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Nature of trace element carriers and organic matter in combined sewer during dry and wet weather

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The impact of combined sewer overflows (CSO) discharges on the receiving natural system is nowadays a major concern in environmental protection. Indeed, the amount of pollutants released from CSO within one storm event may exceed annual discharges from factories and sewage plants. Until now, the pollution issued from CSO has been estimated in terms of total amount of heavy metals, hydrocarbons, nutrients... Such an approach only provides a rough assessment of contaminant mobility and bioavailability in receiving waters since pollutant behavior is mainly governed by its speciation. The aim of this study was then to investigate the speciation of trace elements contained in CSO by TEM-EDX electron diffraction and SEM-EDX, and the nature of organic matter at molecular scale.

Samples of sewage and CSO were collected from the sewer pipe "Liberation", upstream Boudonville detention basin (Nancy, France). The catchment area receives runoff from 246 ha of urban surfaces, both residential and commercial areas ($\sim$ 20 000 inhabitants). Samples were settled for 2 hours to yield a "suspended" and a "sediment" fraction which were then freeze-dried. Electron microscopy observations were

performed with a Philips CM20 TEM (200 kV) coupled with and EDAX energy dispersed X-ray spectrometer (EDX), and with a S-2500 Hitachi SEM equipped with a Kevex 4850-S EDS. Trace element carriers were identified from the elemental analysis of individual particles. The different fractions of the extractable organic matter (saturated and aromatic hydrocarbons as well as polar compounds) were analyzed by gas-chromatography – mass spectrometry.

During dry weather, chalcophile elements were found to accumulate in sewer sediments as early diagenetic sulfide phases. The sulfurization of some metal alloys was also evidenced. The existence of such sulfides can be linked to microbially mediated sulfate reduction in stagnant portions of the sewer system. Other heavy metal carriers detected in sewage include metal alloys, some iron oxihydroxide phases and neoformed phosphate minerals such as anapaite. Zeolitic minerals and clay particles (kaolinite mainly) are also observed in the sewer system but do not appear to represent significant heavy metal carriers. During rain events, the detailed characterization of individual mineral species allowed to differentiate the contributions from various specific sources. Flushing of urban surfaces was associated with the presence of bitumen particles and of solids derived from the corrosion and wearing of motor vehicles (barite from automobile brakes, W-Cr-Co carbide particles used as anti-corrosive metal coatings, rare earth oxides from catalytic exhaust pipes). PbSn alloys and lead carbonates were attributed to zinc-works from roofs and paint from building siding. Contribution from soil run-off could be traced from the presence of clay particles as well as of weathering resistant minerals such as rare earth phosphates and zircons. However, the most abundant heavy metal carriers in CSO samples were the sulfide particles eroded from sewer sediments. SEM microanalyses revealed that the evolution of their abundance closely follows the flow-rate dynamics. This would agree with a pattern of sediment resuspension and redeposition as flow rate changes in the combined sewer during the rain event.

The molecular fingerprint of the organic matter in the sedimentary fraction allowed us to (i) distinguish different organic contributions (higher plants, petroleum byproducts, detergents, combustion byproducts, ...) and (ii) to follow their relative proportion during the storm event.