Pilot Scale Freeze Concentration of HTC Process Water

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Abstract

High-moisture residues (>50%), including sewage sludge, food processing byproducts, and wet agricultural wastes, represent an underutilized resource for sustainable energy and material recovery. Hydrothermal carbonization (HTC) enables their efficient conversion into hydrochar and process water (PW), but the latter remains difficult to treat due to its diluted yet complex composition.

This study evaluates freeze concentration (FC) at pilot scale as a novel treatment for HTC process water (Fig. 1), building on previous laboratory-scale experiments demonstrating >90% solute recovery (Gerner et al., 2023). PW collected from a continuous HTC system was treated using FC after an aging step during storage. This storage-induced aging improved phase behavior and enabled stable operation. Results showed ~99% removal of non-purgeable organic carbon (NPOC), total nitrogen (TN), and phenolic compounds, with most analytes below detection limits. At the same time, valuable carboxylic acids (1–11 g/L) were fully retained in the concentrate. The purified water exhibited a very low residual load with negligible biodegradability. Overall, FC represents a robust approach for simultaneous pollutant removal and resource recovery, improving the sustainability of HTC processes.

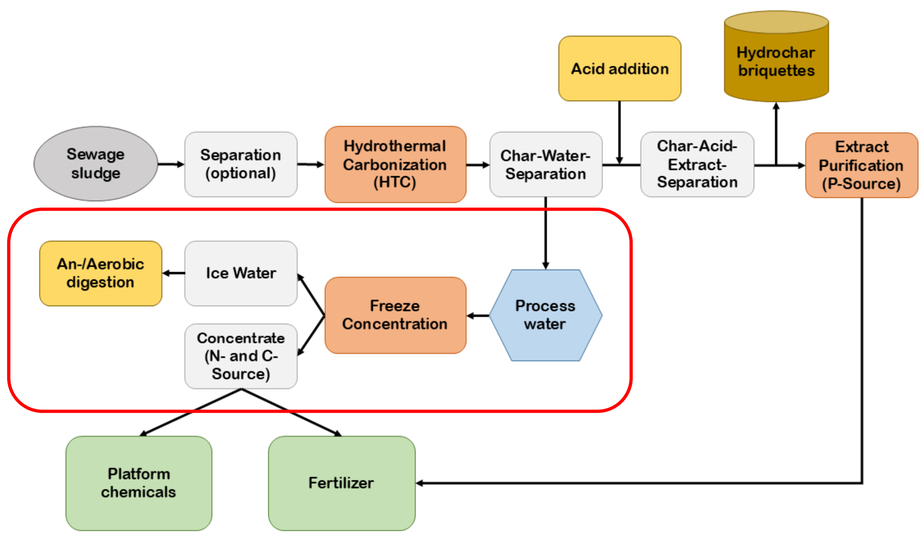

Figure 1: Process flow diagram of a hydrothermal carbonization (HTC) plant including freeze concentration for process water (PW) treatment.

Project goals

- Demonstrate feasibility of freeze concentration at pilot scale (Fig. 2)
- Investigate separation performance for HTC process water
- Characterize purified water and concentrate fractions
- Assess recovery of valuable organic compounds
- Test biodegradability of treated water

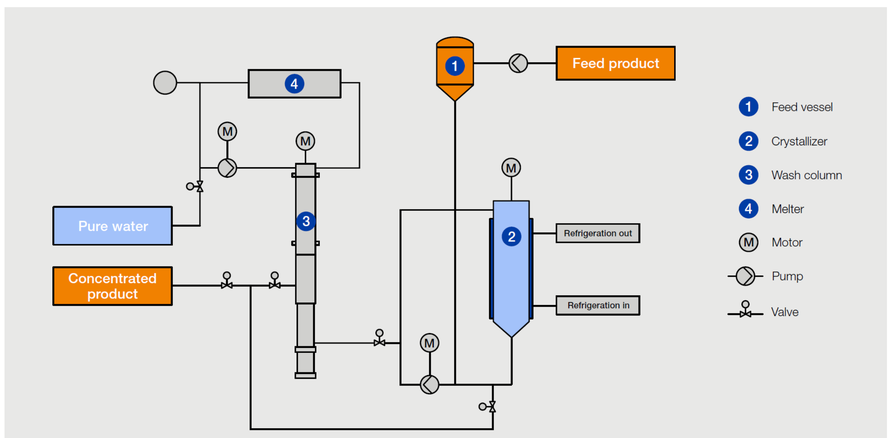

Figure 2: Process flow diagram of the pilot-scale freeze concentration system for process water treatment (From Sulzer Chemtech AG)

Results & Discussion

Process performance

Solid particles and foaming caused failure of the initial FC trial. During subsequent storage (~3 months), aging promoted sedimentation of solids and altered PW properties, enabling stable and successful operation.

A following trial was only partially successful due to lower-than-expected impurity concentrations, which led to operational challenges such as layer formation. Increasing solute concentration (e.g., by salt addition) improved process stability. In the final test, high recovery and purification of PW were achieved, and the process could be operated reproducibly.

Water purification efficiency

- ~99% removal of NPOC, TN, and phenolic compounds
- Residual concentrations: NPOC: 0.9 ± 0.24 mg/L; TN: 21.4 ± 0.93 mg/L; phenolic compounds: ~0.1 mg/L
- Most target compounds below detection limits

Resource recovery and additional findings

- Quantitative transfer of volatile fatty acids (C2–C8) into the concentrate
- High concentrations achieved (1–11 g/L) (Fig. 3A)
  - Includes acetic, propionic, butyric, heptanoic, and octanoic acids
- Strong potential for valorization as platform chemicals
- Minimal transfer of volatile compounds to the purified water
- High-quality effluent with very low contaminant levels
- Clear visual improvement (strong decolorization of process water) (Fig. 3B)

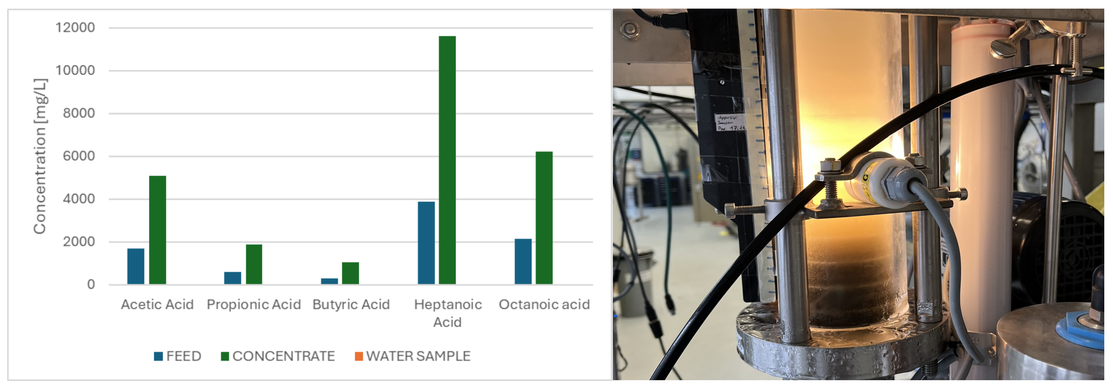

Figure 3: (A) Distribution of volatile fatty acids between feed, concentrate, and purified water. (B) Clean ice disks after separation (Photo from Dal Pos Emanuele).

Conclusions & Outlook

Freeze concentration enables highly efficient removal of dissolved contaminants from HTC process water, producing a clean water stream with very low residual load while concentrating valuable organic compounds in a recoverable fraction. Compared to previous laboratory-scale studies reporting >90% solute recovery, the pilot-scale results demonstrate improved separation performance (~99% removal) and confirm the robustness of the technology under realistic operating conditions. The storage (“aging”) step proved critical for process stability, and overall, FC shows clear advantages over conventional treatment approaches in terms of selectivity and operational reliability.

Further work should address the influence of feedstock variability on process water composition and FC performance, while exploring opportunities for targeted recovery and valorization of specific compounds.

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