# Characterization of cellulose biodegradation kinetics in wastewater in view of increasing a WRRF's capacity by a pre-treatment sieve step

Elena Torfs<sup>1,2</sup>, Julie Doucet<sup>1,2</sup>, Domenico Santoro<sup>3</sup>, Dang Ho<sup>3</sup>, Medhavi Gupta<sup>4</sup>, George Nakhla<sup>3</sup>, Peter A. Vanrolleghem<sup>1,2</sup>

<sup>1</sup>modelEAU, Département de génie civil et de génie des eaux, Université Laval, Québec,

Québec, G1V 0A6, Canada

2 CentrEau, Centre de recherche sur l'eau, Université Laval, 1065 avenue de la Médecine,

Québec, QC, G1V 0A6, Canada

<sup>2</sup>Trojan Technologies, London, Ontario, N5V 4T7, Canada

<sup>3</sup>Department of Chemical and Biochemical Engineering, Western University, London,

Ontario, N6A 5B9, Canada

# Introduction

Increased population pressure in urban areas and the associated increase in hydraulic and organic loads causes water resource recovery facilities (WRRF) to be pushed closer to their design capacities. A well-designed pre-treatment step can alleviate this pressure. However, retrofitting an existing plant with a conventional pre-treatment such as a primary settling tank is not easily done as the space requirements and capital investments are large. Rotating Belt Filters (RBFs) represent an innovative small footprint alternative with a lower capital cost and relative ease of installation. An RBF is a sieve-based pre-treatment that uses filtration to retain suspended solids in wastewater based on their particle size. By engineering the sieving process, through sieve pore size selection and optimization, there is an opportunity to target different COD fractions (Ljunggren et al, 2007, Rusten and Ødegaard, 2006). Such controlled COD removal allows to improve the downstream water quality while minimizing additional carbon supplement as well as to maximize the potential for energy and resource recovery from the retained solids. Hence, in addition to the lower capital costs and space requirements, the RBF technology aligns well with the recent focus of municipalities on minimizing net energy usage and maximizing recovery of materials (Degroot et al., 2015; Scott et al., 2015; Väänänen et al., 2016; Väänänen et al, 2017).

Cellulose (in wastewater mainly originating from toilet paper) is known to be an important component of the solids load of a conventional treatment plant as it represents up to 35% of the suspended solids in raw municiaplwastewater (Ruiken et al., 2015). Moreover, as cellulose consists of rather long fibres this material is specifically suited to be removed by sieving. Recent studies have demonstrated that sludge generated by RBF has a higher cellulosic content compared to sludge from conventional primary settling tanks (Sarathy et al., 2015). The diversion of cellulose fibers to the solids stream will affect downstream processes in the water line (as changes in the wastewater composition will affect sludge production and biological nutrient removal) as well as in the sludge line (as altered sludge properties will influence anaerobic degradability and dewaterability).

Surprisingly, although cellulose makes up a large fraction of the incoming suspended solids in wastewater, very little is known of its degradation kinetics making it difficult to assess its fate throughout the activated sludge process and the potential benefits of its preferential removal by pre-treatment. Therefore, in this study, lab experiments (consisting of BOD tests and respirometric experiments) were carried out on toilet paper to obtain insights into the degradation kinetics of cellulose.

# Materials and methods

# **BOD** test

Long term (25 days) Biological Oxygen Demand (BOD) tests were carried out on different toilet paper samples to characterize their aerobic degradation. Oxytop® bottles were used to measure the pressure difference due to oxygen consumption. NaOH tablets were added to the cap of the bottles to avoid interference by  $CO_2$  production and a few drops of Allyl Thiourea (ATU) solution (5g/l) were added to inhibit nitrification. The temperature was controlled at 20°C for all experiments.

### Respirometry

In addition to long term BOD tests, degradation of the toilet paper samples was analysed with a respirometer (Spanjers and Vanrolleghem (2016)). In comparison to BOD tests where microorganisms are only present at very low concentrations (thus simulating biodegradation under natural conditions), respirometric tests use activated sludge that consists of a concentrated mixture of microorganisms (thus simulating enhanced biodegradation similar to that in WRRFs).

The respirometer (BIOSR developed by INSA, Toulouse) consists of a biological reactor (2L) and a measuring cell (0.5L). The biological reactor is agitated with a mechanical stirrer and continuously aerated by a finely pierced stainless tube connected to an air outlet. A smaller measuring cell is connected to the biological reactor by flexible tubing. The measuring cell is also continuously stirred but not aerated. An oxygen sensor is mounted in this cell to continuously measure the oxygen concentration. The specific set-up of these two cells allows to measure the oxygen consumption during biodegradation of organic material. At a fixed time interval, a fresh sample is pumped from the bioreactor to the measuring cell. Since the measuring cell is not aerated and flow is stopped temporarily during the measurement phase (i.e. batch situation), the oxygen concentration in this sample will decrease when consumed by the sludge thus allowing to measure the rate of oxygen consumption. At regular time intervals a new sample is pumped from the bioreactor to the measure is repeated. This allows to monitor changes in the oxygen consumption rate as a function of time. Temperature in both reactors is controlled at 20°C.

# Results

BOD tests were performed on a raw wastewater sample as well as on that same wastewater sample spiked with toilet paper (5g/l). Several important observations can be made from the results in Figure 1. First, the much higher BOD value measured in the sample with toilet paper clearly shows that the toilet paper in the wastewater is biodegradable. Second, the biodegradation of toilet paper only starts after approx. two to three days (prior to this, the two curves behave similarly). This lag

time could be due to the start-up time it takes for degradation of the long cellulose chains into substrates (carbohydrates) that can be consumed by the micro-organisms present in the wastewater. Finally, according to Table 1, the BOD/COD ratio of the raw wastewater curve is about 67% after 25 days whereas for the raw wastewater with toilet paper this ratio is only about 30%. This shows that the biodegradation of toilet paper is slow and difficult. These results indicate that while cellulose is biodegradable, it will likely be quite persistent in WRRFs and thus have a significant contribution to sludge production.



Figure 1: Effect of addition of toilet paper on the BOD curve of raw wastewater

Table 1: COD	of raw	wastewater and	l raw	wastewater	+	toilet p	oaper
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Sample	COD (mg/L)	<b>BOD</b> <sub>25</sub> (mg/l)	Ratio (%)
Raw wastewater	452	304	67
Raw wastewater + toilet paper	5452	1637	30

These experiments were repeated for wastewater samples on different days, each with addition of 5g/l toilet paper (Figure 2). For each of these experiments a clear increase in BOD concentration from day 2 onwards could be observed confirming the previously discussed lag time. Moreover, experiments were repeated with different brands of toilet paper which all gave similar results.



Figure 2: 5- day BOD curves for two different wastewater samples with the addition of toilet paper (5g/l). Each experiment was performed in duplicate. All four BOD curves show a distinct increase in BOD after 2days, indicating a lag time in the degradation of toilet paper (cellulose).

The results of a respirometric experiment on the raw wastewater sample spiked with toilet paper are shown in Figure 3. The oxygen consumption rate shows a fast, initial sharp peak after addition of the wastewater sample spiked with toilet paper (at t=24). This peak corresponds to the shape of a typical respirogram for raw wastewater samples (Spanjers and Vanrolleghem (2016)) and represents the consumption of oxygen due to the fast degradation of organic matter present in the wastewater sample. However, after approx. t=50h a second, much smoother and quite long peak becomes visible. This peak corresponds to a lot of biological oxygen demand and is normally not observed when wastewater without the toilet paper spike is added. Hence, this second peak can be attributed to the degradation of the added toilet paper. It can thus be observed, similarly to the BOD tests, that microorganisms need a certain time (again two days) to start degrading the toilet paper and that although biodegradation of the toilet paper does occur, it is a rather slow process.



Figure 3: Oxygen consumption rate in function of time measured in a respirometer with two additions of an acetate solution (at t=10h and t=18h, leading to initial concentration of 78 mgCOD/L) and an addition of raw WW spiked with 5g/l of toilet paper (at t=24h, leading to initial concentration of 5452 mgCOD/L).

Subsequently, BOD tests were performed by dissolving toilet paper in different matrices: tap water, raw wastewater and secondary clarifier effluent. To each matrix, 5g/l of toilet paper was added. Moreover, some drops of mixed liquor were added to the samples with tap water and secondary effluent to ensure that sufficient active biomass was present in each test to allow biodegradation. The results (Figure 4) show that the kinetics of toilet paper degradation seem to be largely influenced by the matrix in which toilet paper is present. After 25 days, 1%, 30% and 6% removal of the toilet paper's COD can be observed for the experiments with tap water (Figure 4a.), raw wastewater (Figure 4b) and secondary effluent (Figure 4c), respectively.



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Figure 4: BOD tests with toilet paper (5g/l) in different matrices

These results were further confirmed in additional BOD experiments where different dilutions of effluent and wastewater were spiked with 1g/L of toilet paper (Figures 5 and F 6). Higher BOD values can be observed when toilet paper is dissolved in a more concentrated matrix (more effluent or wastewater). The increase in the observed BOD values is too high to be attributed to the increase in COD of the matrix. Note that the BOD5 values in Figure 6 were normalized with respect to a reference BOD curve of 100% raw WW. For the 80% curve this means that the following formula was used: BOD5<sub>(80% raw WW + TP)</sub> – 0.8\*BOD5<sub>(reference)</sub>.

Moreover, it can be observed (Figure 5) that the BOD curves start to diverge from day 2 onwards which is the point at which cellulose degradation starts(as was shown in Figure 1, 2, 3 and 4b/c). It can be concluded that the biodegradation of toilet paper needs a certain component present in the matrix of wastewater for it to be successful indicating that degradation of cellulose is a complex process that deserves further attention. In particular, it could be interesting to elucidate which type of component is responsible for the biodegradation of toilet paper and to define the specific nature of its relation to the degradation kinetics.



Figure 5: BOD curves for toilet paper in different dilutions of secondary clarifier effluent. 20% signifies a mixture of 20% effluent and 80% tap water.



# Figure 6: BOD5 values for toilet paper in different dilutions of raw wastewater. 20% signifies a mixture of 20% wastewater and 80% tap water. The results were normalized with respect to a reference BOD curve of 100% raw wastewater to illustrate the effect of toilet paper degradation.

# Conclusion

BOD and respirometric experiments were carried out to investigate the biodegradation kinetics of cellulose (originating from toilet paper). Results showed that while toilet paper is clearly biodegradable, a 2-day lag time is required and its degradation is rather slow. As such cellulose in a WRRF can significantly contribute to sludge production. Removal of the cellulose fraction in a pre-treatment step can thus have an important impact on the WRRF's capacity. Indeed, a decrease in the sludge production can not only potentially improve the effluent turbidity by relieving pressure on the secondary clarifiers but it can also improve the WRRF's nutrient removal capacity by allowing for longer SRTs (and thus improved conditions for nitrifying biomass). This opens up important perspectives for the use of RBFs (which have been recently demonstrated to preferentially remove cellulose fibres) as pre-treatment to improve existing WRRFs. Finally, the biodegradation of toilet paper was shown to depend on a certain component present in the wastewater matrix. More research is needed to determine this component and the exact relation of this component to the degradation kinetics in order to properly quantify the

degradation kinetics.

# Acknowledgements

The authors wish to acknowledge the financial support by an Engage grant of the Natural Sciences and Engineering Research Council (NSERC). Julie Doucet benefitted from an NSERC Undergraduate Student Research Award. Peter Vanrolleghem holds the Canada Research Chair in Water Quality Modelling.

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