Effects of thermal hydrolysis on sludge from Kraft pulp mill wastewater treatment

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Abstract: Thermal hydrolysis (TH) is a widely used technology to enhance sludge anaerobic biodegradability. However, it has not been applied to biological sludge from industrial wastewater treatment. In the present work, TH was applied to the sludge from the aerobic treatment of wastewater generated by a Kraft pulp mill industry. Microscopic observations of the sludge with and without TH were made. Response surfaces are presented for the different variables tested (soluble Chemical Oxygen Demand (CODs), Volatile Suspended Solids to Suspended Solids ratio (VSS/VS), and soluble Total Kjeldhal Nitrogen (TKNs). Increases of 600% for CODs and TKNs concentrations were obtained. Reductions were observed for the VSS/VS ratio values from 23% up to 68%.

Keywords: Nutrient's release; Response surface methodology; Thermal hydrolysis

Introduction

TH has physical and chemical effects on organic matter, improving its biodegradability. Physical effects include cellular lysis and flocs disintegration. When flocs rupture occurs, there is a release of extracellular and cellular materials, and the subsequent organic matter solubilization (Prorot et al. 2011; Barber 2016; Bougrier et al. 2008). On the other hand, undesired reactions may occur, leading to recalcitrant compounds. There is a lot of information about TH of sewage sludge, however, there are few references on TH for aerobic sludge from treatment systems of industrial wastewater. Therefore, the goal of the present work was to study the effect of TH on sludge from pulp mill industries. Microscopic observation and changes after the TH in the organic matter and nutrients are presented.

Material and Methods

Sludge from the aerobic wastewater system of a Kraft pulp mill was used for TH. It was performed in a 2 L sealed batch reactor. The temperature and time conditions tested were defined by an experimental plan (Table 1).

The variables measured were: CODs, VSS, orthophosphate, ammoniacal nitrogen and TKNs, according to the Standard Methods protocols (APHA, 2012). CODs and TKNs concentrations were measured after using a centrifuge for 15 min at 6500 rpm. The percentage of TKNs (%TKNs) was calculated using the ratio between TKNs and TKN. Microscopic observations were performed on sludges with TH for 30 min at 165 °C, 205 °C, and without HT. Samples were stained with safranin and x100 amplification was used.

The response surfaces were generated using RStudio *4.0.2 software*. CODs, VSS, VS, TKNs and TKN analysis were performed by triplicate.

Results and Conclusions

Results presented in Table 1 show a significant increase in the measured values as a consequence of the TH, except for VSS that decreased. This is explained by the organic matter and the Nitrogen and Phosphorus release.

Table 1 TH conditions, organic matter solubilization and nutrients release results with the corresponding standard deviation (σ).

Experimental Factors of Thermal Hydrolysis		Organic Matter		Nitrogen			Orthophosphate
T (°C)	t (min)	CODs (σ) (mgCOD/L)	VSS/VS (σ) (%)	NH_4^+ (σ) (ppm NH_4^+-N)	TKN_s (σ) (ppm TKN_s-N)	% TKN_s (σ)	PO_4^{3-} (σ) (ppm PO_4^{3-}-P)
Untreatead	Untreatead	330 (18)	95 (2)	1.6 (0.3)	20.7 (1.5)	5.1 (0.4)	0.86 (0.03)
145	15	3850 (56)	57 (1)	33.5 (1.0)	225.2 (0.6)	47.8 (0.1)	1.01 (0.01)
145	45	4372 (73)	56 (11)	38.4 (3.6)	239.2 (9.7)	56.3 (0.2)	1.28 (0.04)
185	15	5161 (237)	45 (4)	56.0 (1.1)	326.1 (1.5)	73.1 (0.3)	3.20 (0.06)
185	45	5635 (425)	25 (8)	73.7 (0.6)	346.7 (0.8)	77.9 (0.2)	4.11 (0.04)
125	30	2405 (130)	73 (2)	25.1 (0.5)	191.3 (4.6)	41.2 (1.0)	0.52 (0.01)
165	30	5003 (76)	40 (6)	46.1 (1.4)	316.4 (32.0)	75.4 (7.6)	2.29 (0.01)
205	30	6206 (210)	31 (1)	106.2 (1.1)	396.2 (9.7)	87.9 (2.1)	4.69 (0.01)

In Figure 1, VSS/VS *vs* CODs data is presented for each TH condition, including untreated sludge data. A linear correlation was found between both variables, verifying the organic matter solubilization after the TH.

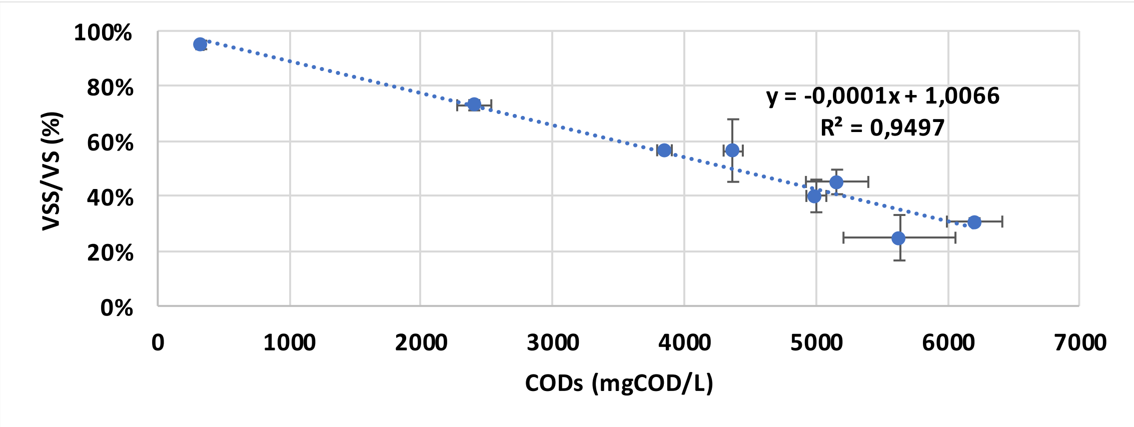

Figure 1 Correlation between VSS/VS and CODs.

Microscopic observations (Figure 2) showed a significant rupture of the flocs at 165 ºC. At 205 ºC a regrouping of the sludge was observed, probably due to caramelization reactions.

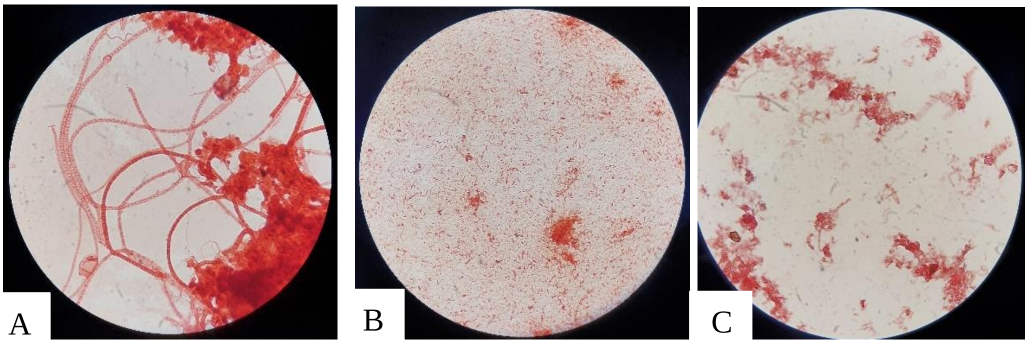

Figure 2 Microscopic observations of the untreated sample (A), TH at 165 °C for 30 min (B) and TH at 205 °C for 30 min (C), x100 objective.

Figure 3 shows the correlation between the Nitrogen compounds and the solubilization of the organic matter. Nevertheless, the amount of solubilized Nitrogen is lower than expected if the sludge composition were due to microorganisms alone. This fact could be explained by the presence of cellulosic material from the industry present in the untreated sludge.

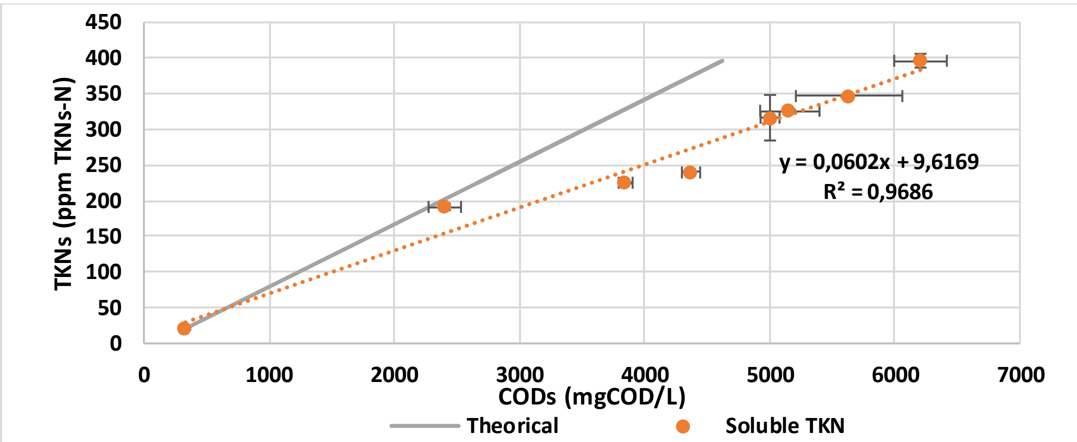

Figure 3 Correlation between TKNs and CODs; in continuous trace the theoretical result if sludge was composed only by microorganisms ($C_5H_7O_2N$).

Two-dimensional contour plots and response surfaces for COD, VSS/VS, and %TKN are presented in Figure 4. Mathematical models are presented in Table 2, *T* and *t* are temperature in ºC and time in minutes, respectively, with a proper fit and statistically significance ($p < 0.05$). In the case of CODs and VSS/VS the parameters with statistical significance are temperature and its square. On the other hand, all the parameters are statistically significant for the %TKN, except the interaction between *T* and *t*. The optimum value for CODs is at 216 ºC and 52 min, which is close to the optimum for %TKN (208 ºC and 31 min). This is in accordance with the correlation between these variables.

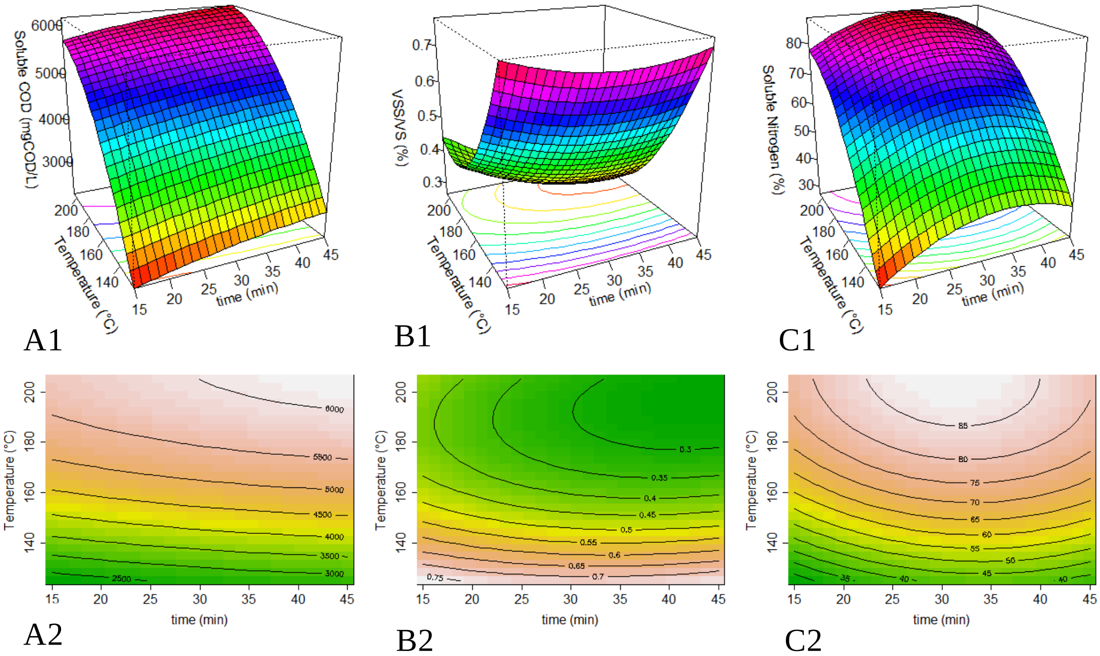

Figure 4 Response surfaces (upper graphs) and two-dimensional contour plots (lower graphs) for CODs (A1 and A2), VSS/VS (B1 and B2) and %TKNs (C1 and C2) responses.

Table 2 Empirical models determined and adjustments for each evaluated response.

Responses	Results for the models	Adjusts Results		
		$P-value$	r^2_{Adjust}	r^2
$COD_s\left(\frac{mgCOD}{L}\right)$	$-14871 + 43.11\,t + 187.64\,T$ $-0.04\,t\,T - 0.33\,t^2 - 0.44\,T^2$	<0.05	0.937	0.953
$VSS/VS(\%)$	$3.6921 - 0.00361\,t - 0.03401\,T$ $-0.00006\,t\,T + 0.00018\,t^2 + 0.00009\,T^2$	<0.05	0.969	0.979
$\%\,TKN_s$	$-263.78 + 3.128\,t + 2.918\,T$ $-0.003\,t\,T - 0.040\,t^2 - 0.007\,T^2$	<0.05	0.985	0.991

TH allowed the solubilization of the organic matter, with an increase up to 1780% in soluble COD. Then, the organic matter would be available in the liquid phase. A possible treatment system could be a separation step for the solid and liquid phases, followed by the treatment of the liquid stream in a high-rate anaerobic reactor. Additionally, the released nutrients can be used in accordance with a circular economy approach.

References

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