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# Elemental composition of biogas residues: Variability and alteration during anaerobic digestion

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## ABSTRACT

Biogas production and the amount of thereby incurred digestates increased remarkably in the last decade. Digestates should be used as soil fertilizers to close nutrient cycles. However, knowledge about the elemental composition of digestates from biogas production and element losses or accumulations during fermentation process is insufficient so far. Intending to enlarge the database for the elemental composition of digestates and to investigate element in- and outputs of biogas fermenters, we measured the concentrations of C, N, P, K, S, Ca, Mg, Fe, Mn, Zn, Cu, Pb, Cd, Ni, Mo and Se of digestates and feedstock (ingestates) of four full-scale biogas plants in Germany monthly over a one year period. Ingestates were sewage sludge, fat and mash (SEW1), sewage sludge and fat (SEW2), pig slurry, treacle and food residues (SL) and maize silage (M). We developed a statistical method to calculate the number of required sampling dates which have to be integrated for the calculation of reliable element budgets between ingestates and digestates for the case when information about the amount and composition of the produced biogas is not available. Our results suggest that two (SEW2), five (SEW1, M) and 10 (SL) sampling dates had to be integrated for reliable balances. All fermenters revealed losses of N, most likely due to volatilization of NH<sub>3</sub>. Losses of S (probably H<sub>2</sub>S), Mg (precipitation of struvite), Cd and Zn (precipitation of sulfides) could be detected in some cases. Iron and Mn accumulations can be attributed to attrition of the stirrer.

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## 1. Introduction

Whilst the worldwide growing demand for energy has to be satisfied, the concentration of greenhouse gases in the atmosphere steadily increases due to emissions from fossil fuel combustions [1]. Thus, in recent decades, the urgent need for the production of renewable energy has been well-known.

Biogas production, for example, expanded rapidly in the last 10 years. In Germany, the number of biogas plants increased from nearly 1600 in 2002 to more than 7000 in 2011 [2]. This development is especially driven by the German policy of promoting renewable energies by the “EEG” (Erneuerbare-Energien-Gesetz) in year 2000 and its amendments in the following years. Biogas can replace fossil fuels in power and heat production and can also be used as gaseous vehicle fuel

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### Abbreviations

SEW1	Digestate derived from sewage sludge, fat and mash
SEW2	Digestate derived from sewage sludge and a small amount of fat
SL	Digestate derived from pig slurry and food residues
M	Digestate derived from maize silage and little grass
EEG	German law “Erneuerbare-Energien-Gesetz”

[1]. However, production of energy crops for anaerobic digestion competes against food production for agricultural land. Additionally, the increasing cultivation of energy crops provokes land-use changes and monocultures which often involve environmental disadvantages such as loss of biodiversity or soil erosion [3]. Therefore, wastes and edible organic residues are preferable feedstocks for anaerobic digestion. An additional benefit of waste fermentation is the reduction of odour and mass reduction of landfilled agricultural and municipal waste due to the conversion of organic material to methane (CH<sub>4</sub>) and carbon dioxide (CO<sub>2</sub>) [4].

For a life cycle assessment of biogas production the whole process chain has to be considered [5]. This also includes the recirculation of digestates and nutrients contained therein to the soil where new biomass is cultivated. Albuquerque et al. [5] even assume advantages of digestate fertilization compared to non-fermented organic fertilizers, such as a greater microbial stability and hygiene as well as greater available nitrogen (N). But before utilization as fertilizer or amendment, the elemental composition of digestates has to be known to evaluate fertilizing effects and to prevent contamination of soils. Since anaerobic digestion alters the chemical properties of the initial biomass (ingestate), knowledge about the composition of the initial material is not sufficient. However, investigations about the elemental composition of the resulting digestates are scarce [4,6,7]. Table 1 shows some chemical characteristics of various digestates which can be found in the literature so far. Furthermore, a number of non-peer-reviewed publications about characteristics of digestates exist but are not cited here.

Scanning the literature reveals that mostly only a few chemical characteristics have been investigated so far, with most authors focussing on N, P and K. In particular, heavy metals like lead (Pb), cadmium (Cd) or nickel (Ni) are under-represented. Thus, more extensive data for digestate characteristics are needed. Table 1 also shows that element concentrations of digestates are highly variable and depend on the kind of ingestate. Additionally, the elemental composition of digestates is influenced by digestion process characteristics. During fermentation, varying amounts of N and S can volatilize from the ingestate [16,18] and heavy metals can accumulate in the ingestate due to attrition from the stirrer, for example [15]. A few authors [19,20] studied the fate of nutrients during fermentation and calculated element mass balances. However, again these studies only focussed on N, P and K. Furthermore, data about the mass of produced biogas are not always available as in the mentioned studies [19,20],

i.e. another way to account for the mass losses due to biogas emission from the substrate has to be found to conduct an element balance in this case.

Thus, the aims of our study were:

1. To give an overview of the elemental composition, particularly heavy metal concentrations, of digestates from four full-scale biogas plants in Germany.
2. To investigate the temporal variability of digestates' elemental composition within one biogas plant.
3. To investigate the difference in digestates' elemental composition between different biogas plants.
4. To identify element losses or accumulations during the digestion process.

## 2. Materials and methods

### 2.1. Digestates and element analysis

We collected digestates and ingestates from four biogas plants (wet fermentation) at intervals of one month over one year. The main process parameters of the biogas plants and digestate abbreviations used in this study are given in Table 2.

Digestates and ingestates were freeze-dried and ground in the laboratory. Carbon (C), N and sulfur (S) were measured by a CNS Analyser (Elementar Vario EL III). Additionally, digestates and ingestates were digested with 10 ml 69% HNO<sub>3</sub> (Suprapur, Roth, Karlsruhe, Germany) for 15 min at 180 °C in a microwave pressure apparatus (Mars Xpress, CEM, Kamp-Lintfort, Germany). All samples were digested in duplicate. Concentrations of calcium (Ca), magnesium (Mg), potassium (K), iron (Fe), manganese (Mn), zinc (Zn) and copper (Cu) were analysed with flame AAS (Perkin Elmer Atomic Absorption Spectrophotometer 1100B). Lead, Cd and Ni were determined by a Varian graphite furnace AAS (SpectrAA 880Z), molybdenum (Mo) and selenium (Se) by an ICP-OES (iCAP 6000 Series, Thermo Scientific) and phosphorus (P) by a Continuous Flow Analyser (880 nm wavelength, Skalar San plus system 5120).

### 2.2. Correction of nitrogen analysis

Mineral N is mainly present as ammonium (NH<sub>4</sub><sup>+</sup>) in digestates due to anaerobic conditions in the biogas fermenter. High pH of the digestates can facilitate gaseous losses of N as ammonia (NH<sub>3</sub>) [21]. Ammonia emissions during digestate preparation in the laboratory can therefore lead to an underestimation of N concentrations in the digestates. Previous acidification of the digestates with hydrochloric acid (HCl) could be a suitable method to prevent NH<sub>3</sub> emissions during sample preparations. Exemplarily, we acidified the digestates prior to any further treatment at six different sampling dates and compared N concentrations to those of non-acidified digestates in order to find the average potential for N losses of the digestates and to get a correction factor which we applied to our N concentration data obtained from non-acidified digestates. We added 1 M HCl to the digestates until a pH of 6 was reached. Under these conditions the amount of NH<sub>3</sub> should be decreased to 0.1% and the majority of mineral N should be present as non-volatile NH<sub>4</sub><sup>+</sup>

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