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Data Article

Data on the effects of filters, storage conditions, and chlorination in fluorescence and absorbance wastewater measurements



Massimiliano Sgroi, Erica Gagliano, Federico G.A. Vagliasindi, Paolo Roccaro*

Department of Civil Engineering and Architecture, University of Catania, Viale A. Doria 6, 95125, Catania, Italy

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ABSTRACT

Data presented in this data article show artifacts (bias and error) that influence fluorescence measurement of dissolved organic matter (DOM) due to samples handling and storage. Data show interferences in fluorescence measurements related to filtration of water by different filter materials, including 0.7 μm glass microfiber filter, 0.45 μm polyvinylidene fluoride (PVDF) membrane, 0.45 μm cellulose nitrate membrane, and 0.45 μm polyethersulfone (PES) syringe filter. Data show also changes of several fluorescence indexes and UV absorbance measurements of wastewater organic matter respect to time under different storage conditions. Particularly, spectroscopic data were acquired using 0.7 μm filtered and unfiltered wastewater samples stored at different temperatures (i.e. room temperature, 4 °C, -20 °C) over a testing period of 21 days. Finally, data show the effect of chlorine disinfection (doses of 0.5–8 mg/L) in fluorescence measurements accomplished in samples from two secondary wastewater effluents. Data of this article are related to the publication "M. Sgroi, E. Gagliano, F.G.A. Vagliasindi, P. Roccaro, Absorbance and EEM fluorescence of wastewater: effects of filters, storage conditions, and chlorination, *Chemosphere*, 243, 2020, 125292

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* Corresponding author.

E-mail address: paolo.roccaro@unict.it (P. Roccaro).

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[1]". Raw data are available in a public repository (<https://doi.org/10.17632/pf86xs7ybk.1>).

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Specifications Table

Subject area	<i>Environmental Science</i>
More specific subject area	<i>Spectroscopic measurements of wastewater and surface water samples</i>
Type of data	<i>Table, figure</i>
How data was acquired	<i>Shimadzu UV-1800 spectrophotometer; Shimadzu RF-5301PC fluorescence spectrophotometer.</i>
Data format	<i>Raw, processed</i>
Experimental factors	<i>Spectroscopic analyses were performed using filtered (0.7 µm), unfiltered and chlorinated samples.</i>
Experimental features	<i>Assessment of interferences in fluorescence measurements due to presence of leaching substances from filters. Tests on handling procedures for samples preservation, including filtration, storage at room temperature, at 4 °C, and at -20 °C. Chlorination tests to evaluate effects in fluorescence measurements.</i>
Data source location	<i>Sicily (Italy)</i>
Data accessibility	<i>Processed data are provided in this article. Raw data are deposited in a public repository (https://doi.org/10.17632/pf86xs7ybk.1)</i>
Related research article	<i>M. Sgroi, E. Gagliano, F.G.A. Vagliasindi, P. Roccaro, Absorbance and EEM fluorescence of wastewater: effects of filters, storage conditions, and chlorination, Chemosphere, 243, 2020, 125292 [1]</i>

Value of the Data

- These data can be used to detect artifacts in fluorescence and UV absorbance analyses of wastewater organic matter due to the use of inappropriate sample storage or handling procedures, or due to the addition of chlorine in wastewater.
- Data in this article can serve as guideline for researchers and laboratory technicians to perform accurate fluorescence and UV absorbance measurements of wastewater organic matter.
- Data are useful for future investigations on natural water/wastewater samples preservation and for future inter-laboratory comparison studies.

1. Data

The use of fluorescence spectroscopy is emerging and increasingly draws attention for water quality assessment and monitoring. The datasets in this article describe methods for samples handling and preservation before analysis, which should assure correctness of the performed measurements.

Fig. 1 and 2 shows triplicate measurements of fluorescence excitation-emission matrices (EEMs) of blank samples obtained filtering 100 mL of Milli-Q water by glass microfiber filters (0.7 µm), polyvinylidene fluoride (PVDF) membrane filters (0.45 µm), cellulose nitrate membrane filters (0.45 µm), and filtering 10 mL of Milli-Q water by polyether sulfone (PES) syringe filters (0.45 µm). Fluorescence peak intensities shown in the EEMs of Milli-Q water are related to fluorescing substances leached from the utilized filters.

Fig. 3 shows fluorescence EEM and UV absorbance spectra of Milli-Q water after filtration by PES syringe filter (0.45 µm), which was pre-washed with 20 mL of Milli-Q water. Fig. 4 shows EEM of limit of reporting (LOR) values.

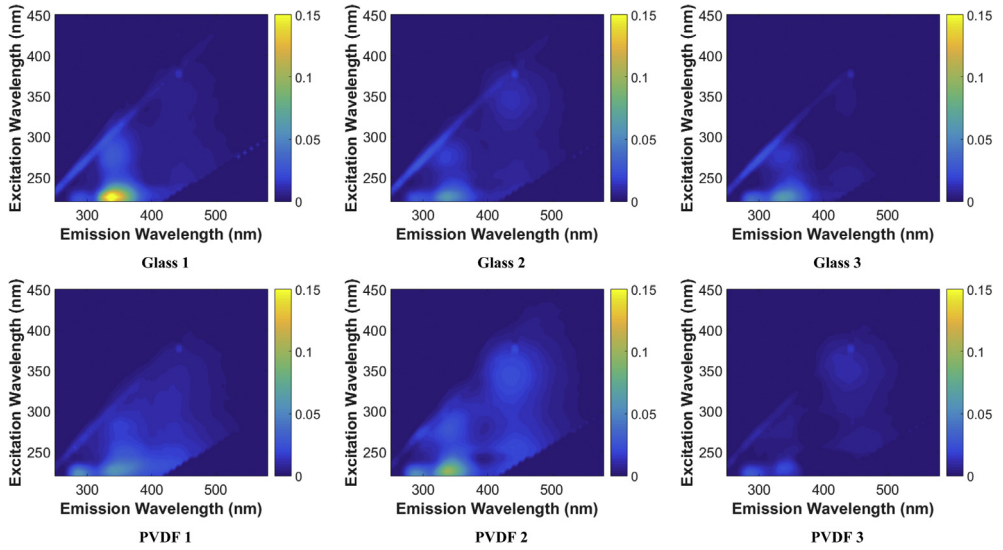


Fig. 1. Fluorescence EEMs of Milli-Q water after filtration with glass microfiber filters (0.7 μm) and PVDF membrane filters (0.45 μm). Measurements were performed in experimental triplicates. A volume of 100 mL of Milli-Q water was used for filtration.

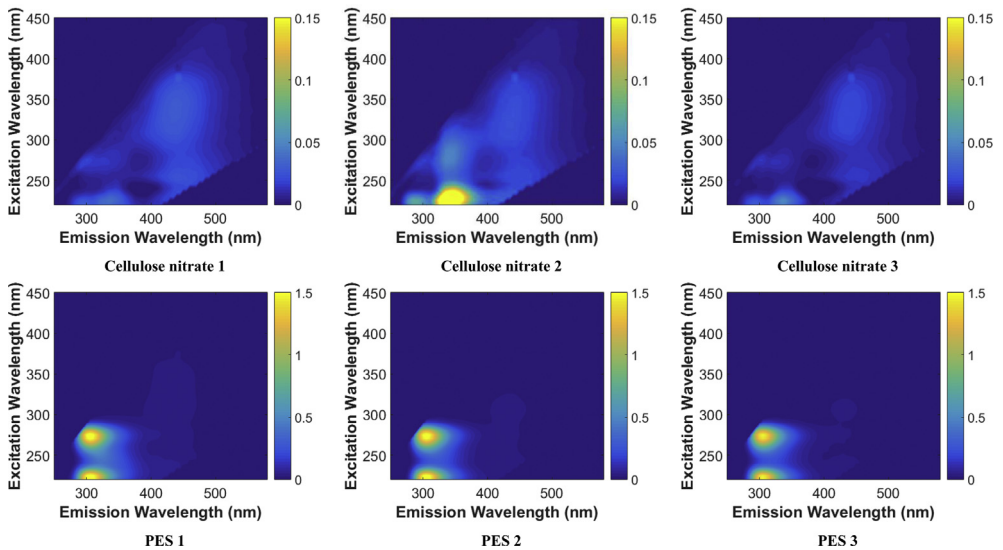


Fig. 2. Fluorescence EEMs of Milli-Q water after filtration with cellulose nitrate membrane filters (0.45 μm) and PES syringe filters (0.45 μm). Measurements were performed in experimental triplicates. A volume of 100 mL of Milli-Q water was used for filtration by cellulose nitrate membrane filters. A volume of 10 mL of Milli-Q water was used for filtration by PES syringe filters.

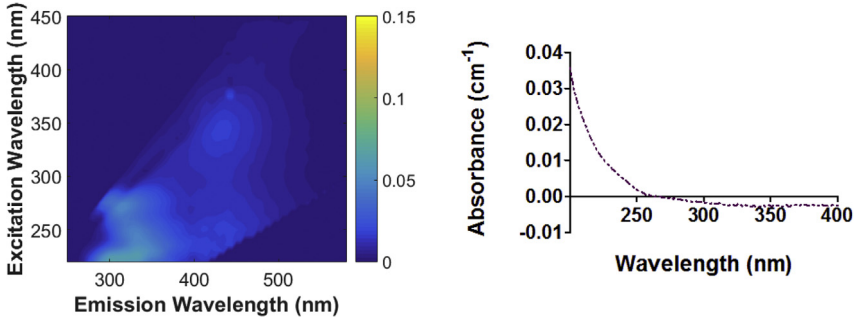


Fig. 3. Fluorescence EEM and UV absorbance spectrum of Milli-Q water after filtration with PES syringe filter (0.45 μm), which was pre-washed with 20 mL of Milli-Q water. Maximum fluorescence intensity measured was 0.064 RU at $\lambda_{\text{ex}}/\lambda_{\text{em}} = 225/330$ nm.

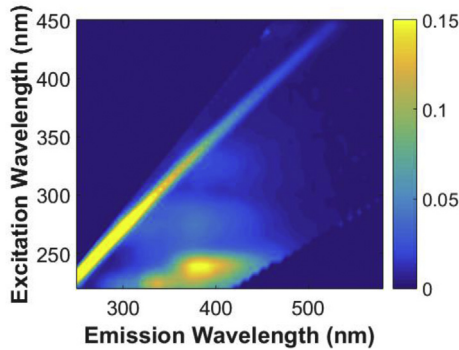


Fig. 4. EEM of calculated LOR values.

Different fluorescence peaks, which are denoted as I_1 , I_2 , I_3 , I_4 , I_5 , were used to test fluorescence changes respect to time during different storage conditions. Changes of fluorescence were also evaluated for total fluorescence (Φ_T), which is a parameter that has been used as a surrogate for micro-pollutant removal by advanced water treatment processes [2,3].

Fig. 5–8 depict the changes of the selected fluorescence indexes respect to time during storage at 4 $^{\circ}\text{C}$, -20 $^{\circ}\text{C}$ and room temperature (≈ 25 $^{\circ}\text{C}$) for unfiltered and 0.7 μm filtered samples of Bronte tertiary wastewater effluent, Lentini primary wastewater effluent, Paternò secondary wastewater effluent, and Pozzillo surface water, respectively.

Fig. 9 depicts the changes of UV absorbance at 254 nm (UV_{254}) respect to time during storage at 4 $^{\circ}\text{C}$, -20 $^{\circ}\text{C}$ and room temperature for unfiltered and 0.7 μm filtered samples of the four tested water qualities. Fig. 10–13 show similar comparison for the full UV absorbance spectra for samples of Bronte tertiary wastewater effluent, Lentini primary wastewater effluent, Paternò secondary wastewater effluent, and Pozzillo surface water, respectively.

Fig. 14–16 show the comparison of changes of the selected fluorescence intensities and Φ_T respect to time during storage at 4 $^{\circ}\text{C}$ and room temperature for 0.7 μm filtered samples of Bronte tertiary wastewater effluent, Paternò secondary wastewater effluent, and Pozzillo surface water, respectively. In Figs. 14–16, changes of the fluorescence indexes I_1 , I_2 , I_3 , I_4 , I_5 are compared to values corresponding to $\pm 5\%$ and $\pm 10\%$ of the fluorescence intensities measured on the day of collection. To sum up, data shown in Figs. 5–16 depict the effect of different storage conditions of water/wastewater samples in

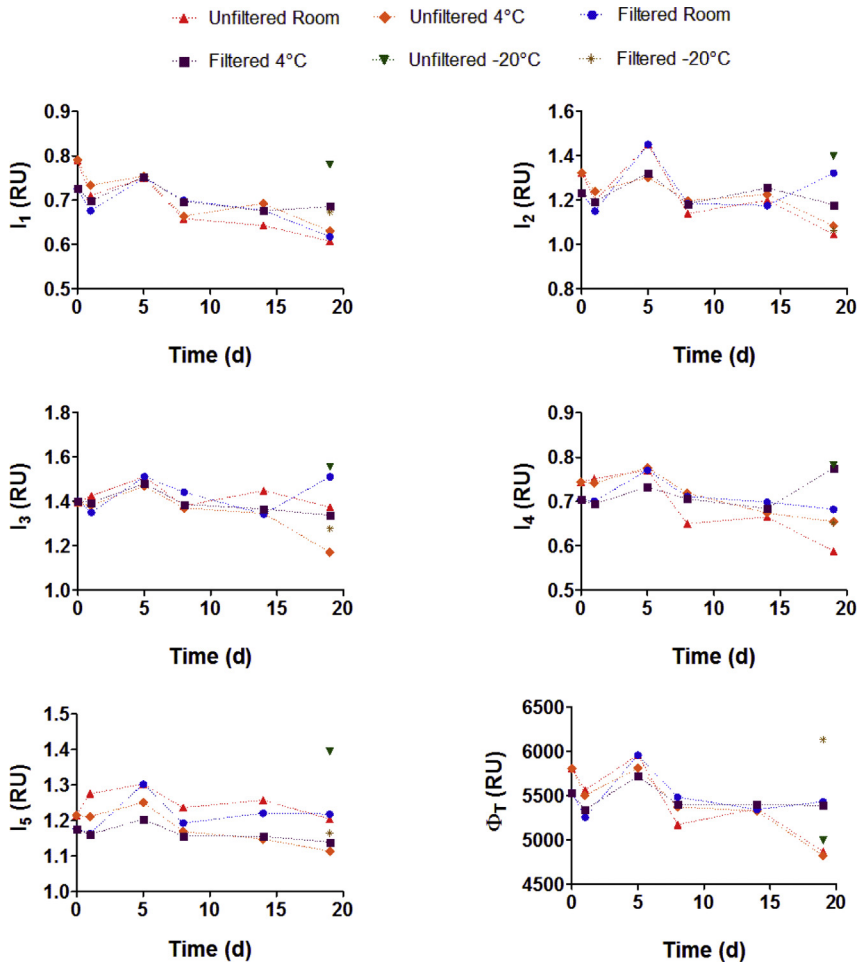


Fig. 5. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to time in unfiltered and 0.7 μm filtered samples of Bronte tertiary wastewater effluent stored at different temperature (4 °C, -20 °C and room temperature).

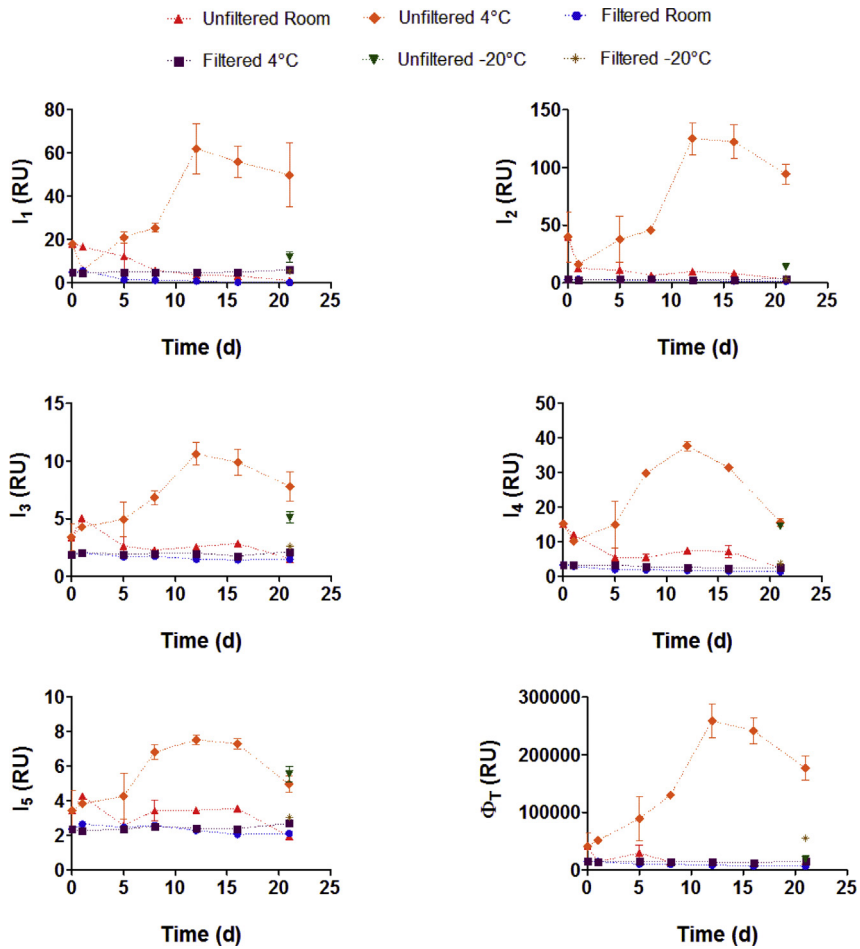


Fig. 6. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to time in unfiltered and 0.7 μm filtered samples of Lentini primary wastewater effluent stored at different temperature (4 $^{\circ}\text{C}$, -20 $^{\circ}\text{C}$ and room temperature). Minimum and maximum values of duplicate measurements are indicated by error bars.

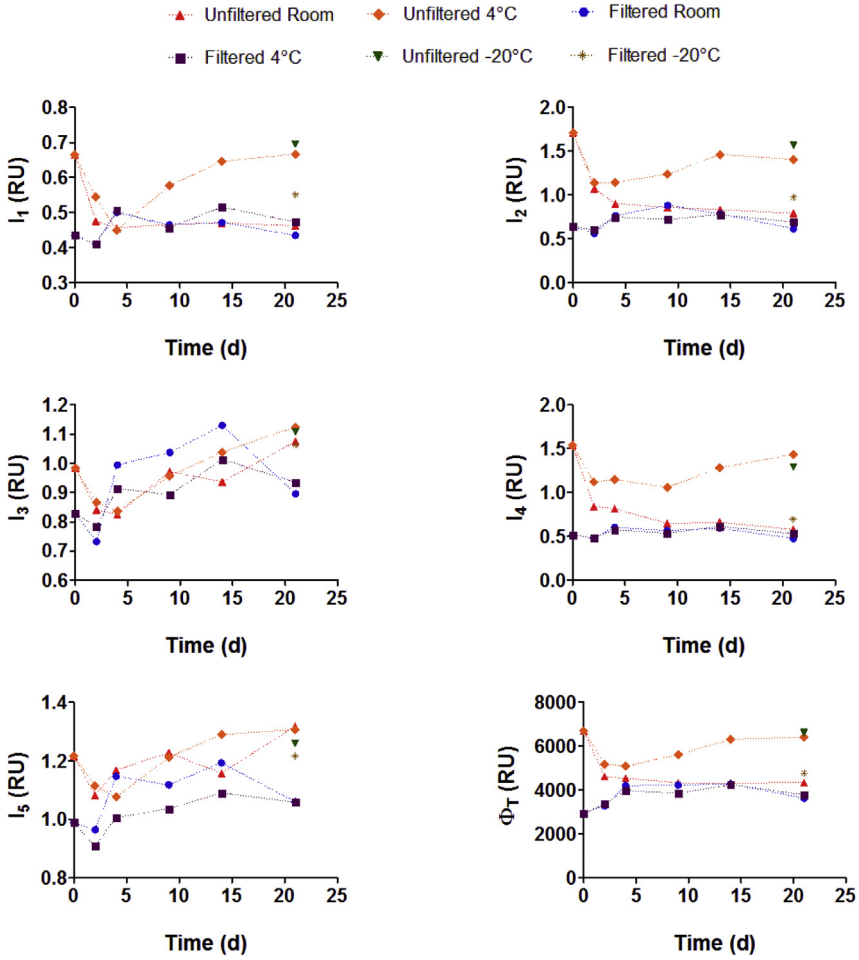


Fig. 7. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to time in unfiltered and 0.7 μm filtered samples of Paternò secondary wastewater effluent stored at different temperature (4 °C, -20 °C and room temperature).

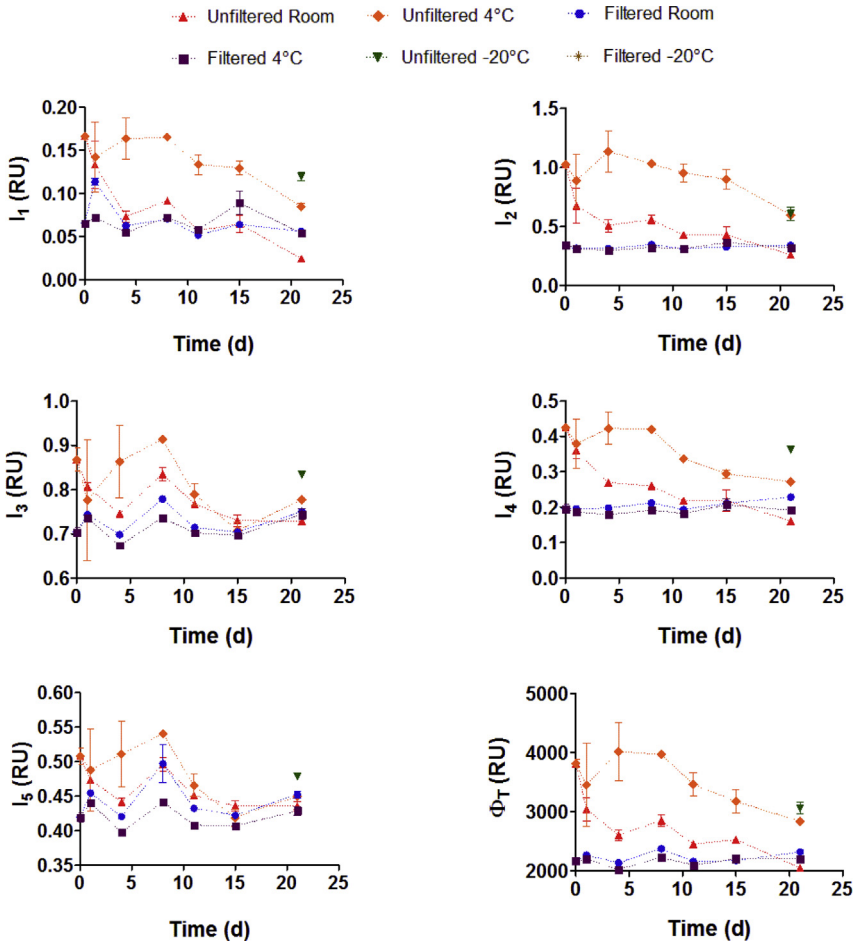


Fig. 8. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to time in unfiltered and 0.7 μm filtered samples of Pozzillo surface water stored at different temperature (4 °C, -20 °C and room temperature). Minimum and maximum values of duplicate measurements are indicated by error bars.

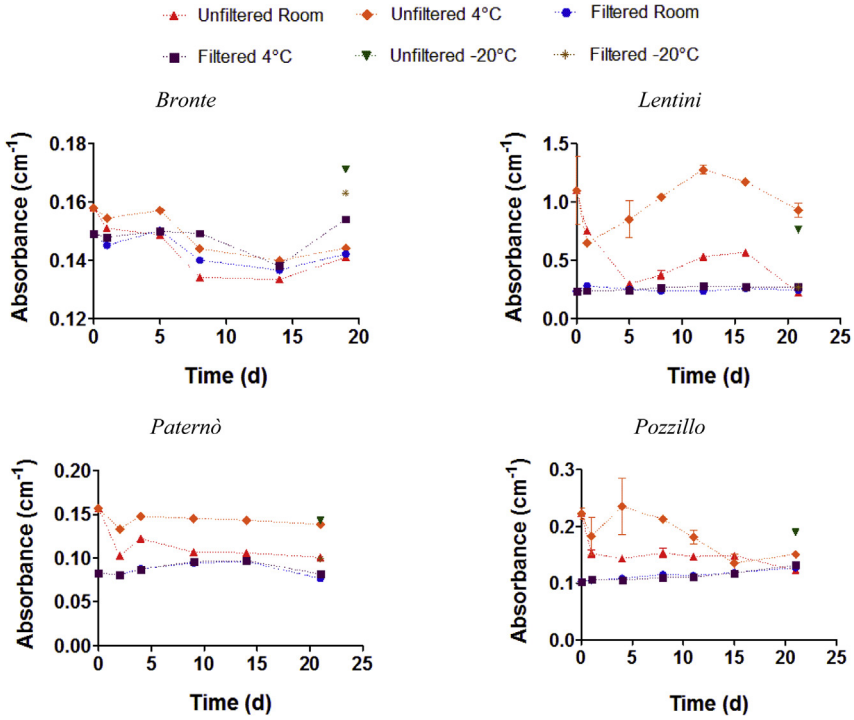


Fig. 9. Changes of UV absorbance at 254 nm respect to time in unfiltered and 0.7 μm filtered samples of all tested waters stored at different temperature (4 °C, -20 °C and room temperature). Minimum and maximum values of duplicate measurements are indicated by error bars.

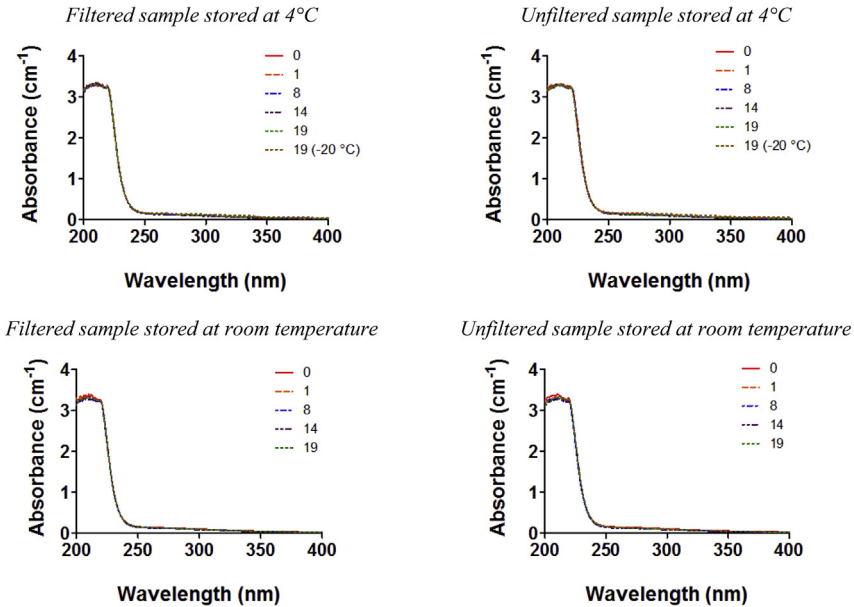


Fig. 10. Changes of UV absorbance spectra respect to time in unfiltered and 0.7 μm filtered samples of Bronte tertiary wastewater effluent stored at different temperature (4 °C, -20 °C and room temperature). Numbers in the legend are indicative of days of storage.

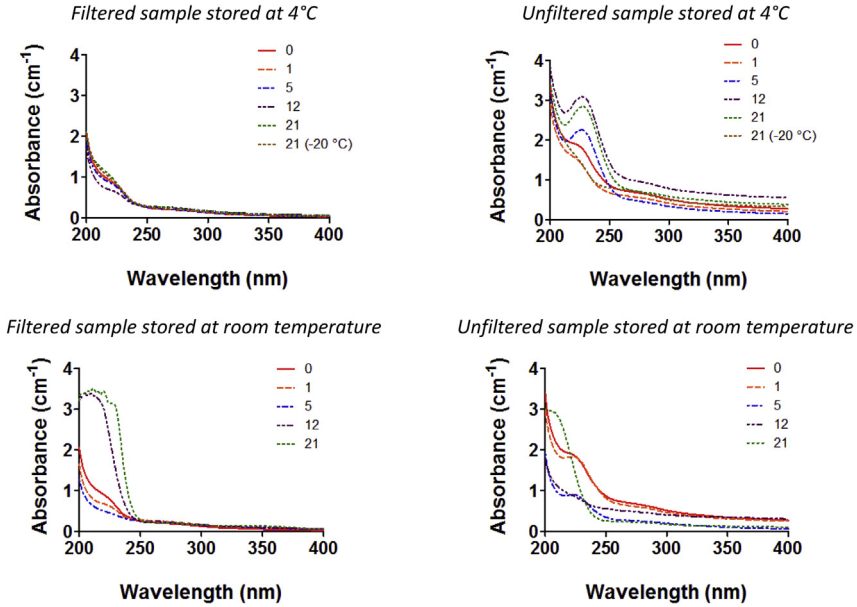


Fig. 11. Changes of UV absorbance spectra respect to time in unfiltered and 0.7 μm filtered samples of Lentini primary wastewater effluent stored at different temperature (4 $^{\circ}\text{C}$, -20°C and room temperature). Numbers in the legend are indicative of days of storage.

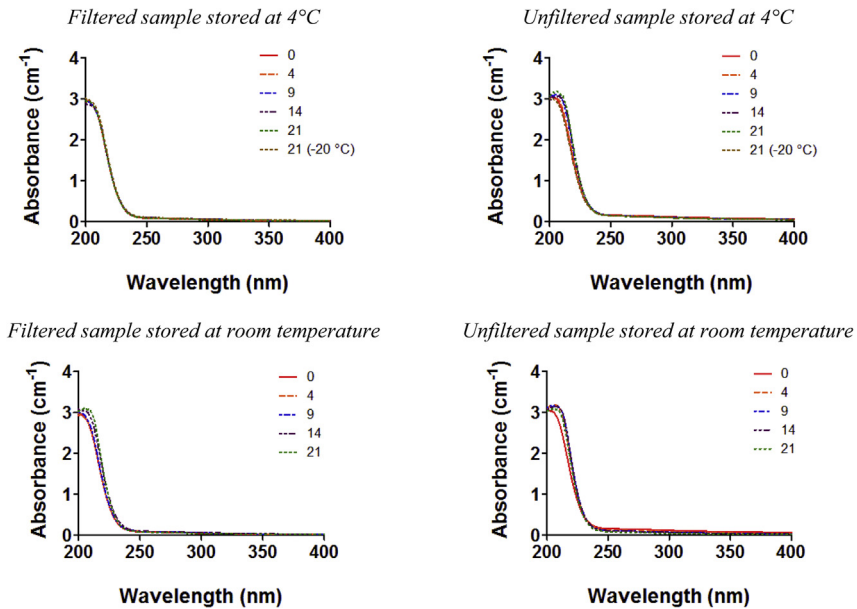


Fig. 12. Changes of UV absorbance spectra respect to time in unfiltered and 0.7 μm filtered samples of Paternò secondary wastewater effluent stored at different temperature (4 $^{\circ}\text{C}$, -20°C and room temperature). Numbers in the legend are indicative of days of storage.

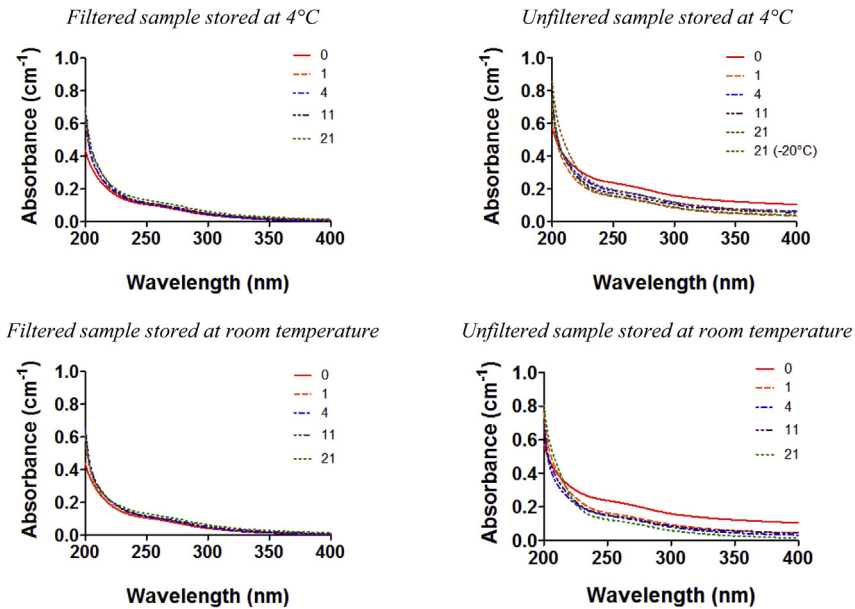


Fig. 13. Changes of UV absorbance spectra respect to time in unfiltered and 0.7 μm filtered samples of Pozzillo surface water stored at different temperature (4 °C, -20 °C and room temperature). Numbers in the legend are indicative of days of storage.

fluorescence and UV absorbance measurements. Discussions and rationales about these data can be found in the manuscript related to this Data article [1].

Fig. 17 depicts the changes of the five selected fluorescence intensities and Φ_T respect to addition of different amount of sodium hypochlorite (0, 0.5, 1, 2, 5, 8 mg/L as chlorine concentration) in the secondary wastewater effluent collected at Lentini wastewater treatment plant (WWTP). On the contrary, Fig. 18 and 19 show the comparison of changes of UV absorbance measurements respect to addition of different doses of sodium hypochlorite in the secondary wastewater effluent collected at Paternò WWTP and in the secondary wastewater effluent collected at Lentini WWTP, respectively. In Table 1 are reported the coefficient of variation of the abovementioned spectroscopic indexes after addition of different doses of sodium hypochlorite in the two tested wastewater effluents.

2. Experimental design, materials, and methods

Data shown in this article were produced using water/wastewater samples collected in four different locations in Sicily (Italy). Particularly, samples were collected in three different WWTPs (i.e., Lentini WWTP, Bronte WWTP, Paternò WWTP), and from an artificial lake (i.e., Pozzillo lake), which is a surface water impacted by wastewater. Lentini and Paternò WWTPs are conventional WWTPs with primary sedimentation and activated sludge unit. On the contrary, Bronte WWTP has an extend aeration scheme with activated sludge unit, sand filtration, but without primary sedimentation. Water quality parameters of tested waters are reported in Sgroi et al [1].

Storage conditions to investigate changes in fluorescence and UV absorbance measurements over time included: 0.7 μm filtered samples stored at 4 °C in the dark; unfiltered samples stored at 4 °C in the dark; 0.7 μm filtered samples stored at room temperature (25 °C) in the dark; unfiltered samples stored at room temperature (25 °C) in the dark; 0.7 μm filtered samples stored at -20 °C in the dark; unfiltered samples stored at -20 °C in the dark.

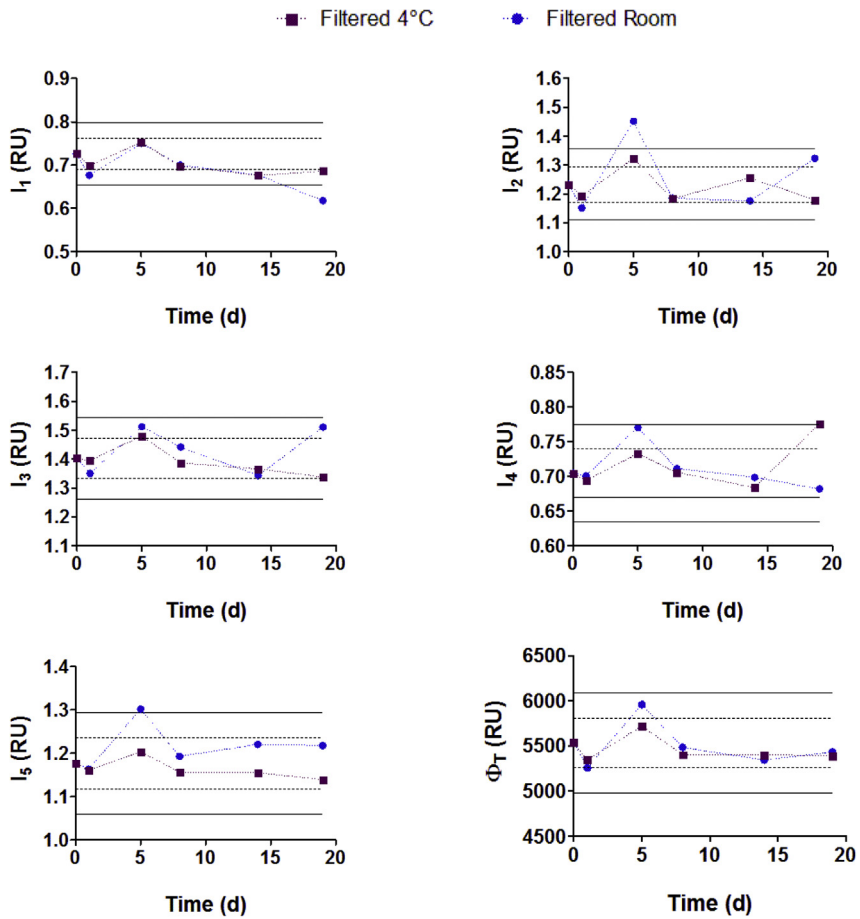


Fig. 14. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to time in 0.7 μm filtered samples of Bronte tertiary wastewater effluent stored at 4 °C and room temperature. Values of $\pm 5\%$ from the fluorescence intensity measured on the day of collection are depicted by dotted lines, whereas values of $\pm 10\%$ are indicated by continuous lines.

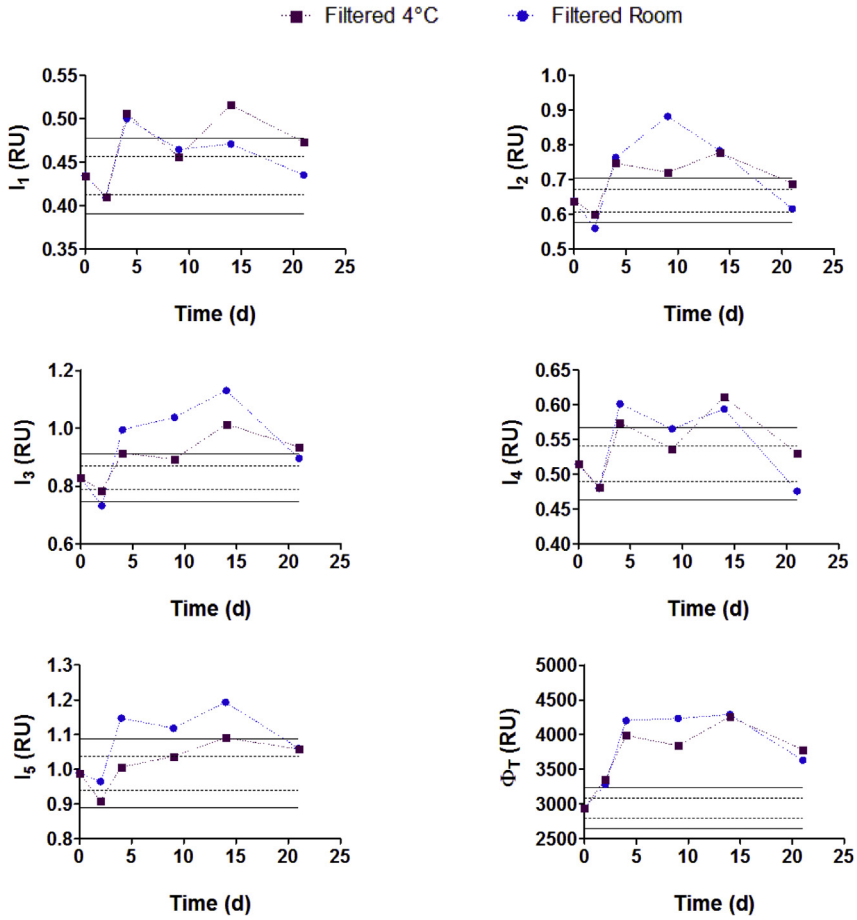


Fig. 15. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to time in 0.7 μm filtered samples of Paternò secondary wastewater effluent stored at 4 °C and room temperature. Values of $\pm 5\%$ from the fluorescence intensity measured on the day of collection are depicted by dotted lines, whereas values of $\pm 10\%$ are indicated by continuous lines.

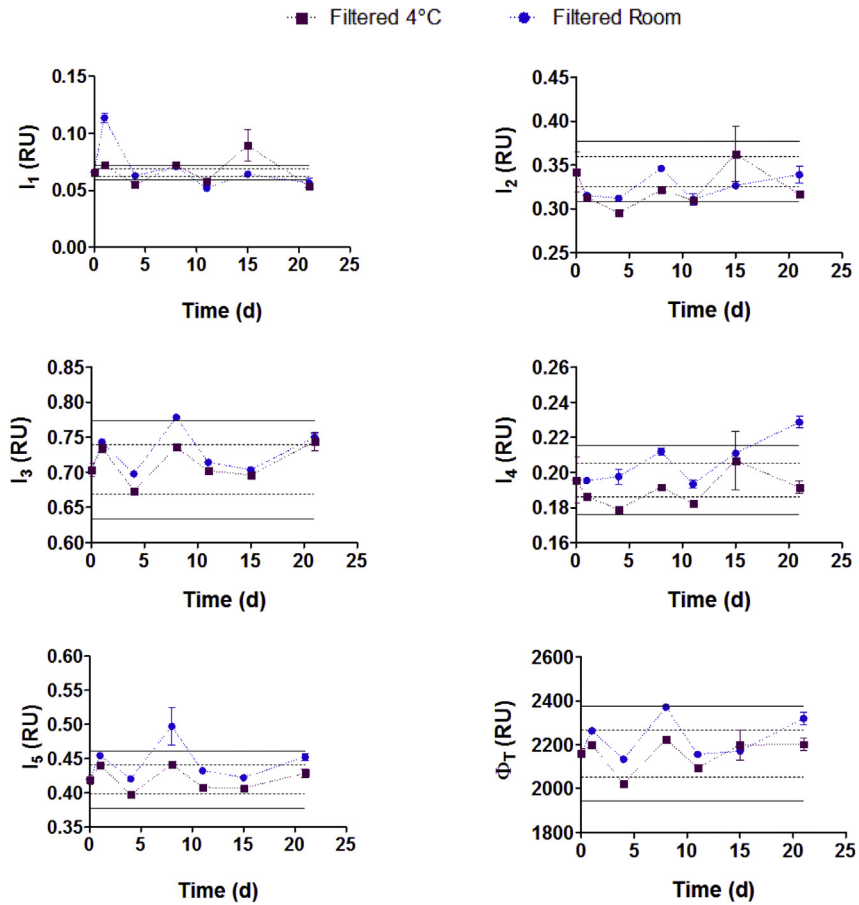


Fig. 16. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to time in 0.7 μm filtered samples of Pozzillo surface water stored at 4 °C and room temperature. Values of $\pm 5\%$ from the fluorescence intensity measured on the day of collection are depicted by dotted lines, whereas values of $\pm 10\%$ are indicated by continuous lines.

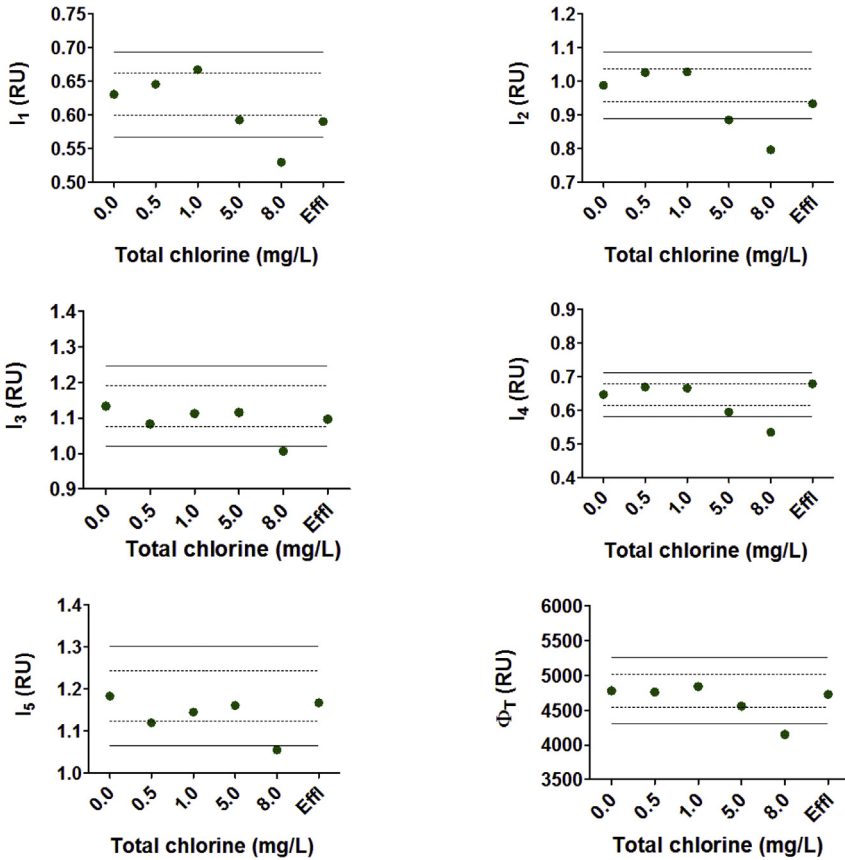


Fig. 17. Changes of selected fluorescence intensities and total fluorescence (Φ_T) respect to addition of different amounts of sodium hypochlorite (0, 0.5, 1, 2, 5, 8 mg/L as chlorine concentration) in Lentini secondary wastewater effluent. Values of $\pm 5\%$ from the fluorescence intensity measured in the non-chlorinated sample are depicted by dotted lines, whereas values of $\pm 10\%$ are indicated by continuous lines. The chlorinated sample collected at Lentini WWTP is denoted by the acronym "Effl".

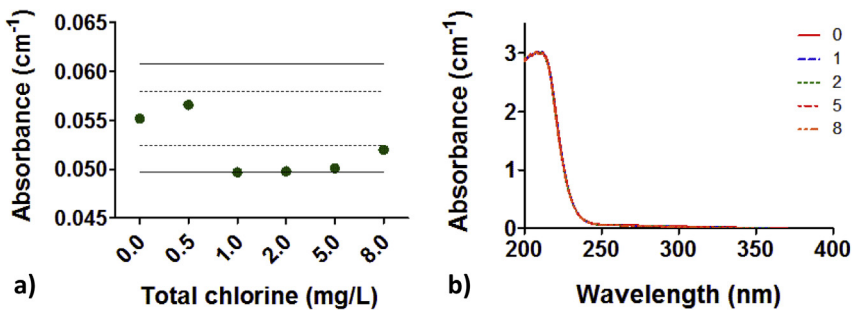


Fig. 18. Changes of UV absorbance spectra respect to addition of different amounts of sodium hypochlorite (0, 0.5, 1, 2, 5, 8 mg/L as chlorine concentration) in Paternò secondary wastewater effluent. In a), values of $\pm 5\%$ from the fluorescence intensity measured in the non-chlorinated sample are depicted by dotted lines, whereas values of $\pm 10\%$ are indicated by continuous lines. In b), numbers in the legend are indicative of dosed chlorine concentration.

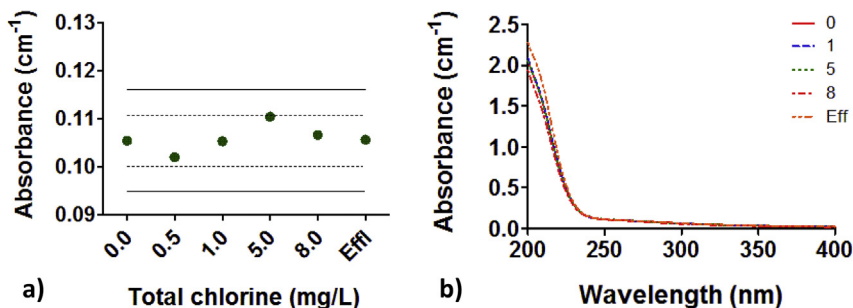


Fig. 19. Changes of UV absorbance spectra respect to addition of different amounts of sodium hypochlorite (0, 0.5, 1, 2, 5, 8 mg/L as chlorine concentration) in Lentini secondary wastewater effluent. In a), values of $\pm 5\%$ from the fluorescence intensity measured in the non-chlorinated sample are depicted by dotted lines, whereas values of $\pm 10\%$ are indicated by continuous lines. In b), numbers in the legend are indicative of dosed chlorine concentration. The chlorinated sample collected at Lentini WWTP is denoted by the acronym "Effl".

Table 1

Coefficient of variation (%) of selected fluorescence intensities, total fluorescence (Φ_T) and UV absorbance at 254 nm (UV_{254}) measured after addition of different amounts of sodium hypochlorite (0, 0.5, 1, 2, 5, 8 mg/L as chlorine concentration) in the secondary wastewater effluents of Paternò and Lentini WWTPs.

Spectroscopic index	Paternò wastewater effluent (%)	Lentini wastewater effluent (%)
I_1	5.5	8.0
I_2	11.1	9.6
I_3	6.6	4.1
I_4	11.0	8.9
I_5	5.6	4.1
Φ_T	4.8	5.5
UV_{254}	5.7	2.6

Five fluorescence peaks (i.e., I_1 , I_2 , I_3 , I_4 , I_5) representative of different fluorescing organic matter components were selected by peak-picking method [4] to assess the effect of storage conditions, and chlorination in UV absorbance and fluorescence measurements of wastewater organic matter. The pair of excitation and emission wavelengths ($\lambda_{ex}/\lambda_{em}$ nm) related to the selected fluorescence peaks were $I_1 = 225/290$ nm (i.e., tyrosine-like fluorescence); $I_2 = 230/355$ nm (i.e., tryptophan-like fluorescence); $I_3 = 245/440$ nm (i.e., fulvic-like and humic-like fluorescence); $I_4 = 275/345$ nm (i.e., tryptophan-like fluorescence); $I_5 = 345/440$ nm (i.e., humic-like fluorescence). Φ_T was calculated following published literature [5].

UV absorbance spectra were measured from 200 to 800 nm at 1 nm intervals using a Shimadzu UV-1800 spectrophotometer (Kyoto, Japan) in a 1 cm quartz cuvette with Milli-Q water used as the blank.

Fluorescence EEMs were analyzed using a fluorescence spectrophotometer (Shimadzu RF-5301PC) with a 1 cm quartz cuvette. The excitation wavelengths spanned from 220 to 450 nm in 5 nm increments, and emission wavelengths from 250 to 580 nm in 1 nm increments. Excitation and emission slit widths were both set to 5 nm. Fluorescence EEM spectra were corrected and processed as described in a previous work [4].

EEM of limit of reporting (LOR) values were calculated according to previous literature [4].

Acknowledgments

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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