Hydrothermal Liquefaction of Municipal Sludge from Biological Nutrient Removal Plant: A Process for the Circular Economy

Farid Alizad Oghyanous[1], Cigdem Eskicioglu*[1,2,3]

[1]UBC Bioreactor Technology Group, School of Engineering, The University of British Columbia, Okanagan Campus, 3333 University Way, Kelowna, BC V1V 1V7, Canada.
[2]ICREA – Catalan Institution for Research and Advanced Studies, Pg. Lluís Companys 23, Barcelona, Spain.
[3]*Current address:* GEMMA – Department of Civil and Environmental Engineering, Universitat Politècnica de Catalunya – Barcelona Tech., c/Jordi Girona 1-3, Building D1, E-08034 Barcelona, Spain.
Keywords: hydrothermal liquefaction, municipal sludge, waste-to-energy, biocrude oil, P recovery.
*Presenting author email: cigdem.eskicioglu@ubc.ca.

ABSTRACT

Municipal sludge, generated from the primary and secondary (biological) treatment processes at wastewater treatment plants (WWTPs), contains approximately 60% of the influent wastewater's chemical oxygen demand (COD). Efficient management of municipal sludge should prioritize energy and nutrient recovery, while mitigating hazardous compounds. Hydrothermal liquefaction (HTL) converts sludge into value-added products under high temperature and pressure within the retention time of minutes. This study evaluated, for the first time, converting biological nutrient removal (BNR) plant sludge into bio-crude oil, hydrochar, and aqueous products under varying HTL temperatures (300-368°C) and reaction times (14.9-16.9 min). Within the conditions studied, the highest energy recovery (ER) was obtained at 350°C and 15 min with a distribution of 67%, 28%, and 6% in bio-crude, aqueous/gas, and hydrochar, respectively, demonstrating the viability of BNR sludge as an HTL feedstock. Additionally, the hydrochar retained 94% of the phosphorus under the same conditions, highlighting its potential as a fertilizer. The aqueous phase accumulated significant amount of soluble COD with biogas potential.

Introduction

Conventional WWTPs use clarifiers to remove solids and the activated sludge process for organic carbon removal. While conventional WWTPs meet earlier, less stringent effluent regulations, growing awareness of pollutants' environmental and health impacts have led to stricter wastewater treatment standards. The Modified Bardenpho is a tertiary wastewater treatment process that biologically removes nitrogen, phosphorus, and carbon through anaerobic, anoxic, and aerobic zones, minimizing the chemical use for phosphorous removal and producing low-nutrient and low-carbon effluent. Municipal sludge, rich in organics and nutrients, is considered a valuable energy source. While anaerobic digestion (AD) of sludge, as a common stabilization method, recovers energy as biogas, HTL offers faster sludge conversion to multiple value-added products, such as bio-crude oil, hydrochar, and an aqueous phase with biogas recovery potential in reactors with small footprint. It also reduces biosolids (stabilized sludge) volume significantly and enhances pathogen and micropollutant removal (Abeyratne, 2023). HTL is a newer technology for municipal sludge treatment, with early studies mainly targeting sludge from conventional WWTPs that focus on carbon removal (Liu et al., 2022; Basar et al., 2024). To the best of our knowledge, no study has evaluated HTL for converting BNR-derived municipal sludge into bio-crude oil. This study evaluated the HTL feasibility of BNR sludge at various temperatures and reaction times and characterized all products (biocrude, hydrochar, gas, and aqueous phase). The study also analysed ER and phosphorus (P) distribution among all products under different HTL conditions.

Materials and methods

BNR sludge was collected from a regional WWTP (British Columbia, Canada), where wastewater undergoes preliminary treatment, primary clarification, and a modified Bardenpho BNR process. At the plant, the gravity-thickened primary sludge is mixed with thickened waste activated sludge in a 40:60 vl.% ratio and dewatered via centrifugation before sent for composting offsite. Compared to primary or secondary sludge, mixed sludge has a more balanced composition of protein, carbohydrates, and lipids, enhancing biocrude oil production (Basar et al., 2024). Therefore, dewatered mixed sludge (total solids: 17.89 ± 0.1 wt.%, volatile solids: 15.5 ± 0.2 wt.%) served as the HTL feedstock. The feedstock had 38.7 ± 0.1 wt.% proteins (dry basis, db), 11.3 ± 2.0 wt.% carbohydrates (db), 6.6 ± 0.4 wt.% lipids (db), 13.9 ± 0.9 wt.% ash (db), and 21,970 ± 114 mg/kg P (db).

In each HTL run, 576.84 g of dewatered mixed sludge was added to a Parr reactor (total volume of 1-L) with mixing and heating modules. The reactor was purged with nitrogen to create an oxygen-free environment, then heated to the selected temperatures (300–368°C) and kept at the target temperature for the selected retention time (14.9–16.9 min). The HTL temperature and retention conditions were selected based on the previous research conducted on municipal sludge from conventional WWTPs (Liu et al., 2022). Once cooled, the aqueous phase was separated by filtration, while bio-crude oil and hydrochar were separated using dichloromethane solvent. ER was assessed by calculating the higher heating values (HHV) of bio-crude oil and hydrochar using elemental analysis with a Costech ECS 4010 automatic elemental analyser. All HTL runs were conducted in duplicate, with ER and yield calculations reported on a dry mass basis (db). For P recovery, the BNR plant sludge and its HTL products, except for the gas phase, were digested using a high temperature/pressure microwave system and analysed with an Agilent 8900 Triple Quad inductively coupled plasma mass spectrometer (ICP-MS).

Results and discussion

The yields of bio-crude oil, hydrochar, aqueous phase, and gas from the BNR plant mixed sludge are summarized in Table 1. Increasing the HTL temperature up to 350°C positively impacts the bio-crude oil yield, with the maximum yield of 39.1 wt.% (db) achieved at 350°C for 15 min. Beyond 350°C, gasification of organic carbon becomes dominant, with gas production increasing from 10.9 wt.% (db) at 350°C for 15 min to 12.7 wt.% (db) at 368°C for 14.9 min, which in return reduces the bio-crude oil yield.

Table 1. HTL product yields from the BNR plant mixed sludge in dry basis at different HTL process severities.

Run	Reaction temperature (°C)	Retention time (min)	Bio-crude oil (wt.%, db)	Hydrochar (wt.%, db)	Aqueous phase (wt.%, db)	Gas (wt.%, db)
1	300	15	34.3±0.1	15.3±0.2	42.6±0.2	7.6±0.1
2	315	16	36.6±0.1	14.3±0.1	39.6±0.1	9.3±0.1
3	332	16.9	38.1±0.1	14.2±0.1	37.3±0.1	10.2±0.1
4	350	15	39.1±0.1	13.6±0.1	36.4±0.1	10.9±0.1
5	368	14.9	36.7±0.1	13.3±0.1	37±0.1	12.7±0.1

Figure 1(a) shows ER in HTL products from the BNR plant mixed sludge under different HTL operational conditions. The highest ER, 66.6%, was achieved from bio-crude oil at 350°C for 15 min. Compared to mixed sludge from a dual-stage biological treatment plant (trickling filter + solid contact tank) that achieved 70.8% ER in bio-crude oil at 332°C for 16.9 min (Liu et al., 2022), the BNR plant sludge had lower bio-crude-associated ER. Compared to primary sludge derived biocrude, the ER was comparable. Biller et al. (2018) reported 66.8% ER for bio-crude oil from primary sludge at 340°C for 20 min. In terms of nutrient recovery, over 93% of P in the BNR plant sludge was recovered in hydrochar, with its concentration rising from 83,200 ± 189 mg/kg at 300°C to 121,218 ± 158 mg/kg at 368°C, making hydrochar a valuable fertilizer (Figure 1(b)). Hydrochar P levels from the BNR plant were higher than that of conventional WWTPs (Liu et al., 2022).

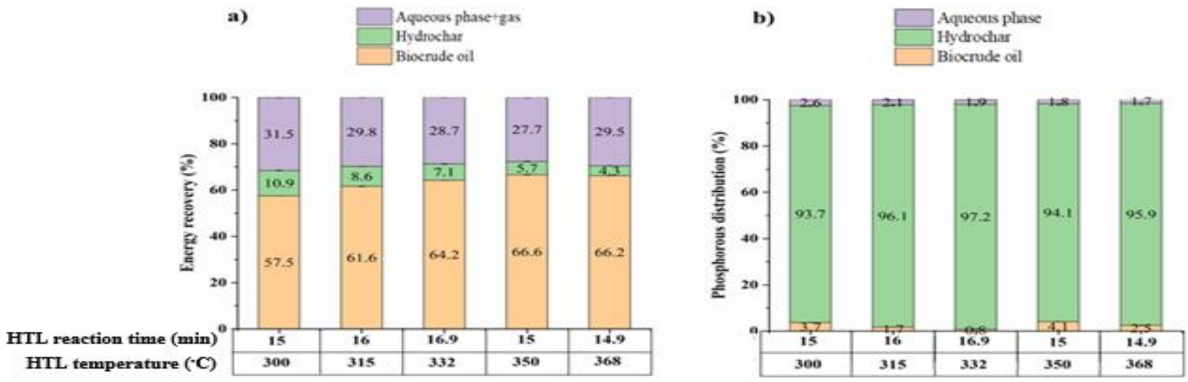

Figure 1. (a) Energy and (b) phosphorus distribution among HTL products derived from a BNR plant mixed sludge at various HTL temperatures and retention times.

Conclusion

This study evaluated the potential of the HTL process for converting the BNR plant sludge into value-added products. The results indicated that up to 39.1 wt.% (db) bio-crude oil can be obtained from BNR sludge, with an energy recovery of 66.6%, making it a viable feedstock for the HTL process. The high energy recovery in bio-crude oil further underscores the feasibility of applying HTL to BNR sludge. Additionally, the elevated phosphorus concentration in the hydrochar phase suggests its potential as an effective fertilizer. The HTL aqueous phase also represents a potential for downstream valorisation for biogas generation. Thermophilic and mesophilic AD experiments for the aqueous streams obtained at different HTL temperature & retention time are ongoing.

References

- Abeyratne, et al. (2023). "Multi-criteria evaluation of energy recovery from urban wastewater sludges by anaerobic digestion and hydrothermal liquefaction." J.Environ. Chem. Eng. **11**(2): 109628.
- Basar, et al. (2024). "Effects of municipal sludge composition on hydrothermal liquefaction products: Aqueous phase characterization and biodegradability assessment." Bioresour. Technol. **400**: 130671.
- Biller, et al. (2018). "Primary sewage sludge filtration using biomass filter aids and subsequent hydrothermal co-liquefaction." Water Research **130**: 58-68.
- Liu, et al. (2022). "Incorporating hydrothermal liquefaction into wastewater treatment–Part I: Process optimization for energy recovery and evaluation of product distribution." Chem. Eng. J. **449**: 137838.