Hydrothermal carbonization of agriculture digestate: the effect of residence time and temperature on chemical and physical properties of hydrochar and process water

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Keywords: agricultural digestate, hydrothermal carbonization, hydrochar, process water.
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Agricultural biogas plants are becoming an increasingly popular alternative to non-renewable energy sources. In biogas plants, a methanogenic fermentation process occurs, involving microorganisms under the influence of appropriate technological parameters. The main product obtained is biogas, along with a by-product known as digestate. Agricultural digestate is a residue in the form of a liquid and solid mixture. It is rich in organic compounds, salts, and chemical elements. Currently, the primary method of its disposal is spreading it on agricultural fields. Due to the potential presence of heavy metals, pathogens, and contaminants, this can pose a serious threat to the natural environment and living organisms. One of the solutions for managing digestate is using it in the hydrothermal carbonization process (HTC) (Mikusińska et al., 2023) (Czerwińska et al., 2023).

In this research, digestate from the BUTOR agricultural biogas plant in Poland was used in the HTC process. The digestate was a mixture of agriculture waste, manure and coffee spent grains. The feedstock did not need to be dried beforehand because its high moisture content is required in HTC. The parameters of the process were: 0.5, 1, 1.5 and 2 h residence time and temperature 190, 200 and 210°C under autogenous pressure. As a result, mixture of liquid and solid phases and small amounts of gaseous product were obtained. The post-reaction mixture was separated into solid and liquid products. Hydrochar was dried and ground, and then FTIR and SEM analyses were carried out. The hydrochars were also examined for toxicity, which was performed in the Microtox Model 500 analyzer in accordance with the ASTM D-5560 standard. As the indicator organisms *Allivibrio fischeri*, *Daphnia magna* and *Lemna minor* were used (Jagodzińska et al., 2019). The process water was tested for the content of nitrogen, phosphorus, ammonia, phenol, COD and TOC. The analysis were calculated using Merck spectrophotometer. Additionally, pH and conductivity were measured. The process water and post HTC sludge were tested in biomethane potential analysis, which was measured in the AMPTS II analyzer (Lehtomäki et al. 2007) (Lombardi et al., 2024).

The analyses allowed to assess the possibility of using process water as a fertilizer in the agricultural industry and to examine the morphology and surface of the produced hydrochars. SEM analysis confirmed that the HTC process affects the grain size of the hydrochars structure (Fig. 1). As a result, a material with a finer structure than the raw digestate is produced. The toxicity analysis showed no toxicity for all hydrochars to the growth indicator with the aquatic plant *Lemna minor*. Process water produced at 210°C and 2 h has the highest ammonium and phenol contents. The highest phosphorus content was obtained for process water produced at 200°C and 0.5 and 1 h. For the same parameters, the highest COD values were also obtained. The highest nitrogen content was measured in process water produced at 200°C and 210°C and both residence time 1 h. pH values for all samples were approximately 4 - 5 and the conductivity was 5 - 6 mS/cm. The best biomethane potential was obtained for sludge produced at 200°C and 2 h (Fig.2).

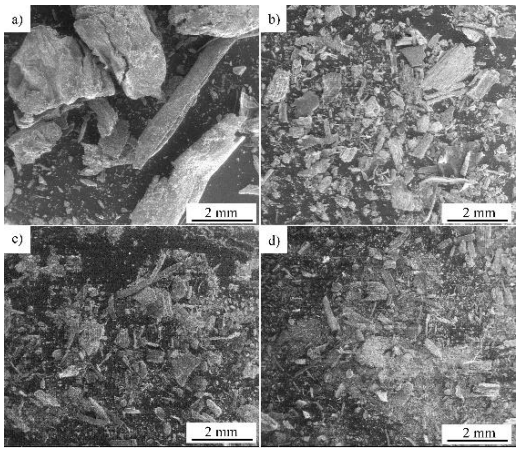

Fig. 1 SEM results: a) raw digestate b) HTC 190/2.0 c) HTC 200/2.0 d) HTC 210/2.0

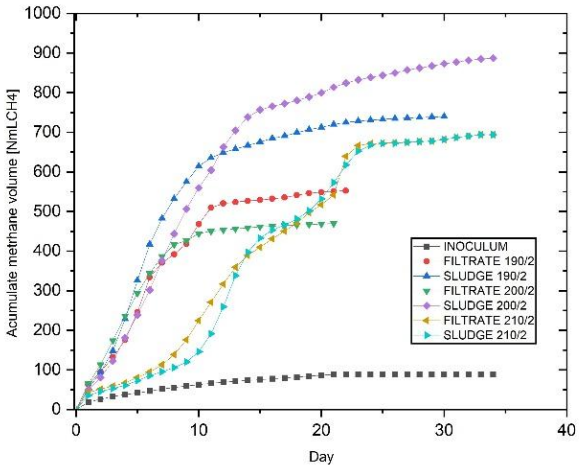

Fig. 2 Biomethane potential results

The conducted research has enriched the knowledge regarding the use of the HTC method in the disposal of waste from biogas plants. The products obtained in this process can be used in various industries. The properties of products obtained in the HTC process are influenced by the residence time and temperature of the process. The conducted research shows that higher temperature improves the fuel properties of the obtained hydrochars.

Acknowledgements

This research project was supported by the programme "Excellence initiative – research university" for the AGH University of Krakow, Poland [project no. 501.696.7996].

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