Supporting information: Development of a continuous hydrothermal treatment process for efficient dewatering of industrial wastewater sludge

Douwe S. Zijlstra [1], Esther Cobussen-Pool [1], Dennis J. Slort [1], Mark Visser [1], Pavlina Nanou [1,2], Jan R. Pels [1,2] and Heather E. Wray [1]*

This supplementary information provides information about specific challenges and issues encountered with the scale-up of the hydrothermal process for paper sludge from batch lab scale to continuous pilot scale.

The long-duration test with biological paper sludge (bio-sludge) demonstrated that it is feasible to perform continuous flow hydrothermal treatment/carbonization on bio-sludge for a prolonged period at pilot scale. However, during the long-duration experiment two practical issues arose. During the long-duration testing it was found that the mixing within the pilot plant was inadequate, likely causing temperature differences and uneven heat distribution and treatment of the sludge. Therefore, baffles were introduced into the pilot plant. In particular, the baffles were designed to create turbulent flow on the bottom-side of the tube and prevent settling of sludge solids. However, during the initial stages of the long-duration test, blockages were unexpectedly occurring on the top-side of the tubes (Figure S1, left). Subsequent laboratory analysis of the behavior of the bio-sludge at elevated temperatures showed that at 60 °C and higher a layer of solids separates and floats on top of the liquid layer (Figure 1, right). Therefore, the baffles were re-designed and modified to prevent the accumulation of solids in the pilot plant at both the top and bottom part of the tubes.

Figure S1: Build-up and blockages of sludge in the reactor (left) and unexpected phase separation of floating sludge upon heating of bio-sludge (right) to explain the blockages at the top of the reactor tubes.

At the end of the long-duration experiment fouling inside the reactor was discovered upon cleaning and inspection of the pilot plant (Figure S2). This fouling was not present during cleaning and inspection of the reactor in the early and mid-stages of the long-duration experiment. Higher concentrations of calcium, aluminum and silicon and a 3-fold increase of phosphorus present in the feedstock at the end of the test compared to the early stages are hypothesized to be the likely the sources of the fouling, combined with periods of operation at lower temperatures at night, when no

operator was present to make adjustments. The scaling also coincided with a decrease in the dry matter content of the obtained effluent, representing a drop in the Torwash effluent quality.

Figure S2: Fouling on the inside of the Torwash pilot plant upon inspection and cleaning at the end of the long-duration tests.

This information highlights the variability in the quality and characteristics of biological paper sludge and the need for a flexible and robust hydrothermal treatment process to adjust to changes in the feedstock and to produce consistent solids quality and dewaterability.

Table S1. Results for the microclave and autoclave experiments at different reaction temperatures on bio-sludge and mixed sludge as feedstock. n.d.: not determined due to low quantity of solids

Reaction temperature (°C)	**Bio-sludge**				**Mixed sludge**			
	microclave		**autoclave**		**microclave**		**autoclave**	
	Solid yield (%)	Dry matter content (%)	Solid yield (%)	Dry matter content (%)	Solid yield (%)	Dry matter content (%)	Solid yield (%)	Dry matter content (%)
160	-	-	-	-	83	59	-	-
170	42	n.d.	-	-	77	63	-	-
180	38	n.d.	-	-	71	61	-	-
190	26	n.d.	30	66	69	62	61	61
200	17	n.d.	-	-	56	54	-	-
210	10	n.d.	-	-	57	54	-	-