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High-resolution sedimentary record of anthropogenic deposits accumulated in a sewer decantation tank

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Abstract

To test the extent to which sediments accumulated in sewers may serve as high-resolution archives of urban evolution, this study examined a sedimentary succession deposited in a decantation tank of the combined sewer network of Orléans (France). The focus was on a 1.43 m sediment core drilled after 10 months of operation since the last cleaning. Sediments were stratigraphically organised into three distinct facies. The lower unit comprised organic remains, the middle unit gravels and sands and the upper unit was fine sands. The evolution of radionuclide activities, bile acid concentrations, and glass microspheres enabled sediments from wastewater and stormwater inputs to be distinguished. Precipitation events that affected the area were the main control on sediment deposition with organic-rich sediments accumulating from wastewater during dry weather and coarser, mineral sediments accumulating from stormwater during precipitation events. These results enable development of a chronological framework for sediment deposition. These findings reveal the potential of sewer sediment accumulations to record relevant information on the evolution of urban socio-ecosystems on monthly to annual scales with a relatively high time resolution.

Keywords: Sewer, wastewater/stormwater, sediments, bile acids, glass microspheres, radioelements

1. Introduction

Human activities are releasing an unprecedented amount and diversity of materials, molecules, isotopes and elements into the environment, some of which are natural but concentrated, while others are synthetic and new to the Earth system (Hazen et al., 2017). Some substances are likely registered in the geological record. This is the founding concept of the Anthropocene, a possible new geological epoch proposed by Crutzen and Stoermer (2000), in which humanity is a major driving force of geological processes on Earth. After identifying the Anthropocene as functionally and stratigraphically distinct from the Holocene (Waters et al., 2016), the Anthropocene Working Group of the International Commission on Stratigraphy proposed a lower boundary of the Anthropocene set in the mid-20th century (Zalasiewicz et al., 2015, 2016, 2017). Cities constitute one of the most emblematic ecosystems (or socio-ecosystems) of the Anthropocene since they concentrate human activities and hence human-derived materials. Apart from the archaeological domain, and with the notable exception of urban speleothems that have proven efficient in recording water pollution and urban management (Pons-Branchu et al., 2014, 2015), the study of geological archives in urban systems for palaeoenvironmental purposes has received little attention up to now (Waters et al., 2018). While urban systems are very active from a sedimentological point of view, with large amounts of materials transported and accumulated, few geological archives are qualitatively comparable to those in more natural environments such as lakes, notably in terms of their temporal resolution. To evaluate the use of sediments accumulated in combined sewer systems as high-resolution archives of the day-to-day urban history, this study addresses the following research questions: (1) What do these sediments comprise? (2) How can we distinguish wastewater and stormwater inputs in a unitary sewer? (3) What is the chronological significance of the vertical distribution of sediments? We analysed sediments accumulated between two clean-out operations in the "Chambre à Sable" of Orléans (France),

a decantation tank that collects wastewater and stormwater from the unitary sewer network of the Orléans conurbation (France). We performed classical sedimentological analyses and compared the evolution of original tracers such as glass microspheres, bile acids and radionuclides to precipitation events in the catchment.

2. Materials and methods

2.1 Study site

The "Chambre à Sable" decantation tank is a 17m deep, 8m diameter cylindrical and then conical underground construction located on the Loire river bank (47°53'51.947"N, 1°53'4.693"E; Figure 1). Nowadays, it collects 7.10^6 m³ of wastewater and stormwater from the unitary sewer network that drains the northern part of the Orléans conurbation (Figure 1), with water flows ranging from 1 to 4 m³/s. The purpose of this construction is to remove sands and gravels from water upstream the La Chapelle-Saint-Mesmin and L'Ile Arrault wastewater treatment plants (WTP, Figure 1).

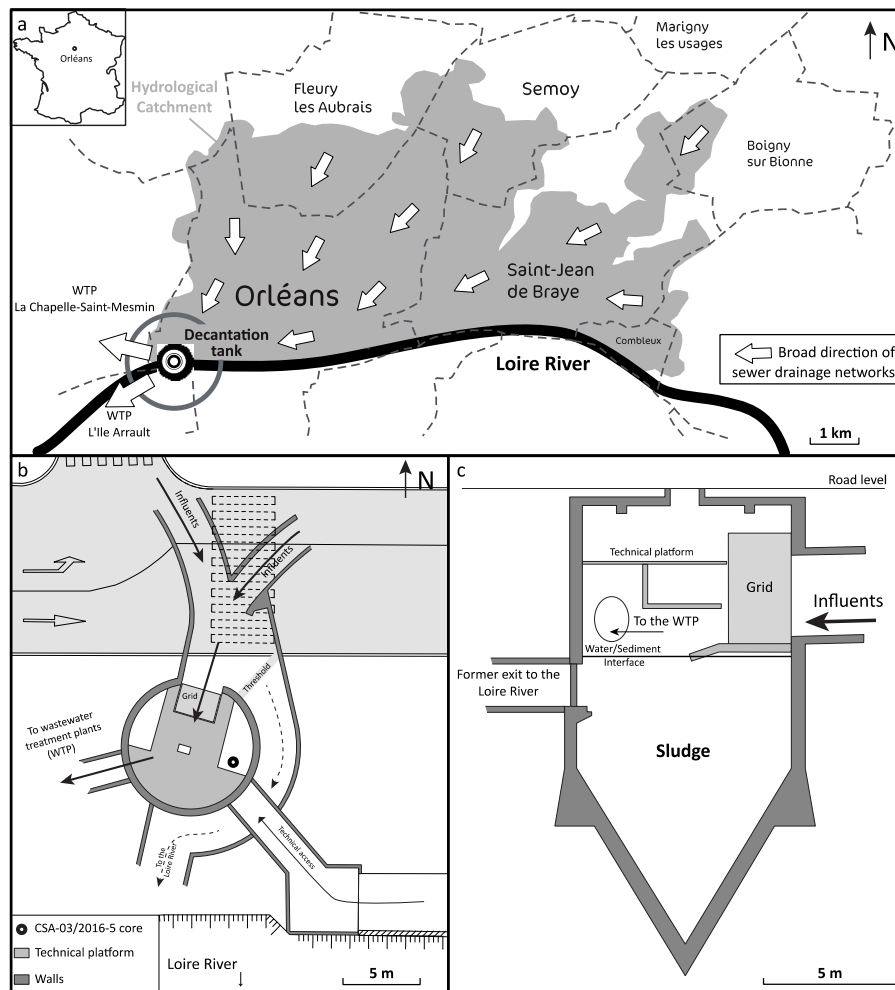


Figure 1: a) Location of the "Chambre à Sable" decantation tank that collects wastewater and stormwater from the unitary sewer network that drains the northern hydrological catchment area of Orléans conurbation; b) Map of the "Chambre à Sable". View from above at the Quai Madeleine with influents and flow directed towards Wastewater Treatment Plants (WTP); c) Cross section of the "Chambre à Sable" illustrating the technical platform and water/sediment interface before the 2015 cleaning operations.

Construction started in 1941 but was only completed in 1947. The full story of the "Chambre à Sable" is accessible, in French, on the "Archives Municipales et Communautaires d'Orléans Métropole" website. In the initial project, the "Chambre à Sable" was directly connected to the Loire River by a 60m long, 1.5m diameter outflow tunnel. At the time, stormwater and wastewater were therefore directly discharged into the Loire River after the decantation of coarse particles and a trap evacuated sands directly into the Loire River after a

certain time of accumulation. According to text records, this system was only operated twice in the first two years and no other system was then designed for sediment clean-out of the "Chambre à Sable". The outflow tunnel to the Loire River was closed in 1980. In the seventies, waters were redirected towards L'Ile Arrault WTP (built in 1969-1972, renovated in 2008-2012) and towards La Chapelle-Saint-Mesmin WTP after 1998. Presently, La Chapelle-Saint-Mesmin WTP processes wastewater and stormwater for 400 000 eq. inhab. and the flow is partially directed towards l'Ile Arrault WTP in the event of heavy rainfall. Despite these developments, the water flow still exceeded the "Chambre à Sable" capacity during intense precipitation and some "grey water" (i.e. wastewater) was released into the Loire River through a siphon. In 2014, local authorities decided to undertake a new phase of renovation of the tank in order to ease clean-out operations and modify flow management.

In May 2015, a large volume of sediments was removed to a depth of 9.7 m below the technical platform, thus affording more space for new sediments to accumulate. Since that time, cleaning has been performed every 6 months/1 year, depending on the volume of sediments accumulated. The water flow downstream the "Chambre à Sable" was measured daily at La Chapelle-Saint-Mesmin WTP. Precipitation was also measured daily with a meteorological station at the same location. Data were acquired by Veolia® and provided by Orléans Métropole.

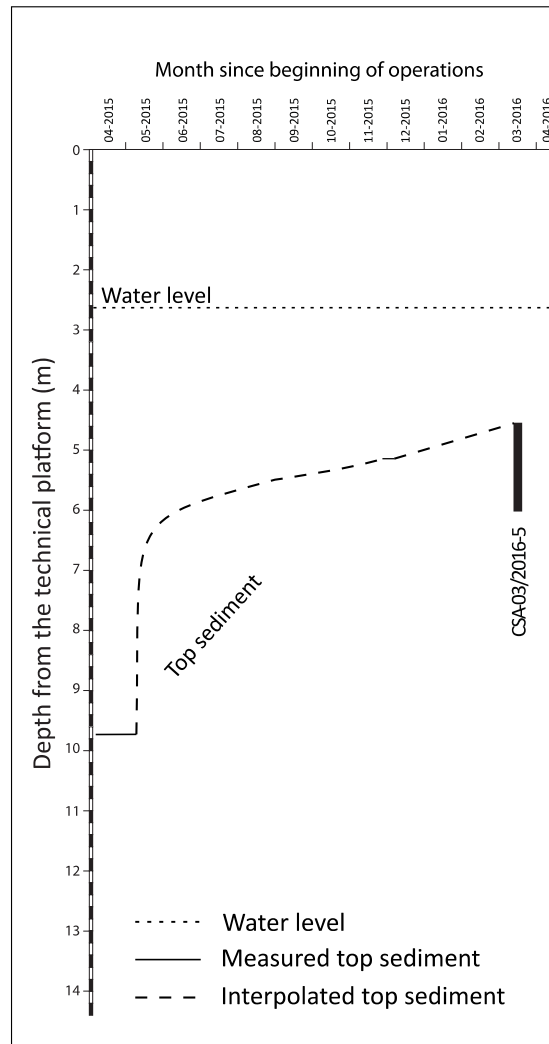


Figure 2: Evolution of bathymetry (i.e. water/sediment interface) in the "Chambre à Sable" since the major cleaning operation in May 2015 and location of core CSA-03/2016-5.

2.2 Coring and sampling

The present study focused on a 1.43 m long sedimentary core drilled with a UWITEC gravity corer in March 18th 2016 (CSA-03/2016-5, 18/03/2016, 63 mm tube diameter; Figure 2), i.e. ten months after the May 2015 major cleaning. At the time, the water/sediment interface was 4.55 m beneath the technical platform (Figure 2). The core was divided into two half cores ("Work" and "Archive") and was then subjected to facies description. 37 samples were collected on the "Work" half core depending on facies (Figure 3).

2.3 Analyses

After sampling, facies and microscopic description, wet sediments underwent extraction of molecular biomarkers. This was first conducted with a mixture of water and methanol (1:1 vol) in order to extract pharmaceutical residues and illicit drugs (not presented here). A second extraction with dichloromethane and methanol (1:1) was carried out to recover lipid biomarkers of which we will only discuss bile acids here (see 2.1.2). Both these extractions were performed with an Automated Solvent Extractor (Dionex ASE 200). Samples were then weighed after drying.

2.3.1 Granulometry, microscopic counting and Scanning Electron Microscopy of glass microspheres

Considering the heterogeneous nature of the sediment (from coarse gravels to sands, silts and clays), granulometry was assessed by sieving the extracted and dry sediment through three sieves with different square mesh sizes ($> 1\text{ mm}$; $>200\text{ }\mu\text{m}$; $> 50\text{ }\mu\text{m}$ and $<50\mu\text{m}$). The sieved components were collected and weighed.

Glass microspheres were counted in duplicate on aliquots of the $50\text{-}200\text{ }\mu\text{m}$ and $< 50\text{ }\mu\text{m}$ fractions with a binocular microscope in all samples from core CSA-03/2016-5. The number of glass microspheres was then averaged per sample and normalized to the weight of dry sediments. The structure and elemental composition of ten isolated glass microspheres randomly collected in several samples were analyzed by scanning electron microscopy (SEM) with a TM 3000 (Hitachi) operating at 15 kV accelerating voltage, coupled with an energy dispersive X-ray spectroscopy (EDS) Swift ED 3000 X-Stream module (Hitachi). SwiftED3000 provides standardless quantitative analyses, normalized to 100%. The AZtecEnergy Analyser Software displays and interprets X-ray data to provide accurate and reliable standardless analysis (Burgess et al., 2007). The acquisition time for EDS point

analyses was 180 s and between 30 and 45 minutes for maps with a resolution of 512 x 512 pixels. Analyses were repeated on 2 to 5 spots on each glass microsphere, either on non-altered surfaces or on particles attached to the surface. Results provided correspond to the mean and standard deviation of all analyses.

2.3.2 Measurement of short-lived radionuclides

In addition to the 37 samples, 6 samples (15-21, 41-46, 64-71, 86-88, 115-116 and 142-143 cm) taken after core description on the "Archive" half core were used for ^7Be ($t_{1/2} = 53$ d) activity determination using well-type germanium detectors installed at the Laboratoire Souterrain de Modane (Reyss et al., 1995). Other radionuclides such as ^{210}Pb , ^{226}Ra , ^{137}Cs , ^{241}Am , were also measured with this method. These measurements were carried out between 7 and 13 June 2016, i.e. 81 to 87 days after core sampling. ^7Be and ^{210}Pb results were corrected accordingly. In each sample, the ($^{210}\text{Pb}_{\text{ex}}$) excess activities were calculated by subtracting the (^{226}Ra -supported) activity from the total (^{210}Pb) activity.

2.3.3 Bulk organic geochemistry

The 50-200 μm fractions of all 37 samples were submitted to Rock-Eval6 analysis (Espitalié et al., 1985a, 1985b; Lafargue et al., 1998; Béhar et al., 2001) in order to determine the Total Organic Carbon (TOC, %), the Hydrogen Index (HI, the degree of hydrogenation of the organic matter; mgHC/gTOC), the Oxygen Index (OI, the degree of oxygenation of organic matter; $\text{mgCO}_2/\text{gTOC}$), and the temperature of maximum production of hydrocarbonaceous compounds during pyrolysis (T_{max} ; $^{\circ}\text{C}$).

2.3.4 Fecal biomarkers

2.3.4.1 Lipid extract preparation

An aliquot of the dichloromethane:methanol (1:1) lipid extract was separated into neutral, acidic and polar compounds on aminopropyl-bonded silica (Jacob et al., 2005). The carboxylic groups of compounds in the acid fraction were methylated by using acetyl chloride in anhydrous methanol (1:2.5) left 1h at 50°C. Then, carboxylic acid methyl esters were separated from esters of hydroxycarboxylic acids by flash chromatography on silica (activated during 24h at 120°C then deactivated with 5% deionized water and stored in heptane). Carboxylic acid methyl esters were recovered with dichloromethane (fraction A1) and hydrocarboxylic acid methyl esters with dichloromethane:isopropanol (2:1; fraction A2). Finally, the alcohol group of hydrocarboxylic acid methyl esters in A2 was silylated with BSTFA in pyridine left during 2h at 60°C. 5 α -cholestane was added to fraction A2 for quantitation.

2.3.4.2 Biomarker identification and quantitation

Silylated A2 fractions were analysed by gas chromatography/mass spectrometry (GC/MS) on a Trace GC Ultra interfaced with a TSQ Quantum XLS MS. The GC was fitted with a Trace Gold TG-5 MS capillary column (60 m x 0.25 mm i.d., 0.25 μ m film thickness). GC operating conditions were as follows: temperature hold at 40 °C for 1 min, then an increase from 40 to 120 °C at 30 °C/min, 120 to 300 °C at 3 °C/min with a final isothermal hold at 300 °C for 70 min. Samples were injected in splitless mode in a 2 μ l volume with the injector temperature set at 280 °C. Helium was the carrier gas at a constant flow rate of 1 ml/min. Esterified and silylated bile acids were identified according to the mass spectra library, relative retention times and interpretation of fragmentation patterns. Quantitation of each compound was performed by measuring the area of its peak on a Total Ionic Current

chromatogram. Then, this area was compared to that of the peak of 5 α -cholestane and related to the weight of dry sediment extracted.

3. Results

3.1 Facies description and granulometry

In core CSA-03/2016-5, sediments are stratigraphically organized and three main units were distinguished according to mean and standard deviation of each parameter (Figure 3):

- (1) Unit 1 extends from 143 to 80 cm and is constituted by brown, fibrous organic sediments that resemble manure and contain 26 ± 9 % of > 1 mm particles, 56 ± 4 % of particles between 200 μ m and 1mm, 17 ± 7 % of particles between 50 and 200 μ m and 1 ± 0.5 % of particles < 50 μ m (Figure 3). A few intercalations of silt/fine sand thin layers (from 128.5 to 128 cm, 122.5 to 120 cm and 119 to 116 cm, respectively) were observed. A level made of cherry, apricot and grape seeds was observed between 120 and 119 cm. Very rare coarse mineral particles were found in this unit, except between 86 and 80 cm depth.

- (2) Unit 2 (from 80 to 51 cm) is constituted by mineral sediments made of a mix of greyish sands and gravels. Grains > 1 mm are the most abundant (61.4 ± 9 %). The fraction between 1 mm and 200 μ m represents 38 ± 9 % and the < 200 μ m fraction only represents 0.3 % on average.

- (3) Unit 3 (from 51 to 0 cm) is made of fine-grained greyish and then yellowish sands (Figure 3). The 1 mm-200 μ m fraction is dominant (82 ± 6 %), the > 1 mm fraction represents on average 17 % (± 6), and the < 200 μ m fraction only represents 0.4 % on average. A level of coarser grains is noted from 36 to 32 cm, above which sediments become more yellowish.

Some distinctive elements were also microscopically identified in all 3 units such as glass debris (at 70, 60 and 52 cm depth, respectively), a rubber stopper (at 123 cm), pieces of

aluminum (between 116 and 86 cm) and asphalt (between 62 and 61 cm), and even a chewing gum (at 23 cm).

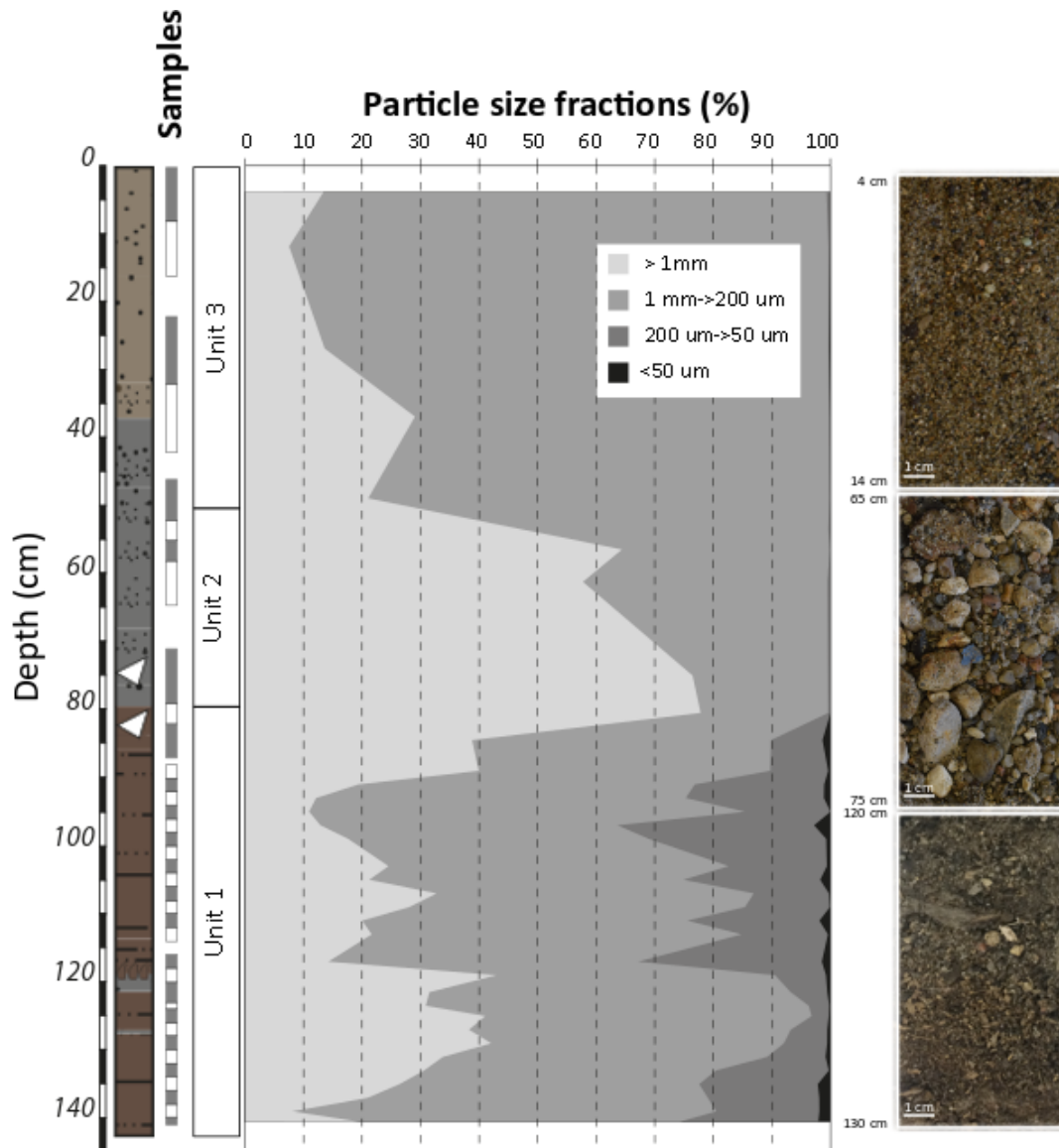


Figure 3: Sedimentary description of core CSA-03/2016-5, samples selected for this study (grey and white rectangles), evolution of particle size fractions (%) and pictures of typical facies encountered in Units 1, 2 and 3.

3.2 Bulk organic geochemistry

In Unit 1, TOC values range from 1.43 to 16.35% with a mean TOC of 5 ± 2 % (Figure 4). Tmax values are around 345°C, except in some samples. HI values range from 270 to 464 mgHC/gTOC (average 341 ± 38) and OI values from 98 to 193 mgCO₂/gTOC (average

142 ± 12). In Units 2 and 3, TOC values range from 0.71 to 1.9% (average 0.7 ± 1.9%) with few variations (Figure 4). HI and OI values are overall within the range of those recorded for Unit 1 (221-429, average 332 ± 53 mgHC/gTOC, and 163-303, average 208 ± 24 mgCO₂/gTOC, respectively) with no particular trend for HI, and a slight increase and punctually higher values for OI.

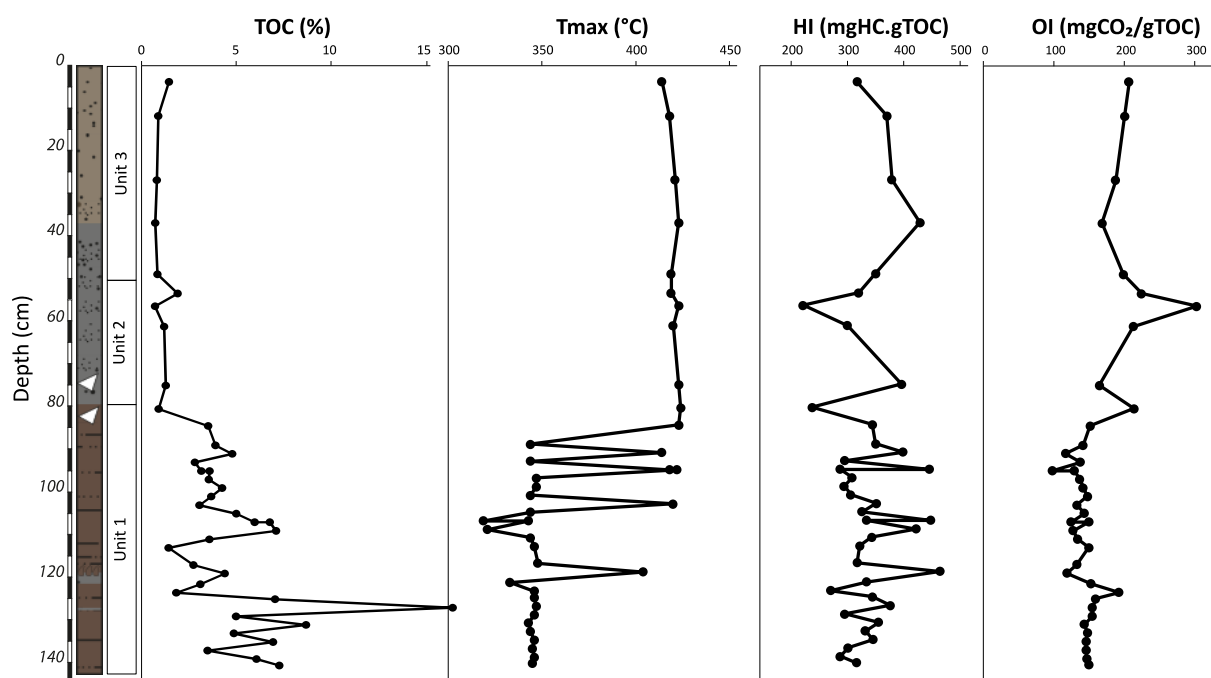


Figure 4: Evolution of Total Organic Carbon (%), Tmax (°C), Hydrogen Index (mgHC/g.TOC) and Oxygen Index (mgCO₂/g.TOC) along core CSA-03/2016-5.

3.2.1 Short-lived radionuclides

Unit 1 shows very low to null ⁷Be activities (Figure 5). ²¹⁰Pb_{ex} (7.2 ± 5.4 Bq/kg) and ¹³⁷Cs activities are very low and close to the detection limit (0.33 ± 0.3 Bq/Kg). In Units 2 and 3, ⁷Be activities are always quantifiable (1.6 ± 1 Bq/kg), ²¹⁰Pb_{ex} present low activities (26 ± 5 Bq/kg) and ¹³⁷Cs activities are also low (5.1 ± 1.4 Bq/kg). ²¹⁰Pb_{ex} and ⁷Be do not present any decreasing trend over the record.

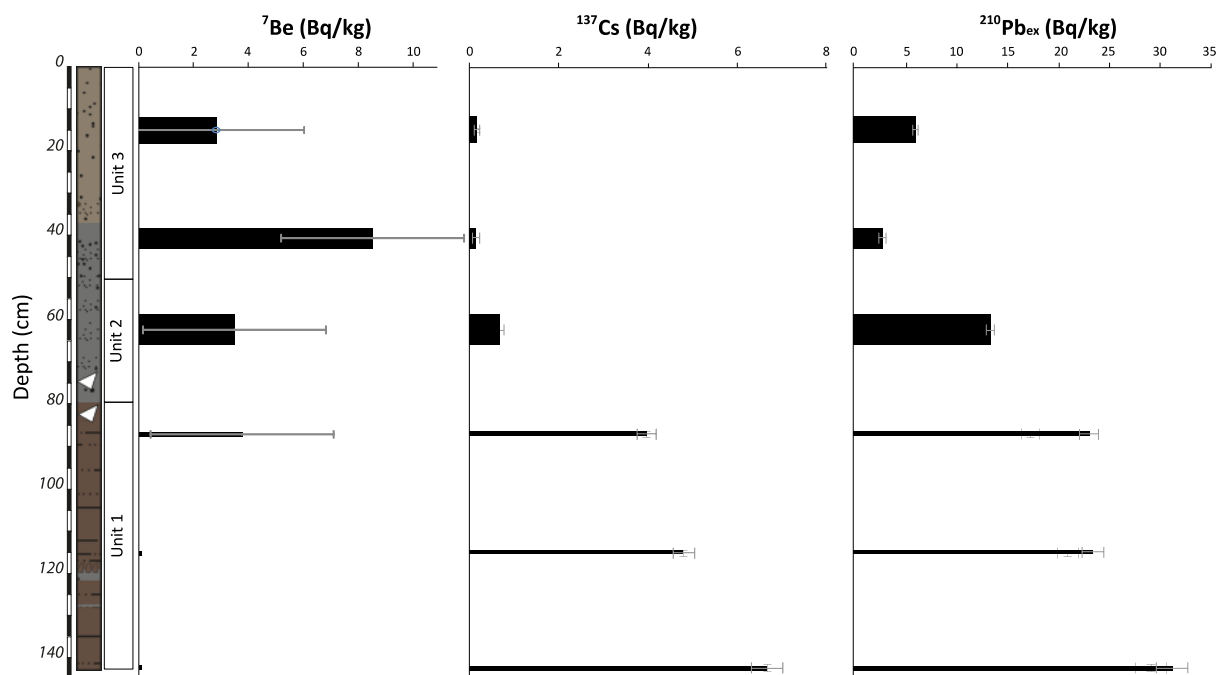


Figure 5: Evolution of ^7Be , ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ activities (Bq/kg) in core CSA-03/2016-5. The vertical thickness of bars corresponds to the thickness of the sample. ^7Be activities for samples at 115.5 and 142.5 cm are below the detection limit. Error bars correspond to analytical error.

3.3 Bile acids

3α -, 3β -deoxycholic, cholic and lithocholic acids were the major bile acids identified in our samples. These compounds are abundant in Unit 1 with a combined concentration reaching $180 \mu\text{g/g.sed}$ (average 60 ± 31 ; Figure 6). Bile acids are far less abundant in Units 2 and 3 with a maximum at $10 \mu\text{g/g.sed}$ (average 4.2 ± 2.8 ; Figure 6).

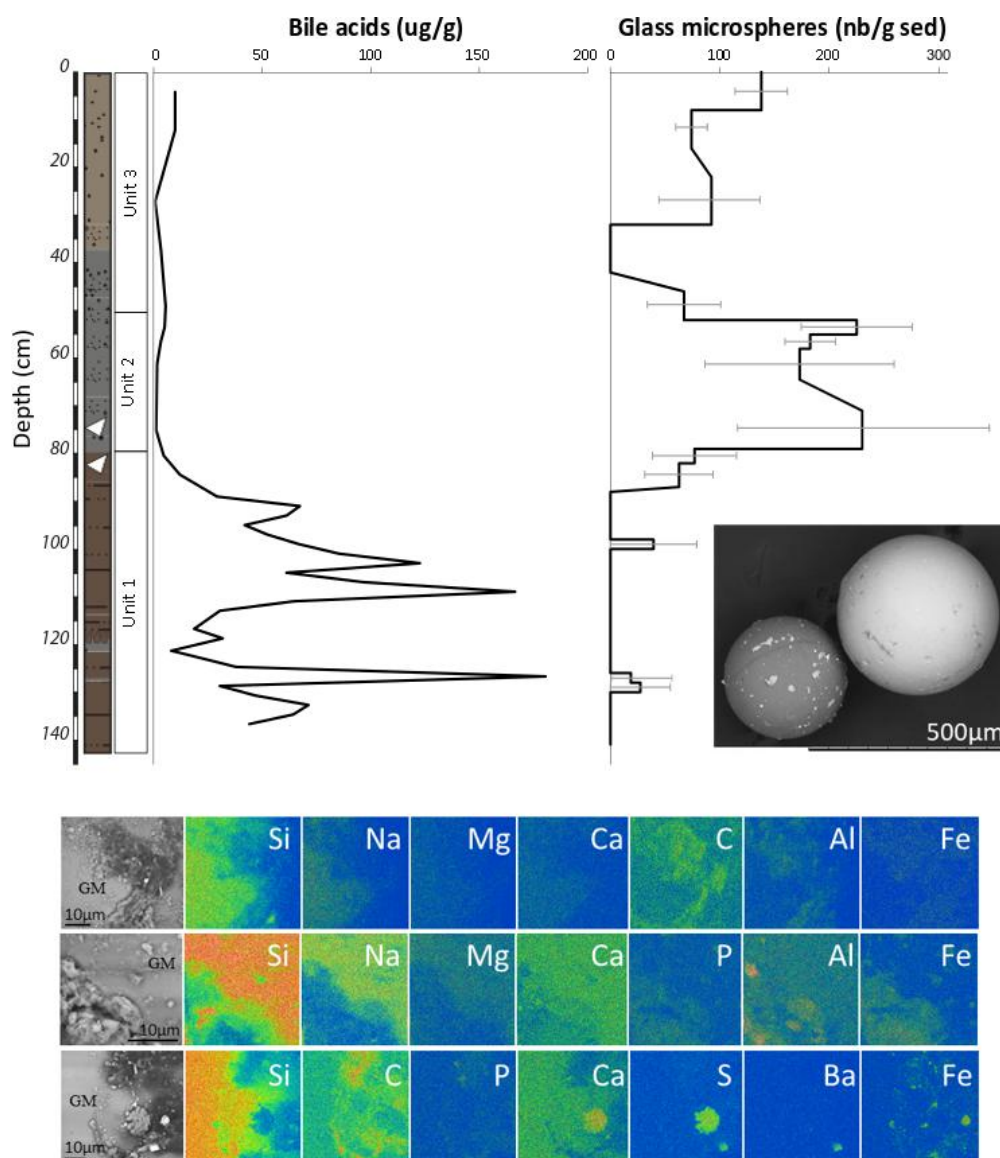


Figure 6: Evolution of the concentration in bile acids (sum of 3 α -deoxycholic, 3 β -deoxycholic, cholic and lithocholic acids; $\mu\text{g/g.sed}$) and the number of glass microspheres (per gram of sediment) with depth in core CSA-03/2016-5. SEM picture of two glass microspheres. Detailed SEM-EDS chemical maps of particles and deposits at the surfaces of another glass microsphere (GM).

3.4 Glass microspheres

Translucid, white or slightly yellow spheres were found in 13 out of 37 samples (Figure 6). Most of them had a diameter between 100 and 200 μm and were found in the 50 – 200 μm fraction, although smaller spheres were found in the <50 μm fraction and larger ones (up to 400 μm) in the 200 μm - 1mm fraction. They are composed of C (33 ± 4 wt%), O (44.7

± 4 wt%), Si (12.7 ± 3 wt%), Ca (2.5 ± 0.5 wt%), Na (3 ± 1.5 wt%), Mg (0.7 ± 0.3 wt%) and Al, Fe, Mn, Cu and Zn traces. This represents ~ 40 SiO₂. This, added to the size of the spheres, is in agreement with values reported in the literature for glass microspheres (or microbeads) originating from traffic paint (Gałuszka and Migaszewski, 2017; Budov and Egorova, 1993). High carbon percentage attests to a surficial carbonaceous film able to trap many various particles of iron oxides (containing Zn or Cu, or both), gypsum, barium sulfates, aluminium, and phosphorous-, sulfur-, or carbon-rich matter (with C > 60 wt% in some locations) (Figure 6). As many as 230 glass microspheres/g.sed. (average 177 ± 25 nb.g.sed) were counted in Unit 2, and 74 ± 33 glass microspheres/g.sed in Unit 3 (Figure 6). These objects were rare in the organic Unit 1 with only three samples showing an average of 60 ± 9 glass spheres per gram.

4. Discussion

4.1 Types of sediment, transport and deposition

Three contrasted facies were distinguished in core CSA-03/2016-5 on the simple evidence of grain size and organic matter content. Unit 1 is mainly made of medium sand and has a high content of organic remains, Unit 2 is made of gravels and sands and Unit 3 is made of sands. This constitutes a first criterion for discussing the factors that control transport in the sewer pipes and sedimentation in the "Chambre à Sable" since these types of sediments correspond well to those described in the classification established by Crabtree (1989) for sediments deposited in combined sewers. Sediments from Unit 2 correspond to Type A (coarse, loose, granular and predominantly mineral). Sediments from Unit 3 correspond to Type C (mobile, fine grained, found in slack flow zones) or Gross Bed Sediment according to Rocher et al. (2004). Sediments from Unit 1 correspond to Type E (fine-grained mineral and organic deposits found in Storm Overflow storage tanks) also noted "Organic Layer" by

Rocher et al. (2004). From these considerations, organic matter and fine grained sediments in Unit 1 as well as fine sands in Unit 3 must have accumulated after suspended transport under lower energy. Sands and gravels from Unit 2 were most probably deposited under strong energy currents/higher flow after bed and near-bed transport (Crabtree et al., 1995).

4.2 Wastewater and stormwater inputs to the sediment

The high TOC values recorded in Unit 1 denote abundant organic matter. This is in agreement with microscopic observations that reveal numerous macroremains such as twigs, leaves, roots and seeds in this interval that could be considered as an organic deposit resembling manure. Conversely, TOC values in Units 2 and 3 indicate a low contribution of organic matter to the sediment, here also in agreement with sedimentological descriptions that indicate abundant gravels and sands in these units. The HI and OI values of all units fall within the range of those encountered in lacustrine sediments for sediments that receive a major contribution from vascular plants (Meyers and Lallier Vergès, 1999). This is not surprising since the major contributions expected from both wastewater (vegetables for nutrition) and stormwater (higher plants thriving on urban soils such as parks, gardens, etc.) consist of vascular plants. Nevertheless, HI values are slightly higher than expected from vascular plant organic matter. This could result from either a contribution of epicuticular waxes that are rich in hydrocarbonaceous compounds (Lüniger and Schwark, 2002) or from fats that are rather common in sewer networks (Crabtree, 1989).

T_{max} values of 345°C and 420°C are typical of an immature organic matter. Nevertheless, the higher T_{max} values in Units 2 and 3 (420°C) could indicate slightly more degraded organic matter (Disnar et al., 2003) than in Unit 1 that contains relatively fresh organic matter (T_{max} = 345°C). This is confirmed by the higher OI values in Units 2 and 3 that could denote slightly more oxygenated/oxidized organic matter than in Unit 1, i.e. organic matter that has

suffered more degradation, for example soil organic matter or reworked organic matter that underwent early diagenesis.

Bile acids are abundant in Unit 1 and rare in Units 2 and 3. These compounds are reputed tracers for fecal contamination by sewage (Elhmmali et al., 1997; Bull et al., 2002). Although they might also originate from fecal material washed from urban surfaces and drained by stormwater, their most probable origin is wastewater since they are abundantly released from drains.

Higher ^7Be activities in the mineral Units 2 and 3 compared to Unit 1 could be the result of lower ^7Be disintegration through time, with regard to a recent deposit, probably less than 6 months to 1 year from the measurement date (June 2016) if ^7Be is still measurable in this coarse material. We observe a nearly complete disappearance of ^7Be in sediments beneath 90 cm depth. This could indicate that these sediments are 6 months to one year older than the coring date. Alternatively, higher ^7Be levels in Units 2 and 3 compared to those recorded in Unit 1 could also denote a stronger contribution of road, sidewalks and soil-derived materials by runoff (LeGall et al., 2017). This is in contradiction with the very low activities of ^{137}Cs in Units 2 and 3 compared to Unit 1. If a major contribution of stormwater inputs is responsible for higher ^7Be activities in Units 2 and 3, one would also expect higher activities for $^{210}\text{Pb}_{\text{ex}}$ and ^{137}Cs . Here we consider that ^7Be can be used as a chronological tracer. Higher activities of $^{210}\text{Pb}_{\text{ex}}$ and ^{137}Cs in the lower Unit 1 could be related either to their high affinity for fine sediments such as clays (with higher specific surface), or to higher accumulation rates in Units 2 and 3 compared to Unit 1.

Although several uses of glass microspheres (or microbeads) are reported in the literature (medicine, abrasive blasting, greenhouse films or hydrogen storage materials), glass microspheres have been mainly used in reflective road, sidewalk, and pedestrian crossing

markings since the 1930s. Gałuszka and Migaszewski (2017) reported that their current consumption reaches several thousand tons per year in some areas due to the necessary renovation of road markings every 6-24 months. As such, the presence and abundance of glass microspheres is related to stormwater inputs.

From these observations on the tracers analyzed in this study, key conclusions can be drawn about the respective contributions of wastewater and stormwater inputs to the sedimentary units. Unit 1 of core CSA-03/2016-5 appears to be mainly composed of materials carried by wastewaters (organic matter, fine grained sediments, bile acids), whereas Units 2 and 3 appear to be mainly derived from stormwater inputs (sands and gravels, glass microspheres).

4.3 Age model, accumulation rates and relationships with precipitation events

In order to understand the controlling factors that lead to the succession of these three units, we then compare this succession of units to meteorological events that occurred during the time span potentially recorded by these sediments. No quantitative information on accumulation rates is available for CSA-03/2016-5. Age controls are (1) the latest cleaning operation undertaken in May 2015 and (2) the date of coring in March 18th 2016. An additional chronological tie-point is the bathymetric level recorded at 5.15 m (from the technical platform) on December 1st 2015, corresponding to 53 cm depth in the core. In addition, the level made of cherry, apricot and grape seeds between 120 and 119 cm in Unit 1 was most probably deposited during the summer. Lastly, it is worthwhile noting that core CSA-03/2016-5 only represents the top 1.43 m over the 5.15 m of sediments that accumulated between May 2015 and March 2016 (Figure 2).

The "Chambre à Sable" was designed for particles to settle after incoming waters have followed a circular trajectory in the tank. As the tank fills with sediments and the water-

sediment interface approaches the altitude of the incoming water flux, the circular current might be less active and the flow may tend to bypass the tank. As such, the decantation is probably less effective for lighter particles to settle when the "Chambre à Sable" is saturated with sediments. The transition from fine organic sediments in Unit 1 to coarse sediments in Unit 2 could reflect this saturation but not the transition to even finer sediments in Unit 3. Therefore, the sedimentary succession in core CSA-03/2016-5 cannot be explained by saturation of the "Chambre à Sable". Other controls must be examined.

The hypothesis that mineral and organic units respond to the respective wastewater and stormwater inputs can be evaluated by comparing grainsize evolution with both precipitation data over the drainage area, and flow evolution in the sewer system (Figure 7).

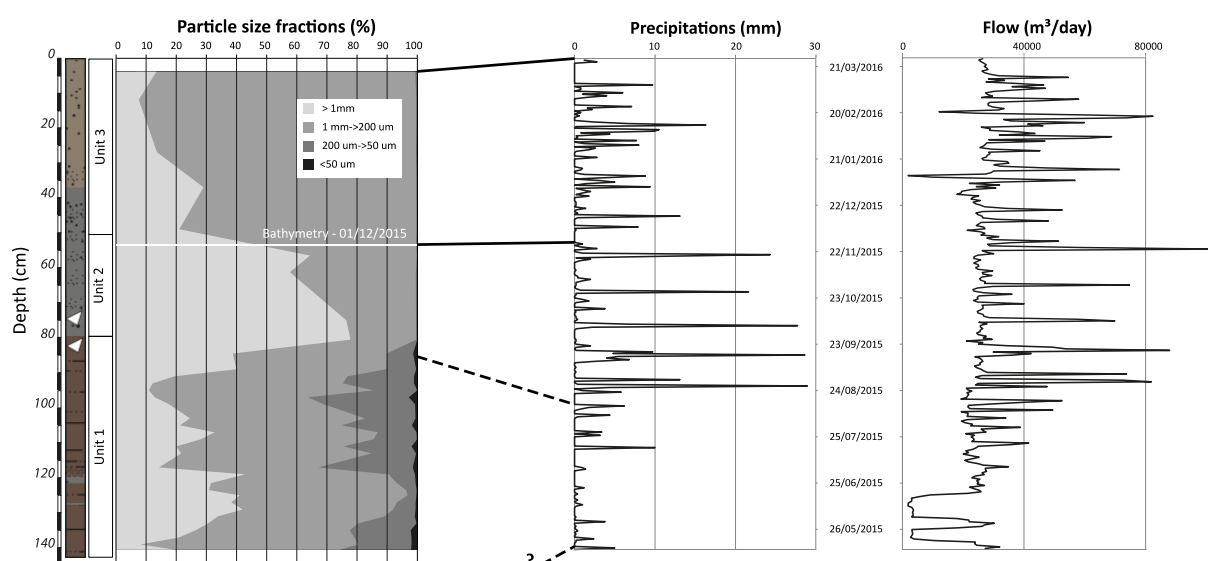


Figure 7: Evolution of particle size fractions in core CSA-03/2016-5 (with depth) compared to the evolution of precipitation in the area and flow upstream La Chapelle-Saint-Mesmin treatment plant over the May 2015-March 2016 period. The flow in the sewer network during the May 2015-June 2015 period was not recorded. Dark thick and dashed lines between particle size fraction evolution with depth and precipitation evolution with time correspond to established and proposed correlations between sediment depth and time.

Figure 7 compares the evolution of particle size distribution with depth in core CSA-03/2016-5 with the flow upstream La Chapelle-Saint-Mesmin WTP (comparable to that in the "Chambre à Sable") and precipitation recorded at this location. The flow is characterized by a nearly continuous background at ca. 300000 m³/day that corresponds to wastewater flow discharged by households. Flow spikes are linked to rainfall events.

The base of Unit 3 (at 51 cm) can be dated from December 2015 from the bathymetric measurement performed at that time. Hence, Unit 3 records the December 2015-March 2016 time-interval. During this period, 13 precipitation events between 5 and 16 mm/day are recorded, leading to a maximum flow of 82000 m³/day.. Fine sands deposited in Unit 3 could have been deposited during these moderate precipitation events.. The average sedimentation rate during this period reaches around 13 cm/month.

Unit 2 was most probably deposited during the whole period marked by five episodes of heavy rainfall (average 26 mm/day), between August and December 2015. These episodes probably washed away coarse materials originating from roads, sidewalks, parks and gardens that could be then transported to the "Chambre à Sable" with elevated flow (average 83860, maximum 104260 m³/day during precipitation events) . The mean sedimentation rate during this period is calculated at around 6 cm/month, i.e. half the sedimentation rate of Unit 3. This is surprising since one would expect higher flows recorded during precipitation events to result in higher sedimentation rates. It is likely that deposition was partly counter-balanced by erosion due to strong water flow during periods of heavy rainfall; an additional factor is that the sediment-water interface altitude is close to that of the entering pipes and can be affected by incoming water flux (Figure 1). Due to the heterogeneous nature of the sediments, no erosive surface attesting to erosional processes could be clearly distinguished in the sediment core.

Lastly, Unit 1 probably corresponds to the May/August 2015 time-interval, when no major rainfall event was recorded. The flow in the network was reduced at that time and sediment is mostly constituted by fine organic material carried by wastewater. This is consistent with the presence of the layer of seeds recorded between 120 and 119 cm reflective of summertime. Dating the Unit 1 / Unit 2 limit at the end of August 2015 is also consistent with radiogenic results. The total disappearance of ^7Be beneath 90 cm depth would have taken ~6 months from August 2015 to March 2016 (or 9 months until the measurement date in June 2016). The age of the lower limit of Unit 1 is harder to define. Unit 1 is only 63 cm long compared to the 4.35 m that accumulated between May and August 2015. Our assumption is that the "Chambre à Sable" rapidly accommodated several meters of sediments (not reached by core CSA-03/2016-5) after the cleaning operations of May 2015 (Figure 2). Then, fine-grained organic particles of Unit 1 started decanting under the influence of wastewater, and in the absence of major precipitation events.

5. Conclusions

This study of the "Chambre à Sable" sediments constitutes a proof of concept for considering sediments accumulated in sewer networks as potential high-resolution archives of environmental and anthropic changes in an urban catchment. By combining standard sedimentological approaches with original tracers, we were able to provide answers to our three research questions: (1) Sediments are stratigraphically organized into three units. The lower Unit 1 is constituted of fine-grained/organic deposits, Unit 2 of mineral gravels and sands, and Unit 3 of fine-grained sands. (2) Wastewater and stormwater inputs can be discriminated with specific tracers such as glass microspheres and bile acids. Coarse, mineral layers can be attributed to stormwater inputs under strong and erosive precipitation events that washed available material on roads, parks, gardens and sidewalks away, as attested by the

presence of glass microspheres. The wastewater contribution is evidenced by bile acids in fine-grained organic deposits since they result from the decantation of organic and light material during dry periods. The unit made of fine sands could correspond to less erosive rainfall events in the catchment during periods devoid of intense precipitation events. (3) The vertical sequencing of these distinct facies and inputs appears strongly responsive to precipitation events in the catchment. Our results suggest that, in this specific case, 1.5m of sediments accumulated in less than one year, affording very high sedimentation rates.

Future work should focus on a more detailed description of facies and examine additional tracers. This would make it possible to relate specific deposits to meteorological or social events and hence improve the chronological framework. Sediment accumulations in similar settings but other locations should also be examined in detail in order to evaluate the extent to which the wastewater/stormwater model proposed here as a proof of concept can be extrapolated to other contexts.

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References

Archives Municipales et Communautaires d'Orléans Métropole - <http://archives.orleans-metropole.fr/f/encycloedifices/24639/fiche/?> (accessed January 2020)

- Behar, F., Beaumont, V., Penteado, H.D.B., 2001. Rock-Eval 6 technology: performances and developments. *Oil Gas Sci. Technol.* 56, 111–134.
- Budov, V.V. Egorova, L.S., 1993. Glass microbeads, application, properties, and technology (review). *Glass and Ceramics* 50, 275-279.
- Bull, I.D. et al., 2002. The origin of faeces by means of biomarker detection. *Env. Int.* 27, 647-654.
- Burgess, S., Statham, P., Holland, J., Chou, Y.H., 2007. Standardless quantitative analysis using a drift detector, what accuracy is possible from live and reconstructed data? *Microsc. Microanal.* 13, 1432-1433.
- Crabtree, R.W., 1989. Sediments in sewers. *Water and Environment Journal* 3, 569-578.
- Crabtree, R.W., et al., 1995. Mousetrap: modelling of real sewer sediment characteristics and attached pollutants. *Wat. Sci. Tech.* 31, 43-50.
- Crutzen, P.J., Stoermer E.F., 2000. The Anthropocene. *IGBP Newsletter* 41, 12.
- Disnar, J.R., et al., 2003. Soil organic matter (SOM) characterization by Rock-Eval pyrolysis: scope and limitations. *Org. Geochem.* 34, 327–343.
- Elhmmali, M.M., et al., 1997. Bile acid as a new class of sewage pollution indicator. *Env. Sci. Tech.* 31, 3663-3668.
- Espitalié, J., et al., 1985a. La pyrolyse Rock-Eval et ses applications; première partie. *Rev. Inst. Fr. Pétrol.* 40, 563–579.
- Espitalié, J., et al., 1985b. La pyrolyse Rock-Eval et ses applications; deuxième partie. *Rev. Inst. Fr. Pétrol.* 40, 755–784.
- Gałuszka, A., Migaszewski, Z.M., 2017. Glass microspheres as a potential indicator of the Anthropocene: A first study in an urban environment. *The Holocene* 28, 1-7.
- Hazen, R.M., et al., 2017. On the mineralogy of the "Anthropocene Epoch." *American Mineralogist* 102, 595-611.

- Jacob, J., et al., 2005. Pentacyclic triterpene methyl ethers in recent lacustrine sediments (Lagoa do Caçó, Brazil). *Org. Geochem.* 36, 449–461.
- Lafargue, E., et al., 1998. Rock-Eval 6 applications in hydrocarbon exploration, production, and soil contamination studies. *Rev. Inst. Fr. Pétrol.* 53, 421–437.
- LeGall, M., et al., 2017. Investigating the temporal dynamics of suspended sediment during flood events with ⁷Be and ²¹⁰Pbxs measurements in a drained lowland catchment. *Scientific Reports* 7, 42099.
- Lüniger, G., Schwark, L., 2002. Characterisation of sedimentary organic matter by bulk and molecular geochemical proxies: an example from an Oligocene maar-type Lake Enspel, Germany. *Sediment. Geol.* 148, 275-288.
- Meyers, P.A., Lallier-Vergès, E., 1999. Lacustrine sedimentary organic matter records of Late Quaternary paleoclimates. *Journal of Paleolimnology* 21, 345-372.
- Pons-Branchu, E., et al., 2014. A geochemical perspective on Parisian urban history based on U-Th dating, laminae counting and yttrium and REE concentrations of recent carbonates in underground aqueducts. *Quat. Geochron.* 24, 44-53.
DOI:10.1016/j.quageo.2014.08.001
- Pons-Branchu, E., et al., 2015. Three centuries of heavy metal pollution in Paris (France) recorded by urban speleothems. *Sci. Tot. Env.* 15, 86-96.
DOI:10.1016/j.scitotenv.2015.02.071
- Reyss J.L., et al., 1995. Large low background well type detectors for measurements of environmental radioactivity. *Nucl. Instrum. Methods* 357, 391-397.
- Rocher, V., et al., 2004. Hydrocarbons and heavy metals in the different sewer deposits in the ‘Le Marais’ catchment (Paris, France): stocks, distributions and origins. *Sci. Tot. Env.* 323, 107-122.

- Waters, C.N., et al., 2016. The Anthropocene is functionally and stratigraphically distinct from the Holocene. *Science* 351. DOI: 10.1126/science.aad2622
- Waters, C.N., et al., 2018. Global Boundary Stratotype Section and Point (GSSP) for the Anthropocene Series: Where and how to look for potential candidates. *Earth-Science Reviews* 178, 379-429.
- Zalasiewicz, J., et al., 2015. When did the Anthropocene begin? A mid-twentieth century boundary level is stratigraphically optimal. *Quat. Int.* 383, 196–203.
- Zalasiewicz, J., et al., 2016. The geological cycle of plastics and their use as a stratigraphic indicator of the Anthropocene. *Anthropocene* 13, 4–17.
- Zalasiewicz, J., et al., 2017. The Working Group on the Anthropocene: summary of evidence and recommendations 2016. *Anthropocene* 19, 55–60.

Figure captions

Figure 1: a) Location of the "Chambre à Sable" decantation tank that collects wastewater and stormwater from the unitary sewer network that drains the northern hydrological catchment area of Orléans conurbation; b) Map of the "Chambre à Sable". View from above at the Quai Madeleine with influents and flow directed towards Wastewater Treatment Plants (WTP); c) Cross section of the "Chambre à Sable" illustrating the technical platform and water/sediment interface before the 2015 cleaning operations.

Figure 2: Evolution of bathymetry (i.e. water/sediment interface) in the "Chambre à Sable" since the major cleaning operation in May 2015 and location of core CSA-03/2016-5.

Figure 3: Sedimentary description of core CSA-03/2016-5, samples selected for this study (grey and white rectangles), evolution of particle size fractions (%) and pictures of typical facies encountered in Units 1, 2 and 3.

Figure 4: Evolution of Total Organic Carbon (TOC, %), Tmax (°C), Hydrogen Index (HI, mgHC/g.TOC) and Oxygen Index (OI, mgCO₂/g.TOC) along core CSA-03/2016-5.

Figure 5: Evolution of ⁷Be, ¹³⁷Cs and ²¹⁰Pb_{ex} activities (Bq/kg) in core CSA-03/2016-5. The vertical thickness of bars corresponds to the thickness of the sample. ⁷Be activities for samples at 115.5 and 142.5 cm are below the detection limit. Error bars correspond to analytical error.

Figure 6: Evolution of the concentration in bile acids (sum of 3α-deoxycholic, 3β-deoxycholic, cholic and lithocholic acids; µg/g.sed) and the number of glass microspheres (per gram of sediment) with depth in core CSA-03/2016-5. SEM picture of two glass microspheres. Detailed SEM-EDS chemical maps of particles and deposits at the surfaces of another glass microsphere (GM).

Figure 7: Evolution of particle size fractions in core CSA-03/2016-5 (with depth) compared to the evolution of precipitation in the area and flow upstream La Chapelle-Saint-Mesmin treatment plant over the May 2015-March 2016 period. The flow in the sewer network during the May 2015-June 2015 period was not recorded. Dark thick and dashed lines between particle size fraction evolution with depth and precipitation evolution with time correspond to established and proposed correlations between sediment depth and time.

