Supporting Information

Removal of Cellulose to Facilitate Microplastics Detection in Wastewater

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S1 - The removal of cellulose by 6% NaOCl, rt, 24 h – 30% H_2O_2, 40°C, 24 h

The current study adopted the two-step digestion protocol using 6% NaOCl at rt for 24 h, followed by 30% H_2O_2 at 40 °C for 24 h, as proposed by Bellamy et al.[1] To accurately determine cellulose dissolution in real wastewater, it was essential to determine whether the digestion agent has any cellulose-removing potential. The findings informed the approach for determining or calculating the CED removal of cellulose in real wastewater.

To test cellulose removal by the digestion agents, 25, 50, and 100 mg of cellulosic products, including hygienic paper, paper roll, and cotton ball, were used. The exact volumes of the digestion agents used to digest real wastewater were examined for cellulose removal. First, 50 mL of 6% NaOCl at rt was contacted with the respective reference cellulose samples and left undisturbed for 24 h. After the time elapsed, the content was sieved using a stainless-steel sieve with 20 µm pores and washed with 40 mL of Milli-Q water four times to drain the NaOCl solution. After, the residue was collected into a 100 mL glass vial and further contacted with 20 mL of 30% H_2O_2 at 40 °C for an additional 24 h. Similarly, the content after the test was sieved using the same sieve and washed with 40 mL Milli-Q water four times. The final residue was vacuum filtered using a 1.5 glass fibre filter, dried at room temperature for 4 days, and weighed. The sieving and draining were not only to replicate the conditions used for the CED dissolution test but also to prevent NaOCl from coming into direct contact with H_2O_2, as that could be hazardous.

As seen in Figure S1, the two-step digestion was highly effective for cellulosic products at 25 mg. At a higher amount of cellulose, the efficiency of the digestion agent drops considerably. At 25 mg of cellulose, the total average removal for the combined cellulosic product was 89.4 ± 7.3%. However, the efficiency dropped to 71.5 ± 4.7% for 50 mg of cellulose and further to 58.4 ± 5.2% for 100 mg of cellulose. The results show that the two-step digestion is less efficient at higher cellulose concentrations; thus, for adequate cellulose removal in wastewater with a high cellulose load, an additional protocol may be required, justifying the inclusion of CED for the selective removal of cellulose.

Since the digestion agents could remove cellulose to a certain degree, it was decided to use the dried sample weight after the two-step digestion of the wastewater as the initial weight of wastewater cellulose before contact with the CED in order to accurately determine how much of the cellulose in the wastewater was removed by the addition of the CED solution.

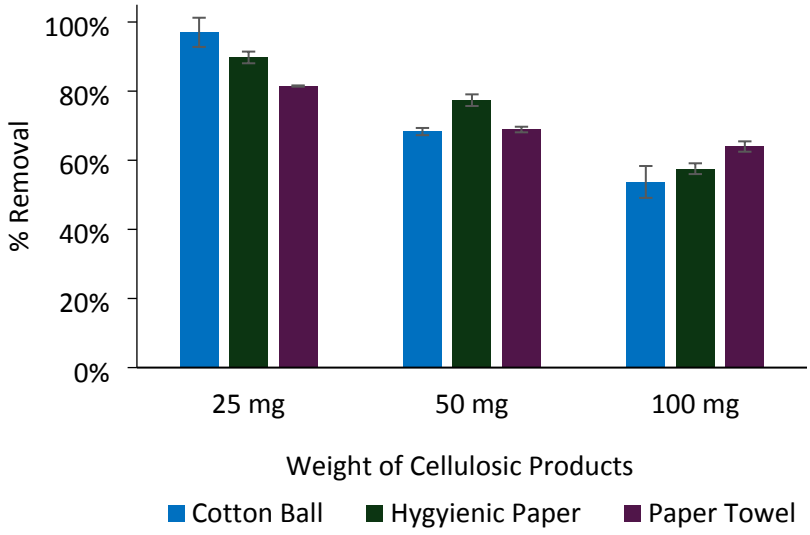

Figure S1. Removal of cellulose by 6% NaOCl, rt, 24 h – 30% H_2O_2, 40 °C, 24 h.

S2 – Real Wastewater Cellulose

The white fibres observed after the influent wastewater samples were digested with 6% NaOCl, rt, 24h – 30% H_2O_2, 40 °C, 24 h (Figure 4-A) were analysed by FTIR to determine their identity. The FTIR spectra shown in Figure S2 revealed the white materials to be cellulose, with a similarity or correlation index of 0.88 - 0.93 to the FTIR library cellulose.

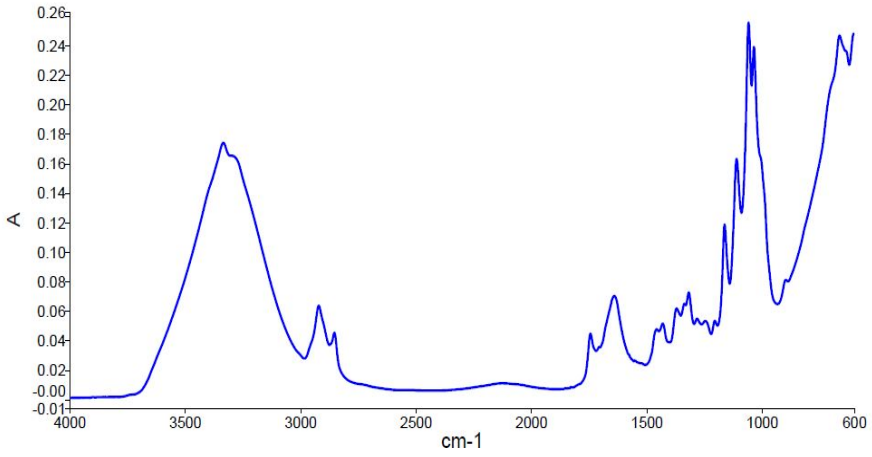

Figure S2. FTIR spectra of cellulose in the dried digester wastewater sample.

References

(1) Bellamy, A., Comeau, Y., & Claveau-Mallet, D. (2025). Effects of Chemical Pretreatment on Natural Fibers Removal and Microplastics Integrity for Wastewater Characterization. *ACS ES and T Water*. https://doi.org/10.1021/acsestwater.4c01233.