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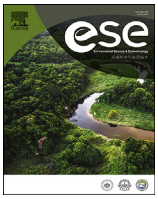

Discussion

Synergies from off-gas analysis and mass balances for wastewater treatment — Some personal reflections on our experiences

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A B S T R A C T

Looking back at over a decade of research by herself and her group, the author advocates the added value of gas phase measurements and the application of mass balances, as well as the synergetic benefits obtained when combining both. The increased application of off-gas measurements for greenhouse gas emission monitoring offers a great opportunity to look at other components in the gas phase, particularly oxygen. Mass balances should not be strictly reserved for modellers but also prove useful while conducting lab experiments and studying full-scale measurement data. Combining off-gas measurements with mass balances may serve not only to quantify greenhouse gas emission factors and aeration efficiency but also to follow dynamic concentration profiles of dissolved components without dedicated sensors and/or to calculate other unmeasured variables. Mass-balance-based data reconciliation allows for obtaining reliable and accurate data, and even more when combined with off-gas analysis.

1. Introduction

Being trained as a chemical engineer, I have been convinced for decades that mass balances are essential for anything a process engineer may want to do. While changing application fields to environmental technology and, more specifically, biological wastewater treatment during my PhD, I have experienced how the conservation principles proved their worth. When I started my career as a professor over ten years ago, studying nitrous oxide and methane emissions from wastewater treatment was the first novel research line for me to start — we also bought our analyser. We soon found that combining off-gas measurements with thorough mass balance analysis leads to synergetic results.

This manuscript is not an exhaustive nor a complete review of everything a wastewater treatment researcher or engineer can achieve by combining mass balances and off-gas analysis. It is a first-person commentary of a selected number of applications and examples from our own experiences, aiming to indicate the power of these engineer's tools and to point out the synergetic effect of applying mass balances and off-gas analysis together. It is the author's aspiration that the combination of mass balances and off-gas analysis, so far largely unexploited, prove their enormous potential for many researchers worldwide.

Some typical applications of these two engineer's tools are reviewed first: off-gas analysis for greenhouse gas emission monitoring (Section 2) and mass balances to gain fundamental or practical process insight (Section 3). Next, it is demonstrated what can be gained by combining off-gas measurements and mass balances (Section 4). Furthermore, mass-balance-based data reconciliation, possibly combined with off-gas data, is advocated (Section 5). Some general take-home messages conclude this contribution.

2. Greenhouse gas emission monitoring

The contemporary application of gas phase measurements has become increasingly prevalent for quantifying greenhouse gas emissions from wastewater treatment plants (WWTPs), a critical endeavour in pursuing carbon-neutral wastewater treatment. Besides carbon dioxide (CO_2), which is mainly related to WWTP energy consumption and the dosing of fossil-derived fuels, WWTPs also emit nitrous oxide (N_2O) and methane (CH_4). Nitrous oxide and methane are very strong greenhouse gases, having a global warming potential over a 100-year time horizon of 298 and 34 CO_2-equivalents, respectively [1].

Measuring gas–phase concentrations at full-scale installations is relatively simple when dealing with a covered WWTP, in case you can simply measure off-gas concentrations and flow velocities in

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the exhaust pipe. However, most WWTPs consist of largely uncovered unit processes that require a floating hood and involve challenges to sample representative parts of the WWTP — typically the aeration basin. The gas phase analyser measures multiple components, such as N_2O, CH_4, CO_2, and O_2. The choice of measured variables and the measurement range is flexible and can be agreed upon with the manufacturer — other typical gas phase components measured are NO and NH_3.

We conducted the first-of-a-kind long-term, online monitoring campaign at a full-scale WWTP over ten years ago. We measured for over 16 months at the Kralingseveer municipal WWTP, Rotterdam, the Netherlands. Nitrous oxide emissions showed a very high variability overtime on a daily and monthly scale [2]. The very high diurnal and seasonal variability of the nitrous oxide emissions has clear implications for the required sampling strategy, which also depends on the goal: to reliably determine the emission factor, long-term sampling is indispensable — be it not necessarily online, whereas the more detailed study of nitrous oxide emission and formation dynamics and their relation with process variables requires high-frequency (online) sampling. Guidelines for planning adequate sampling campaigns, taking into account the relation between the obtained precision as a function of the number of samples (length of the campaign), are given by Ref. [3].

We also quantified methane emissions from wastewater treatment. We pioneered the method of quantifying methane flows across various unit processes in a wastewater treatment plant by employing mass balances, encompassing methane content in each liquid and gas stream [4]. This allowed us to identify important sources and sinks of methane. We found that 25% of the methane from wastewater treatment originates from the sewer system. This methane is generated within the sewer system and enters the WWTP with the incoming wastewater. The extent of its release depends on the type of headwork involved. The remaining major part (75%) of the methane emissions is attributed to the equipment and unit processes associated with biogas production. This includes methane slip in the exhaust gas engine and emissions from the primary sludge thickening, the buffer storage tank after the digestion, and the storage tank after dewatering. Overall, care must be taken to ensure that the avoided CO_2-equivalents associated with biogas production are not outcompeted by methane emissions. A key mitigation strategy involves covering storage tanks. Furthermore, utilising off-gas streams from those tanks with the highest methane concentrations as combustion air for cogeneration units presents a viable approach for emission reduction. Besides methane sources, the application of off-gas analysis and mass balances also allowed us to identify an important methane sink: 80% of the methane that enters the biological reactor is converted, more specifically, in the aerobic part of the biological tank. This phenomenon, which is most likely attributed to aerobic methane-oxidizing bacteria, could be further exploited by adapting the reactor design and operation [5].

The overall carbon footprint of the full-scale WWTP under study [6] was dominated by nitrous oxide emissions (78%), while methane emissions also took up a substantial share (14% — at the time still considering the relatively lower Intergovernmental Panel on Climate Change (IPCC) 2007 [7] impact factor of 25 CO_2-equivalents for methane). The direct CO_2 emissions were relatively less important and amounted to 8%, a number that is strongly influenced by a country's energy mix. The emission factors quantified for methane and nitrous oxide amounted to 1.1% (kg CH_4 per kg COD_{influent}) and 2.8% (kg N_2O-N per kg TKN_{influent}) and are both higher than the ones used in the IPCC 2006 report [8], being 0.85 and 0.035, respectively. The findings from our study ([2,4,6]) were used in the 2019 Refinement to the 2006 IPCC Guidelines for national greenhouse gas inventories [9].

Over the last decade, several full-scale greenhouse gas emission monitoring campaigns have been conducted (e.g., Refs. [10–13]). While the first studies were mainly conducted by academics, in recent years, a lot of interest has arisen from utilities and industries, driven by legal and/or sustainability incentives to decrease the carbon footprint of wastewater treatment. Consequently, gas–phase measurements have become increasingly commonplace in WWTPs. However, we believe much more could be gained from gas-phase measurements when combined with mass balances and considering multiple components simultaneously, as will be pointed out in the following sections.

3. Mass balances

Mass balances are an engineer's best friend, offering a multifaceted approach to validating measurements over (combinations of) unit processes (see also section 5.), calculating conversion rates (as exemplified in section 2 for the calculation of methane fluxes), and fostering deeper scientific understanding. An interesting application example of the latter is the identification of the anammox reaction stoichiometry. The anammox reaction stoichiometry experimentally determined by Strous et al. [14] has been widely recognised, and every researcher who ever studied the anammox process has the numbers 1.32 and 0.26 in mind, referring to the nitrite:ammonium consumption and the relative nitrate production compared to ammonium consumption (molar) ratios, respectively. Nevertheless, significantly different stoichiometries for the anammox reaction have been determined by other researchers, e.g., Lotti et al. [15]. Starting from (elemental and redox) mass balances and setting up the overall metabolic reaction as the combination of catabolic and anabolic reactions, the overall anammox stoichiometry can be calculated as a function of the biomass composition ($CH_xO_yN_z$) and the yield coefficient (Y^{Met}_{X/NH_4}) (equation (1) [16], all components in mole units):

$$\frac{1}{Y^{\text{Met}}_{X/NH_4}}NH_4^+ + \left(\frac{4+x-2y-3z}{2} + \frac{1}{Y^{\text{Met}}_{X/NH_4}} - z\right)NO_2^- \rightarrow \left(\frac{1}{Y^{\text{Met}}_{X/NH_4}} - z\right)N_2 + \left(\frac{4+x-2y-3z}{2}\right)NO_3^- + CH_xO_yN_z \tag{1}$$

While the anammox stoichiometry proposed by Lotti et al. [15] matches the generalised anammox stoichiometry (equation (1)), this is not the case for the stoichiometry of Strous et al. [14], which basically indicates that the latter does not comply with first principles. The numbers 1.32 and 0.26 are not as universally true as often accepted. The stoichiometry derived by Strous et al. [14] was likely compromised by measurement errors and an incomplete enrichment degree of anammox bacteria. A revised anammox stoichiometry, based on the findings of Strous et al. [14] but matching the general stoichiometry has been proposed by Jia et al.

[16], according to a general procedure which can easily be applied for other experimental studies — a spreadsheet tool to this end has been provided [16].

Mass balances can also aid in quantifying the relative contribution of various conversion pathways taking place simultaneously by measuring component conversion rates and, at the same time, taking into account the reaction stoichiometries. For instance, we applied mass balances to study the effect of influent organic matter on the performance of a lab-scale granular sludge anammox reactor [16]. Four variables were measured in the system: ammonium, nitrite, nitrate, and organic carbon. The following conversions could occur: the anammox conversion, heterotrophic denitrification, and decay of both anammox and heterotrophic biomass. Considering denitrification as a two-step process with nitrite as an intermediate, this makes a set of five reactions in total, which would be one unknown to many. By measuring the biomass concentration in the reactor and using the anammox decay coefficient anammox from the literature, one of the reaction rates could be estimated, leaving four reactions to be calculated based on the four measured components. Overall, the mass balance analysis revealed that approximately 18% of the nitrate produced from the anammox conversion was reduced to nitrite by heterotrophic denitrification, resulting in a higher overall nitrogen removal than in the absence of organic carbon. The demonstrated method (with spreadsheets) [16] can be applied straightforwardly to experimental data from other studies.

4. Combining off-gas measurements and mass balances

Off-gas measurements are increasingly applied at WWTPs to quantify greenhouse gas emissions, mainly nitrous oxide and, in some cases also, methane. The author advocates that simultaneously monitoring other components in the gas phase, particularly oxygen (O_2) and CO_2, brings substantial benefits in plant performance monitoring and should, therefore, be considered part of standard practice.

Why so much interest in off-gas analysis? First, multiple components can be measured with the same gas phase analyser device, including component-specific analysis, which does not make up a major fraction of the total cost. While the investment costs for a gas phase analyser may be substantial, maintenance costs are relatively low due to limited fouling and corrosion effects — the gas phase is typically 'cleaner' than the wastewater itself. Moreover, the gas sample is typically well-mixed and has been in contact with a large volume of water, which makes it inherently easier to have a representative sample.

What do we learn from off-gas analysis? Typically, air is provided to wastewater treatment processes to provide oxygen to the microorganisms performing the biological conversions. As the air bubbles rise, their oxygen concentration decreases while carbon dioxide and nitrogen gas concentrations increase. The composition of the air bubbles, as measured through the off-gas, thus reflects the ongoing biological processes. So, while monitoring greenhouse gas emissions through off-gas measurements, it is very worthwhile to closely examine the off-gas composition.

To prove my point, let me explain through examples what can be achieved by combining off-gas measurements and mass balances. First, liquid−gas transfer rates can be calculated from gas phase mass balances based on the measured in- and outgoing gas phase concentrations and the gas in- and outflow rates [17]. This allows, for instance, to monitor the oxygen absorption and carbon dioxide emission rates (i.e., negative and positive liquid−gas transfer rates, respectively), which show a comparable trend as they are coupled in the oxidation of organic carbon oxidation and nitrification — even though carbon dioxide is not produced during nitrification as such, it results in carbon dioxide emission because of proton production and the associated shift in the CO_2/bicarbonate equilibrium. Nitrous oxide and methane emission rates can also be directly derived from off-gas measurements. Even though liquid−gas transfer rates can be calculated from off-gas analyses for both continuous and cyclically operated systems, they can be more easily related to the processes in the liquid phase and, therefore, provide more process insight in the case of cyclic reactor operation. For instance, nitrous oxide emissions in an aerobic granular sludge reactor showed two peaks each cycle, attributed to different production pathways, and methane emissions showed a stripping profile [17].

Secondly, off-gas analysis can be applied to monitor WWTP aeration performance. The oxygen transfer rate (OTR), i.e., the liquid−gas transfer rate for oxygen, is determined from measured oxygen concentrations in the gas phase and gas flow rates. From the OTR, two important parameters characterising the aeration performance can be calculated: the oxygen transfer coefficient and the oxygen transfer efficiency. The oxygen transfer coefficient ($k_La_{O_2}$), is calculated as the OTR per liquid volume relative to the difference between the dissolved oxygen concentration in the reactor (which needs to be measured) and its equilibrium concentration. The oxygen transfer efficiency (OTE) expresses which fraction of the injected oxygen mass flow is transferred from the gas phase to the liquid phase, i.e., taken up by the bacteria. The higher the $k_La_{O_2}$ and the higher the OTE, the better the aeration performance. Baeten et al. (2021) investigated the aeration performance of an aerobic granular sludge reactor and found that the oxygen transfer efficiency increases along each cycle. This finding was confirmed by long-term data analysis, analysing the average aeration performance increase in time, along each cycle, for 175 batch cycles of an aerobic granular sludge process [18]. A similar increase was reported along the tank for an activated sludge process [19] — the spatial component similar to the time component in the aerobic granular sludge process cycle. The increase in the aeration efficiency in time/space is the decrease in surfactants, specifically the soluble biodegradable organic carbon [18].

Thirdly, the application of off-gas analysis and mass balances allows the calculation of dissolved gas concentration dynamics, even without liquid phase sensors at hand. This application is particularly interesting for calculating the concentration of nitrous oxide and methane in the liquid phase, for which dedicated sensors are often unavailable and/or their application is limited to research purposes because of their fragility and limited lifetime. Over the years, we have developed and demonstrated two main approaches for monitoring dynamic dissolved gas concentration profiles. The first one is the gas stripping method [20]. In this method, a liquid sample from the reactor is transferred to a stripping flask. Here, the dissolved component is stripped out, and the resulting gas phase concentration profile is used to calculate the original concentration in the liquid (see the tutorial provided with [21]). The second approach [17] calculates the dissolved concentration of a component, e.g., N_2O, from its liquid−gas transfer rate (calculated from the gas phase mass balances for measured gas phase concentrations and the gas flow rates) and from its liquid−gas transfer coefficient (e.g., $k_La_{N_2O}$). This estimation is typically estimated based on the assumed relation (involving the diffusion coefficients) with the oxygen transfer coefficient ($k_La_{O_2}$), on its turn requiring a liquid phase oxygen concentration measurement — which is quite common.

Finally, the integration of off-gas analysis with liquid phase measurements significantly enhances the capability to close mass balances and calculate unmeasurable variables. More specifically, the mere addition of oxygen gas phase concentration measurements, besides available liquid phase concentration measurements for ammonium and nitrate, enabling calculate the amount of oxygen used for organic carbon conversion and nitrification and the amount of nitrogen denitrified and even the sludge production — at least for cyclically operated reactors [17]. While the latter condition is indeed important, it may be less restrictive than it seems, given the strong advance in aerobic granular sludge reactors, besides other widespread cyclically operated wastewater treatment technologies.

5. Data reconciliation

Finally, I want to advocate mass-balance-based data reconciliation as a fantastic tool to transition from more data to more information. While it is often said that ‘more data leads to more information’, this is not true *per se* but depends greatly on the data quality. Data reconciliation is a proven technique [22] to evaluate the consistency of collected data and detect gross errors. This process involves a statistical procedure of optimally adjusting estimates for variables. These adjustments satisfy the conservation laws, such as mass balances, and other constraints. Consequently, the reconciled data is more accurate than the original values, which is reflected by a smaller uncertainty bound. While proven in the field of (bio)chemical engineering, its application to wastewater treatment processes in general and full-scale WWTPs, in particular, remains limited, even though it holds a lot of potentials [23]. Including off-gas data allows the set-up of additional mass balances, which means that even more variables could be reconciled and facilitates gross error detection [24].

Data reconciliation proves crucial, even indispensable, in performing full-scale measurement campaigns. An example is our application of data reconciliation during the plant-wide quantification of total sulfur mass flows in a full-scale WWTP [25] for all water and sludge streams and the gaseous emission of sulfur as hydrogen sulfide (H_2S). The available measurements consisted of composite samples in the water line, grab samples in the sludge line and continuous gas phase H_2S measurements. To apply data reconciliation, we established several mass balances over different unit processes or their combinations. These balances exhibited a high degree of accuracy, proving that total sulfur is a conservative quantity, as are COD (chemical oxygen demand), total nitrogen, and total phosphorus. This instance marked the inaugural application of data reconciliation for total sulfur components. The correction applied to the data was relatively small, with only a 1—15% difference between the measured and reconciled mean value, underscoring the reliability of our measurements. Furthermore, data reconciliation significantly improved the average precision of the key variables in the water and sludge lines, reducing measurement uncertainty by up to 72%. Regarding the gas streams, they constituted a negligible mass flow of total sulfur, rendering them unverifiable by mass balances. Therefore, to accurately quantify H2S emissions, direct measurements are necessary.

6. Take-home messages

Off-gas analysis and mass balances are essential and synergetic tools for wastewater treatment engineers striving for energy-efficient and low-carbon footprint processes. This paper reflects on our experiences, aiming to incentivise researchers to gain more from their available data, including those obtained from off-gas analysis.

- Energy and CO_2 emission savings can be realized by optimising the aeration performance, quantified through the oxygen transfer rate, oxygen transfer efficiency, and oxygen transfer coefficient.
- Carbon footprint reduction can be established by reducing greenhouse gas emissions. There is a growing imperative to measure emissions of potent greenhouse gases like N_2O and CH_4. Currently, the potential of off-gas monitoring devices, increasingly deployed in full-scale wastewater treatment plants, remains underutilised. Indeed, combining off-gas analysis with mass balances, rather than mere emission factors, liquid—gas transfer rates, and dissolved concentrations of nitrous oxide and/or methane can also be determined. This approach garners more information and deepens our understanding, thereby facilitating the development of more effective strategies for emission reduction.
- Better quality data, characterized by its reliability and comprehensive information content, is obtained through data reconciliation. This includes the application of mass balances to calculate variables which cannot be measured and to increase data accuracy. Adding off-gas analysis measurements to the available data set increases data accuracy and process knowledge.

CRediT authorship contribution statement

Eveline I.P. VOLCKE: Conceptualization, Writing - Original Draft, Writing - Review & Editing.

Declaration of competing interest

The author declares 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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