The Effect of Hydrothermal Carbonization of Sewage Sludge on Microplastic behaviour

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Due to its persistence and widespread distribution, microplastic pollution is a growing environmental concern. These synthetic particles that are less than 5 mm in size can adsorb harmful chemicals and pose risks to ecosystems and public health through bioaccumulation. Wastewater treatment plants partially retain microplastics, but many accumulate in sewage sludge, often reused as agricultural biosolids (Worek et al., 2025). This raises concerns about secondary contamination, as microplastics can persist in soil and impact microbial communities, plant uptake, and water infiltration. Before reuse, advanced treatment strategies are needed to degrade or remove microplastics from biosolids. Hydrothermal Carbonization (HTC) is a promising thermochemical process that converts sewage sludge into valuable solid hydrochar while degrading pollutants (Prus et al., 2024; Xu, Z. & Bai, X., 2022). Moreover, the breakdown of plastic particles can improve potential applications of hydrochar, making it more valuable material for land and energy uses (Zhang, L. et al., 2022).

This study examines the effect of the HTC process on microplastic present in digested biosolids. Digested sewage sludge was collected from the Plaszow wastewater treatment plant in Krakow, Poland, and mixed with distilled water. HTC experiment was conducted using a 1 L steel Zipperclave Stirred Reactor equipped with a MagneDrive Agitator, supplied by Parker Autoclave Engineers, USA. Process parameters were selected as the maximum possible temperature of device (220 °C) and 4 h residence time, as longer residence time can enhance microplastic degradation, and maximum 3 h were investigated so far (Xu, Z. & Bai, X., 2022; Wang, Y. et al., 2022). The resulting hydrothermal slurry was collected. Next, it was freeze-dried to remove free water and moisture, and crushed in a mortar to homogenise. Microplastics were then extracted from 2 g of untreated biosolid and hydrochar samples by density separation using saturated CaCl₂ solution, chemically digested by 15% H₂O₂, and filtered out on GF-5 glass fibre microfilters to collect all polymer particles. Then, the samples were identified and visualized by microscopic and spectroscopic analyses to evaluate the impact of hydrothermal carbonization process on microplastic behaviour (Figure 1).



Figure 1. The methodology used for the study

Consequently, Raman microspectroscopy (WiTec Alpha 300 R confocal microscope with 532 nm excitation laser beam and UHRS 300 spectrometer) and Fourier-transform infrared spectroscopy (FTIR) technique (Bruker 400 FTIR-ATR spectrometer) were used to observe size differences and evaluate particle degradation, identifying oxygen-containing functional groups and changes in aromatic structures.

Furthermore, microscope observation of a randomly selected 10 mg subsample under a $10 \times \text{ air}$ lens confirmed that post-HTC microplastic particles are smaller and more widely distributed across the field (Fig. 2). Moreover, they are more brittle, as evidenced by the reduction in laser energy required for collecting Raman spectra. FTIR-ATR analysis has shown that hydration and oxygenation in the structure occurred (Fig. 3), which contributes to particle destabilisation.

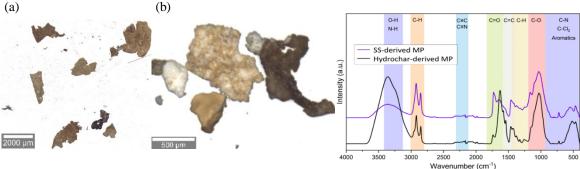


Fig. 2. Observed microplastic separated from digested sewage sludge (a) and hydrochar (b).

Fig. 3. FTIR spectra for separated microplastic

These findings have expanded the understanding of the fate of microplastic transformations during the HTC of sewage sludge. The research demonstrates that the HTC method can alter the chemical and structural properties of microplastics in biosolids, making them more fragile and susceptible to further degradation, decreasing the risk of secondary contamination from agricultural and land use of biosolids. Microscopic and spectroscopic analyses confirmed that particle sizes have been reduced with increased brittleness.

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References

Worek, J.; Kawoń, K.; Chwiej, J.; Berent, K.; Rego, R.; Styszko, K. Assessment of the Presence of Microplastics in Stabilized Sewage Sludge: Analysis Methods and Environmental Impact. Appl. Sci. 2025, 15, 1. https://doi.org/10.3390/app15010001

Prus, Z.; Styszko, K.; Wilk, M. The Influence of Hydrothermal Carbonization Process on Reduction of Heavy Metals and Polycyclic Aromatic Hydrocarbons from Sewage Sludge—A Short Review. In Proceedings of the European Young Engineers Conference, Warsaw, Poland, 15–17 April 2024; Available online: https://www.eyec.ichip.pw.edu.pl/wp-content/uploads/12th_EYEC_ Monograph_final.pdf (accessed on 27 February 2025)

Xu, Z.; Bai, X. Microplastic Degradation in Sewage Sludge by Hydrothermal Carbonization: Efficiency and Mechanisms. Chemosphere 2022, 297, 134203. https://doi.org/10.1016/j.chemosphere.2022.134203 Zhang, L.; Wang, Q.; Xu, F.; Wang, Z.; Zhang, G. Insights into the evolution of chemical structures in hydrochars from hydrothermal carbonization of PVC. J. Energy Inst. 2022, 105, 323–333. https://doi.org/10.1016/j.joei.2022.09.004

Wang, Y.; Yu, Y.; Huang, H.; Yu, C.; Fang, H.; Zhou, C.; Yin, X.; Chen, W.H.; Guo, X.C. Efficient conversion of sewage sludge into hydrochar by microwave-assisted hydrothermal carbonization. Sci. Total Environ. 2022, 803, 149864. https://doi.org/10.1016/j.scitotenv.2021.149874

Jiang, C.; Chen, Z.; Lu, B.; Li, Z.; Zhang, S.; Liu, Y.; Luo, G. Hydrothermal pretreatment reduced microplastics in sewage sludge as revealed by the combined micro-Fourier transform infrared (FTIR) and Raman imaging analysis. Chem. Eng. J. 2022, 450, 138163. https://doi.org/10.1016/j.cej.2022.138163

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