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Hydrothermal carbonization of digested sewage sludge and microalgae biomass

Phosphorus and energy recovery

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Akademisk avhandling

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Abstract
Sewage sludge and microalgae biomass are by-products of wastewater treatment, requiring careful management to avoid environmental and health risks. Both sewage sludge and microalgae have high moisture content, making thermochemical conversion challenging and energy intensive. Hydrothermal carbonization (HTC) presents a promising solution for converting these wet feedstocks into valuable resources. The thesis aimed to study HTC of sewage sludge and microalgae biomass, individually and combined (i.e., co-HTC). It focused on process parameters, mixing ratios, product characteristics, primary and secondary char formation, and resource recovery, with especial emphasis was on phosphorus and energy recovery as potential applications of the resulting hydrochars. Both the HTC and co-HTC experiments were conducted at 180, 215 and 250°C for 2 h (**Papers I**–**IV**).

Paper I investigated co-HTC by combining microalgae and sewage sludge in various ratios, from 0 to 100% of sewage sludge. Results showed that higher sewage sludge proportions and carbonization temperatures led to lower degradation and carbonization rates. The addition of sewage sludge influenced secondary char formation and composition, reducing carboxylic acid and ketones while increasing higher molecular weight cholesterols. Moreover, sewage sludge hydrochars contained larger phosphorus quantities.

In acid-leaching experiments (**Papers II** and **III**) using sewage sludge, phosphorus-extraction efficiencies surpassed 75%. Complete phosphorus recovery (100%) was achieved only with oxalate extraction at pH=1. Organic acids, utilized at a lower concentration (0.25 M) compared to mineral acids (2.5 M), acted as both acids and chelating agents, facilitating phosphorus recovery. Regardless of acid type, leaching from hydrochar transferred not only phosphorus but metals and heavy metals into the P-rich leachate, requiring post-treatment purification.

Combustion studies of microalgae and sewage sludge co-hydrochar, and phosphorus extracted hydrochar from sewage sludge as solid fuels showed notable improvements in physicochemical and energy-related properties. Acid treatment improved carbon content, heating values, and fuel ratio, while significantly reducing ash content compared to untreated hydrochars (**Paper IV**). These properties decreased in the co-hydrochars with higher sewage sludge proportions due to differing carbon, volatile matter, and ash content between microalgae and sewage sludge. Thermodynamic equilibrium calculations predicted liquid slag and solid phase formation at combustion temperatures up to 1200°C. Experimental comparison with combustion ashes, analyzed through DRIFTS, SEM-EDS, and XRD, validated simulated compounds including Fe_2O_3, SiO_2, feldspar, whitlockite, $CaCO_3$, and $CaSO_4$ in **Paper IV**. Notably, $CaCO_3$ presence in ashes was confirmed by XRD but not reflected in calculation results. Microalgae hydrochar ashes were primarily composed of calcium phosphates and Fe_2O_3, visually confirmed by EDS mapping due to XRD limitations.

The result of this thesis suggests that the HTC process offers a pre-treatment means of improving hydrophobicity and significantly reducing feedstock volumes. Additionally, the resource-recovery approach studied in this thesis, which uses sewage sludge and microalgae-derived hydrochars generated in wastewater treatment plants, is a step towards being an efficient management strategy for by-products generated by these plants.

Keywords
Wastewater treatment plant by-products, HTC, resource recovery, hydrochar formation, primary char, secondary char, inorganic matter distribution, acid leaching, mineral acid, organic acid, thermal analysis, combustion, thermodynamic calculations.

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