other vegetations. One of the possible solutions to this challenge is to utilize pine needles to produce value added products following the concept of circular economy. The present work focuses on utilizing pine needle waste to produce biochar and biochar derived functional carbon materials. The attempt has been made to carbonize pine needles into biochar through pyrolysis at different temperatures of 300°C, 400°C, 550°C and 700°C. Top Lit Updraft and Retort kiln method has also been explored to achieve large-scale conversion rate. The produced biochar is characterized for morphological and structural characteristics through CHNSO elemental analysis, SEM, TEM, FTIR, Raman, XRD, EDAX and XPS. The characterization results show that pine needle biochar produced through retort heating method provides good degree of carbonization and aromatisation with significant polarity index widening the versatile applications of the material. The biochar thus produced can be activated or functionalised to a range of significant functional carbon materials such as activated biochar, nano biochar, graphitized biochar etc. Further, an attempt has been made to graphitize the biochar through chemical activation using $Fe(NO_3)_3$ followed by thermal treatment. The result shows that biochar has been successfully converted into graphitized biochar at quite low temperature i.e. <1000°C. Graphitized biochar produced exhibits crystalline aromatic structure with well-organized layers as evident by SEM, FTIR, XRD and Raman studies, and can serve as a source to derive costly graphene-based materials. This adopted pathway of making biochar, thus exhibits the potential to solve the problem associated with accumulation of pine needle waste, it can serve as an alternative precursor for the synthesis of value-added synthetic functional carbon materials applicable to wide range of specific applications including adsorbent or catalysts for water remediation, CO_2 sequestration, filler for polymer composites, electrode materials, energy storage, etc.

1. TGA of pine needles

To understand the decomposition behaviour, decomposition rate and thermal stability including weight loss profile of Pine needles TGA analysis was performed. TGA of biomass (pine needles) provides the understanding of the chemical processes that take place during thermochemical degradation of biomass that could significantly be related with the processing parameters for producing biochar and final structural properties of the biochar. Thermogravimetric analysis (TGA) studies on Pine needles were carried out on a Shimadzu's TGA- 50 for thermal analysis at a heating rate of 10°C·min^{-1} under N_2 atmosphere in the temperature range starting from 15°C to 800°C.

Figure S1 represents the TGA of Pine needles that shows the initial loss of moisture at room temperature that continues till 180°C, with mass loss of approximately up to 12.81%. The temperature change from 180°C to 420°C is accompanied by maximum mass loss i.e. 84.38% and is ascribed to the degradation of cellulosic and hemi-cellulosic components which constitute the major fraction in the pine needles. The final weight loss of 13.40% could be observed in the temperature range of 420°C to 800°C. This decomposition occurs at slower rate as it involves the structural decomposition of the lignin and remaining cellulose content of the pine needle biomass. Actual lignin degradation occurs majorly after 500°C due to the rigid structural lattice presented within lignin that resisted thermal decomposition and led to its slow degradation [1].

2. Energy-dispersive X-ray analysis (EDAX)

Figure S2 shows EDAX graph combined with elemental mapping for CH_TLUD and CH_retort respectively. The elemental mapping shows presence of carbon, nitrogen, oxygen as the main elements along with potassium, calcium, magnesium and silica as minor elements on the surface of biochar [1–4]. CH_retort demonstrates very effective degree of carbonisation with increased carbon content compared to CH_TLUD. It can be seen that CH_retort has greater weight percentage of carbon as well as oxygen making it a potential substrate for various applications such as wastewater remediation, reinforcement in polymers, etc [3].

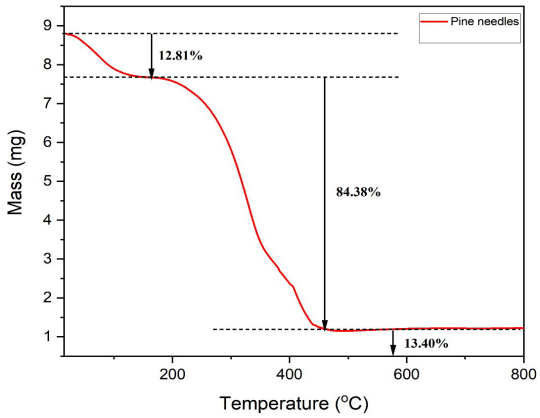


Figure S1. TGA of raw Pine needles.

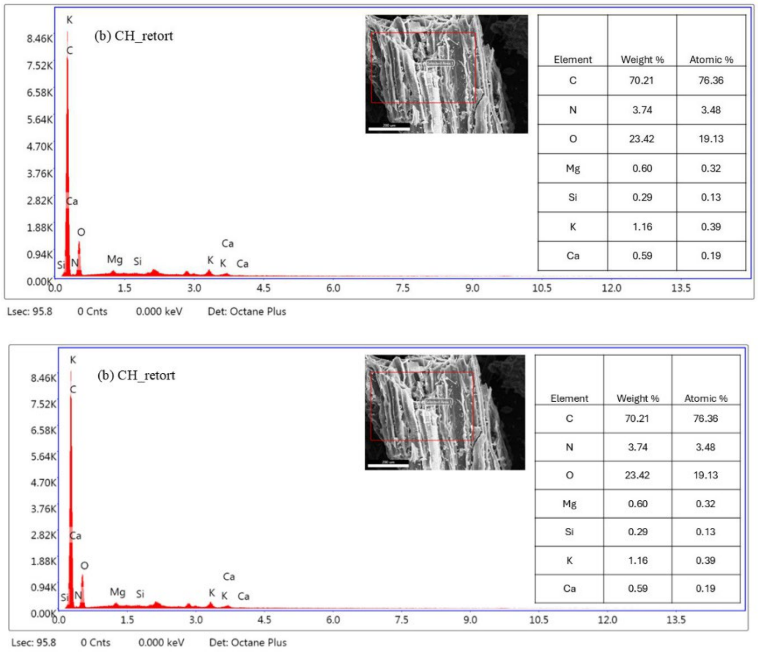


Figure S2. EDAX mapping of (a) CH_TLUD, and (b) CH_retort.

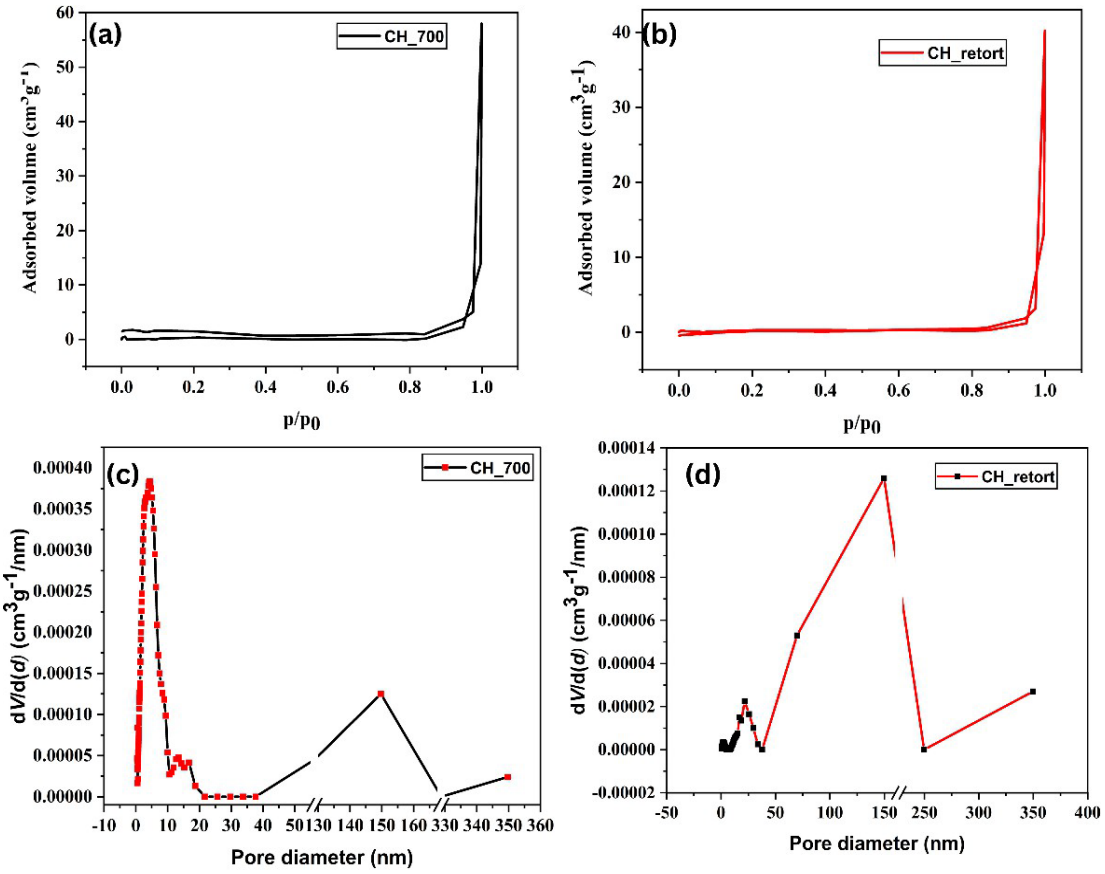


Figure S3. N_2 adsorption-desorption isotherm of (a) CH_700, (b) CH_retort and Pore size distribution of (c) CH_700, and (d) CH_retort.

3. Brunauer-Emmett-Teller (BET) analysis

The N_2 adsorption-desorption isotherms as depicted in Figure S3 (a–b) show low adsorbed volume with type II adsorption isotherm indicating biochar majorly consisting of macropores with limited development of micropores or mesopores in absence of any activator [5]. NL-DFT (Non-Local Density Functional Theory) provided the pore distribution curves as shown in Figure S3(b–c), that depicts average pore diameter as 86 nm for CH_retort and 30 nm for CH_700. The same can be correlated with the SEM image already provided in the Figure 3(i) of the manuscript.

This indicates that surface of CH_retort is dominated by large transport pores with minor contribution from smaller pores. The macropores in biochar are significant when used for water remediation, soil amendment materials and energy storage systems. The pore size can be altered by activating the biochar with suitable chemical or physical activating agents to produce mesopores and micropores [6–8].

CH_retort biochar with dominating macroporous structure offer structural advantages such as effective catalyst infiltration through the larger transport pores. The improved infiltration of catalyst such as $Fe(NO_3)_3$ is beneficial during catalyst impregnation and subsequent catalytic graphitisation at comparatively low temperature, making CH_retort a very good precursor material for the production of graphitised carbon. The macropores promote uniform catalytic distribution during impregnation and aids in efficient carbon reorganization during graphitization [9].

References

[1] R. Kumar, P. K. Rakesh, and D. Sreehari, "Investigation on physico-chemical, mechanical and thermal properties of extracted novel pinus roxburghii fiber," *Journal of Natural Fibers*, vol. 20, no. 1, Art. no. 2157924, 2023.

[2] M. A. Islam, M. I. Parvin, T. K. Dada, R. Kumar, and E. Antunes, "Silver adsorption on biochar produced from spent coffee grounds: Validation by kinetic and isothermal modelling," *Biomass Conversion and Biorefinery*, vol. 14, no. 22, pp. 28007–28021, 2024.

[3] I. Cakin, S. Pap, P. P. J. Gaffney, and M. A. Taggart, "Designing a biochar-based pretreatment method for distillery effluents entering constructed wetlands," *Journal of Cleaner Production*, vol. 476, p. 143790, 2024.

[4] P. Bulak, K. Proc-Pietrycha, M. Kaczor, K. Złotko, C. Polakowski, D. Wiącek, H. Waniak-Nowicka, E. Zieba, A. Waśko, P. Oleszczuk, and A. Bieganowski, "A novel type of biochar from chitinous Hermetia illucens waste with a built-in stimulating effect on plants and soil arthropods," *Scientific Reports*, vol. 13, no. 1, p. 8306, 2023.

[5] C. Sun, Z. Luo, P. Yu, and Q. Wang, "Comparative study on the performance and mechanism of adsorption–oriented phosphorus–modified high–efficiency and durable activated biochar from fast pyrolysis," *Energies (Basel).*, vol. 16, no. 14, p. 5363, 2023.

[6] Z. Xie, Y. Zhang, S. Zhang, H. Chen, C. Du, and C. Zhong,

"Preparation of S-doped biochar with sodium thiosulfate as activator and sulfur source and its highly efficient adsorption for Hg^{2+}," *Arabian Journal of Chemistry*, vol. 17, no. 10, p. 105955, 2024.

[7] Z. Han, D. Wang, J. Wu, Q. Xiong, Y. Zhang, and K. Zhang, "Study on the preparation of magnetic pine needle biochar and its adsorption performance for the dye," *Advances in Materials*, vol. 10, no. 4, p. 75, 2021.

[8] L. Zhu, N. Zhao, L. Tong, and Y. Lv, "Structural and adsorption characteristics of potassium carbonate activated biochar," *RSC Advances*, vol. 8, no. 37, pp. 21012–21019, 2018.

[9] A. Gomez-Martin, Z. Schnepp, and J. Ramirez-Rico, "Structural evolution in iron-catalyzed graphitization of hard carbons," *Chemistry of Materials*, vol. 33, no. 9, pp. 3087–3097, 2021.