

Mechanisms controlling the release of inorganic contaminants, organic matter fractions, and ammonium from solid landfill waste: pH dependent leaching experiments and geochemical modelling

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ABSTRACT

With the intention of making landfilling more sustainable, three full-scale projects have been set up to investigate the effects of leachate recirculation and aeration on the stabilization of contaminants. Prior to this (an)aerobic stabilization, solid waste was sampled from all three landfills to identify the mechanisms behind contaminant leaching and stabilization, as a reference point for evaluation of the treatment effects at a later stage of the project. Processes underlying the leaching of a wide range of inorganic contaminants were identified using a combined approach of pH dependent leaching experiments, organic matter fractionation, and geochemical modelling. We additionally focused on organic matter, being an important surface controlling the leaching of inorganic elements, and ammonium, for which compliance with intended environmental limit values for sustainable landfill management is challenging for many landfills. Three types of pH dependent leaching behaviour could be identified for the investigated elements: non-reactive behaviour, anionic behaviour, and cationic behaviour. Reactive elements, including ammonium, showed maximum dissolved concentrations around the upper and lower ends of the pH range, while leaching of non-reactive elements was pH-independent. Organic matter became increasingly soluble with increasing pH, while its relative composition became increasingly enriched in fractions reactive for metal binding, most notably humic acid, only for the most organic landfill. The organic signature of the waste samples, together with their varying organic matter content, resulted in distinct differences in leaching behaviour between landfills. These differences result from the seemingly strong influence of organic matter on the pH at which minimum solubility of elements occurs, being lower for elements with higher organic matter binding affinity, an effect being more pronounced for more organic-rich samples. The model, based on independently determined generic sorption parameters, provided adequate descriptions of most elements, yet arsenic and ammonium were poorly described due to mechanisms not captured by the model while chromium was inadequately described due to limitations of the current binding parameters. As the three landfills have been shown to differ strongly with respect to waste properties (e.g. organic versus. inorganic) an important outcome of this study is that landfill-specific combinations of proposed treatment strategies (aeration, leachate recirculation) are necessary to reduce the leaching of specific contaminant groups to below limit values and are anticipated to be most effective for less mature landfills characterized by a lower ratio of solid to dissolved OM.

1. Introduction

In striving towards more sustainable waste management, many governments are striving to decrease the fraction of waste ending up in landfills, e.g. the waste hierarchy of the European Waste Framework directive proclaiming preferring re-use and recycling over disposal (European Parliament and Council of the European Union, 2008).

Nevertheless, landfills remain necessary for untreatable waste and residues. Landfills are a long-term environmental threat due to gaseous emissions and the generation of leachate rich in (in)organic contaminants, which – if not properly managed – can enter the underlying soil and pollute ground- and surface waters (Kjeldsen et al., 2002; Moody and Townsend, 2017; Powell et al., 2016; Siddiqua et al., 2022). To prevent this pollution, landfill closure is in many countries followed by

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legally prescribed “eternal” aftercare, which involves leachate treatment and the placement of artificial barriers to restrict flow (Laner et al., 2012; Scharff et al., 2011). Eternal aftercare is perceived an unsustainable practice, which shifts its associated costs to future generations. In an effort to explore more sustainable practices in the Netherlands, a so-called “Green Deal” was signed between the Dutch government, the Dutch Sustainable landfill Foundation, and landfill operators in 2015 (Brand et al., 2016). This agreement instigated three landfill-scale projects in 2016, which investigate the effects of long-term “active treatment” on existing landfills, i.e. leachate recirculation (Reinhart and Al-Yousfi, 1996) and/or aeration (Ritzkowski and Stegmann, 2012). The intended effect of this treatment, which is anticipated to last for 10 years, is to reduce contaminant concentrations in the leachate through flushing, degradation, or retention in the solid phase. If effective, the emission potential of the landfill is expected to be reduced and thus its aftercare period significantly shortened (Berge et al., 2009; Rich et al., 2008). The embedding of the projects within the Green Deal and a large associated research programme ensures an extensive monitoring programme of emissions in the leachate and gas phase, together with sampling and characterization of the solid waste at the beginning and end of the treatment period. This approach results in the acquisition of a large data set, with high temporal resolution, over an extended period of active treatment on a landfill scale. Ultimately, leached contaminant concentrations need to comply with legally derived site-specific emission target values for the projects to be a success and for the landfills to be released from their aftercare (Brand, 2014; Brand et al., 2016; Dijkstra et al., 2018).

Taking effective steps in reducing contaminant leaching, and having a scientific underpinning that the thus obtained reductions are sustainable, requires an understanding of the underlying processes. The determination of leaching behaviour at different pH levels has proven its value in the identification of leaching processes in both natural materials (Cappuyns and Swennen, 2008; Carter et al., 2009; Dijkstra et al., 2004, 2009; Uddh-Söderberg et al., 2024) and waste materials (Astrup et al., 2006; Cappuyns and Swennen, 2008; Dijkstra et al., 2006a; Komonweera et al., 2015; Quina et al., 2009; Van Der Sloot et al., 2017; H. Zhang et al., 2008). As shown in several of these studies, further identification of underlying mechanisms can be accomplished through complementary geochemical modelling to calculate the speciation of targeted elements by taking into account mineral formation and adsorption to different reactive surfaces. This complementarity has previously allowed for the distinction of elements showing three different types of environmental behaviour: non-reactive, anionic, and cationic (Hyks et al., 2009; Luo et al., 2019; Van Der Sloot and Dijkstra, 2004; Zhang et al., 2008). While elements with non-reactive behaviour are characterized by a limited interaction with the solid matrix and hence no pH dependence, elements with anionic and cationic behaviour both show a maximum in dissolved concentrations at the upper and lower ends of the pH range; the minimum solubility occurs around pH 5 for anions and at variable but higher pH for elements with cationic behaviour. This behaviour is related to the pH dependent charge and solubility of metal (hydr)oxides and organic matter (OM), protonation and hydrolysis of the element, and precipitation of mineral forms. Cations and particularly anions can strongly bind to metal (hydr)oxides (Brandely et al., 2022; Gustafsson and Antelo, 2022; Tibergh et al., 2020), which shift from a positive net surface charge below the point-of-zero charge (PZC) to a negative net charge above the PZC (Hiemstra and Van Riemsdijk, 2009; Kosmulski, 2016), resulting in enhanced cation adsorption and decreased anion adsorption with increasing pH. The solubility of these metal (hydr)oxides increases below pH 5 and above pH 8 (Liu and Millero, 1999), where their (partial) dissolution results in a release of adsorbed ions into solution. Cations can furthermore strongly bind to OM, which has a negative charge already at low pH and becomes increasingly negatively charged with increasing pH (Milne et al., 2001; Van Zomeren et al., 2009). The enhanced solubility of OM with increasing pH (Dijkstra et al., 2009) results in an increased

solubility at higher pH for cations with an affinity for OM binding. Additionally, protonation of anions at low pH – reducing their negative charge – and increasing complexation of cations with hydroxyl ions (OH^-) at high pH – reducing their positive charge – generally reduces binding for elements of both groups to these reactive surfaces and partially off-sets the aforementioned effects of pH on their adsorption. Finally, potentially solubility controlling mineral phases of several anions typically have a solubility minimum around pH 5, e.g. Wulfenite (PbMoO_4) (Conlan et al., 2012; Vlek and Lindsay, 1977); for cations, the pH at which potentially solubility controlling minerals are stable varies, yet for common hydroxide and carbonate minerals is generally above pH 7.

To our knowledge, the combination of pH dependent leaching, detailed organic matter fractionation as a function of pH, and geochemical modelling has not previously been applied to landfilled solid waste. The high OM content present in landfill waste (Östman et al., 2006) is expected to result in a high importance of OM solubility. The OM solubility is known to be strongly affected by pH (Dijkstra et al., 2004, 2009), yet a thorough investigation of the pH dependent solubility of specific OM fractions relevant for element binding, i.e. humic acid (HA) and fulvic acid (FA) (Hayes and Swift, 2020), has to our knowledge so far only been published for municipal solid waste incinerator bottom ashes (Dijkstra et al., 2006b). In addition, geochemical modelling calculations were found to be most sensitive to the description of OM binding properties in soil with varying organic matter contents, ranging from 0.3 to 12 % (Wiersma et al., 2025). Instead, oftentimes general assumptions are made regarding the reactive fraction of OM (e.g. 50 % of dissolved OM (DOM) is HA (Dijkstra et al., 2009) or 100 % of DOM is FA (Uddh-Söderberg et al., 2024)), while the DOM composition is likely to shift with changing pH considering the differences in pH-dependent solubility between the humic fractions (Van Zomeren and Comans, 2007). Additionally, there have not yet been any published investigations on the pH dependent solubility of ammonium (NH_4^+) from landfilled waste, despite that it is considered one of the contaminants of highest concern in landfill waste. Ammonium accumulates in the leachate due to constant release during waste degradation and the lack of a removal pathway under anaerobic conditions (Berge et al., 2005). The generally high NH_4^+ concentrations in the leachate require major reductions to comply with the current emission target values (Brand et al., 2016). Insights into the mechanisms that control NH_4^+ partitioning between the solid waste and leachate are thus of major importance to enable the development of effective measures to reach the defined target values.

This study explores the underlying geochemical processes governing contaminant release from solid waste of three different landfills, using the combined methodology of pH dependent tests with geochemical modelling. Particular focus is placed on the solubility of OM and its reactive HA and FA subfractions, enabling novel insights in the speciation of these important fractions and enabling a more complete model parametrization as opposed to the commonly made assumptions, as well as on NH_4^+ . To this end, waste samples from all three landfills were taken prior to the start of active treatment. Similar sampling will be conducted at the conclusion of the pilot to investigate the development of waste stabilization and improvement of leachate quality. The solid waste was subjected to pH dependent tests, where underlying mechanisms were further explored using geochemical modelling. Ultimately, the insights gained through the results of this study are linked to prospects of reducing environmental emissions in the three different landfill pilots based on different treatment strategies.

2. Materials and methods

2.1. Solid waste samples

The sampling procedure developed for this project is outlined in detail in (Lammen et al., 2019). In short, waste sampling was conducted

at the start of the pilot experiments in 2016 and 2017 for all three pilot landfills, a thorough description of which is given by Yi et al. (2025): Braambergen 11 north (Bra), which was landfilled from 1999 until 2008 with predominantly contaminated soil, soil remediation residues, and construction and demolition (C&D) waste, and which was subjected to aeration starting from 2017; Kragge (Kra), which was landfilled from 1997 until 2007 with predominantly commercial and domestic waste as well as C&D waste, and which was subjected to leachate recirculation starting from 2018; Wieringermeer (Wie), which was landfilled from 1992 until 2003 with predominantly commercial waste, composting waste, and contaminated soils, and which was subjected to aeration starting from 2017. Due to the large scale of the pilot compartments (4, 5, and 3 ha for Bra, Kra, and Wie, respectively) and the large heterogeneity inherent to landfill waste, large amounts of waste had to be properly sampled, homogenized, and pre-treated to obtain the highest representativeness that was practically achievable. Consequently, 12 boreholes were drilled per landfill compartment in vertical steps of 2 m using a Sonic Samp drill with a diameter of 88 mm. All landfills are covered with a layer of top soil. For Wie the first 2 m drilling was discarded to not include this top soil, whereas for Kra and Bra this drilling was included as the shallower top soil layer of these landfills (~1 m compared to 2 m for Wie) meant a significant portion of the first drilling consisted of waste. Inclusion of the cover layer likely slightly “diluted” the top samples of Kra and Bra, as the cover layer has a relatively lower content of inorganic elements and reactive surfaces. The samples were mixed at three different depth intervals (dubbed “top”, “mid”, and “bot”), resulting in 9 “mixed” samples total (Table 1). Data on landfill depth and solid waste characteristics are presented in Table 1. After mixing, samples were pre-treated to fulfil experimental criteria – namely a particle size <4 mm – using sieves and a shredder. Portions of 20 kg of the waste were stored at −18 °C until waste characterization and leaching tests as described below.

2.2. Waste characterization

2.2.1. pH static leaching test

The pH static leaching test (NEN-EN 14997, 2015) characterizes the leaching of contaminants as a function of pH. In short, fresh samples, conserved for 5 years at −18 °C, were extracted with ultrapure water (UPW) at a UPW/waste ratio of 10 L/kg in 8 parallel batches at pre-defined pH values. This was done through continuous automatic addition of acid (HCl) or base (NaOH) to vary the pH between 2 and 12; one batch was extracted at the natural pH (i.e. no acid/base addition). Contrary to preliminary experiments (Lammen et al., 2019; Van Raffe et al., 2021), HCl was used instead of HNO₃ in order to not disturb the speciation of NH₄⁺ in the leachates (see Results section). Samples were equilibrated for 48 h under continuous stirring, and subsequently

filtered (0.45 µm) before determining a wide range of inorganic elements using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Optical Emission Spectrometry (ICP-OES) in acidified filtrates. Organic and inorganic dissolved carbon (Skalar San++ segmented flow analyzer), chloride, and ammonium (Discrete Analyzer) were determined in unacidified filtrates. Dissolved OM was further fractionated into HA, FA, hydrophilic acid (Hy), and hydrophobic neutrals (HON) using the rapid batch fractionation method (ISO 12782-5, 2012; Van Zomeren and Comans, 2007). While only HA and FA are assumed to contribute to element binding, Hy and HON are determined for complete OM characterization.

2.2.2. Solid waste characteristics

The solid phase was further analyzed for several parameters required for the geochemical modelling. Solid HA, FA, Hy, and HON were estimated using the rapid batch fractionation method (ISO 12782-4, 2012; Van Zomeren and Comans, 2007). Crystalline and amorphous/nanocrystalline iron (Fe) (hydr)oxides were estimated using a dithionite extraction (DCB) (ISO 12782-2, 2012). Amorphous/nanocrystalline Fe and aluminium (Al) were estimated after an ascorbic acid (asc) extraction (ISO 12782-1, 2012) and an ammonium oxalate (AmOx) extraction (ISO 12782-3, 2012) respectively. Geochemically reactive amounts of contaminants were estimated using the 0.43 M HNO₃ extraction (Groenenberg et al., 2017; ISO 17586, 2016).

2.3. Geochemical modelling

Geochemical modelling was conducted akin to the modelling approach of Dijkstra et al. (2004) but with important differences regarding, amongst others, the representation and parameterization of solid and dissolved organic matter, as described below. Element speciation – including binding to DOM, binding to solid reactive surfaces (Fe- and Al (hydr)oxides, OM, clay), and mineral precipitation/dissolution – was calculated in the ORCHESTRA framework (Meeussen, 2003). Specifics on the used binding models and their relevant inputs are outlined below. General inputs consisted of the fixed setpoint pH, pe (assuming pH + pe = 12, implying overall oxidizing conditions based on preliminary evaluation of pH-dependent leaching behaviour of the redox sensitive elements Fe and Mn and the observed absence of sulphidic conditions in most samples), dissolved concentrations of background solutes, and the total reactive concentration of the to-be-modelled element. Compared to Dijkstra et al. (2004) a more elaborate approach was followed in which elements were each modelled in separate model runs, instead of all elements simultaneously in a single model run. This approach allows for more accurately taking competition effects into account between the element under study and other elements competing for sorption sites. To this end, the to-be-modelled

Table 1
Characteristics of the analyzed waste.

Landfill	Layer	Depth [m - surface]	Natural pH [-]	SHA ^a [g C/kg]	SFA ^a [g C/kg]	HFO ^b [g/kg]	DHA ^c [mg C/l]	DFA ^c [mg C/l]
Braambergen ^d	Top	0–4	7.85	2.26	0.02	17.88	0.03	3.01
Braambergen	Mid	4–8	7.5	1.24	0.34	18.75	0.05	10.79
Braambergen	Bot	8–12	7.5	0.67	0.36	17.35	0.00	12.08
Kragge ^d	Top	0–6	7.5	5.21	0.46	18.75	1.19	15.00
Kragge	Mid	6–12	7.5	4.09	0.80	27.72	13.76	51.02
Kragge	Bot	12–16	7.45	4.38	1.03	25.53	13.61	66.03
Wieringermeer	Top	2–6	7.75	1.76	0.26	14.12	0.08	5.19
Wieringermeer	Mid	6–10	7.55	3.80	0.44	15.38	0.42	16.83
Wieringermeer	Bot	10–14	7.75	2.80	0.46	12.62	0.55	19.85

^a Solid humic acid and solid fulvic acid, determined according to (ISO 12782-4, 2012).

^b Hydrous ferric oxide, calculated using the amount of amorphous (ISO 12782-1, 2012; ISO 12782-3, 2012) and crystalline (ISO 12782-2, 2012) iron and aluminium (hydr)oxides.

^c Dissolved humic acid and dissolved fulvic acid, measured at natural pH according to (ISO 12782-5, 2012).

^d Including approximately 1 m of cover soil.

element had its input set to the total reactive amount; the background dissolved concentrations of all other major elements and competing elements were fixed to their measured dissolved concentrations.

Adsorption to Fe- and Al (hydr)oxides was modelled using the generalized two layer model (Dzombak and Morel, 1990). This approach combines the separately measured amounts of Fe/Al (hydr)oxides into one proxy: hydrous ferric oxide (HFO). Al is combined with Fe due to the limited database of binding parameters for Al (hydr)oxides (Meima and Comans, 1999), and is assumed to behave similarly to Fe oxides (Karamalidis and Dzombak, 2010; Mathur and Dzombak, 2006). As such, amorphous Al and Fe (hydr)oxides (as measured using the AmOx and asc extraction, respectively) are recalculated assuming a molar ratio of 95 g Fe/mol HFO and 84 g Al/mol HFO (Hiemstra and Van Riemsdijk, 2009), and assigned a reactive surface area of 600 m²/g (Dzombak and Morel, 1990). Crystalline oxides are calculated by subtracting the amorphous/nanocrystalline (hydr)oxides from DCB extractable (hydr) oxides, and recalculated assuming a molar ratio of 89 g Fe/mol HFO and 78 g Al/mol HFO, and assigned a reactive surface area of 100 m²/g (Dijkstra et al., 2009; Van Eynde et al., 2020). Total HFO was subsequently corrected for binding to SOM and DOM, based on separate model calculations, and for mineral dissolution, based on dissolved Fe and Al as measured in the pH dependent test and assuming dissolution of amorphous (hydr)oxides. Alterations of the binding affinities listed by (Dzombak and Morel, 1990) are adopted from the following studies: for the elements lead (Pb) (Meima and Comans, 1998a), antimony (Sb) (Meima and Comans, 1998b), molybdenum (Mo) (Gustafsson, 2003), Fe (II), and carbonic acid (Appelo et al., 2002).

Adsorption to OM was modelled using the NICA-Donnan model (Kinniburgh et al., 1996). Model inputs consisted of solid HA, solid FA, dissolved HA, and dissolved FA according to their measured concentrations. All fractions were assumed to have a carbon content of 50 % (Pribyl, 2010). In general, element binding was calculated using the generic binding parameters derived by Milne et al. (2003, 2001). Fe binding to FA was calculated using the binding parameters of Hiemstra and Van Riemsdijk (2006). Tin (Sn) binding to HA was calculated using the binding parameters of (Dijkstra et al., 2009), and Pb binding to FA and HA was calculated using the binding parameters of Xiong et al. (2013). Furthermore, vanadium (V) was set to only form the reduced V (III), which is known to be prevalent in organic environments and to bind strongly to OM (Gustafsson, 2019).

Controlling mineral phases were determined based on preliminary modelling calculations. In a first step, saturation indices (SI) were calculated for all minerals in the MINTEQA4 database together with additional potentially solubility controlling minerals (see Table S1 for a list of added minerals, and their reaction equations in ORCHESTRA) with pH, pe, and dissolved concentrations of elements, HA, and FA as model input. If for at least one pH value the SI was between -1 and 1, the mineral was considered potentially solubility controlling. In a next step, minerals were selected based on their likelihood of presence and/or formation under landfill conditions. Finally, potentially solubility controlling minerals were individually modelled to test how adequately they explained the experimentally observed pH dependent behaviour. An overview of ultimately considered minerals – predominantly carbonate, hydroxide, and silicate minerals – and altered parameter values are given in Table S2.

Preliminary modelling, assuming a significantly overestimated clay content of 20 % based on unpublished data, and using a Donnan approach according to Dijkstra et al. (2009, 2004), showed clay to be of little importance on modelled pH dependent leaching patterns (Fig. A1), in agreement with previous studies (Dijkstra et al., 2004). Therefore, electrostatic adsorption to clay was not further taken into account for this study.

3. Results & discussion

3.1. Waste homogeneity and vertical heterogeneity

A preliminary experiment, in which pH was adjusted with HNO₃ instead of HCl (Van Raffe et al., 2021), showed chloride (Cl) to remain stable for all landfills and layers across the entire pH range (Fig. 1). With Cl being a fully mobile element the limited pH dependent effects are expected, yet the relatively invariant concentrations between each of the parallel batches of the pH dependent test are an indication of the effectiveness of the sample homogenization strategy. Additionally, the consistently lower Cl concentration in each landfills' top layer gives first insights into leaching mechanisms. This lowered concentration may be partly due to inclusion of the relatively Cl-poor cover soil, yet as this soil was discarded for the Wie landfill this is not the sole reason. Presence of older waste of different composition in the deeper layers can be an alternative explanation, yet this would also result in different concentrations in deeper layers for other elements, e.g. Al or Zn, while this is not found. Lower concentrations in the top layers are in some cases also found for other poorly reactive elements, i.e. potassium (K) for Kra and Wie and sulphur (S) for Bra (Fig. A2), as well as DOM and associated elements (as discussed in the following sections). Based on the above considerations, this feature is interpreted as a depletion from the top layer due to down-flushing and subsequent accumulation in deeper layers.

3.2. pH dependent leaching behaviour and speciation

The measured pH dependent leaching is depicted for major and trace elements together with corresponding modelling predictions (Figs. 2 and 6, & A3) and for OM fractions (Fig. 4 & Figure A26). The calculated solid- and dissolved phase speciation of selected elements for the Kra landfill is shown in Fig. 3; solid phase speciation is divided between adsorption to the different reactive solid surfaces and mineral formation; dissolved phase speciation is divided between the free cation or anion in solution, all inorganic species including protonated forms, and adsorption to dissolved HA and FA. Detailed speciation graphs for all elements and all three landfills and their different layers are present in the SI (Fig. A4–A25). A thorough evaluation of all measured substances shows that the three typical types of leaching behaviour, i.e. non-reactive, anionic, and cationic, can also be distinguished for these landfill samples, as further outlined below.

3.2.1. Non-reactive behaviour

Elements with non-reactive behaviour are characterized by a pH independent leaching pattern, which can be observed for Cl (Fig. 1) and to some extent for K, Li, and S (Fig. A2). This pH independence is related to the limited interactions of these elements with the solid waste matrix, resulting in only limited differences between the landfills and landfill layers.

3.2.2. Anionic behaviour

Anionic behaviour is observed for the elements Mo (Fig. 2), arsenic (As), phosphorus (P), and Sb (Fig. A3). The multi-surface model describes this behaviour adequately for Mo and Sb, predicting the formation of Wulfenite (PbMoO₄ (Conlan et al., 2012; Vlek and Lindsay, 1977)) at low pH and particularly for Bra and Wie which are characterized by a lower content of reactive surfaces compared to Kra. At higher pH the model predicts the adsorption to Fe/Al (hydr)oxides for both anions, together with a shift between the free ion and inorganic complexes of Mo in the dissolved phase (Fig. 3, A4, and A12). The model adequately describes the solubility of P at high pH (Fig. A3), predicting the precipitation of Ca-phosphate (Fig. A13), while the pH dependent solubility is inadequately described at low pH. Note that P in Kra shows a more pronounced anionic behaviour compared to Wie and Bra, particularly in the middle and bottom layer. This is likely related to stronger

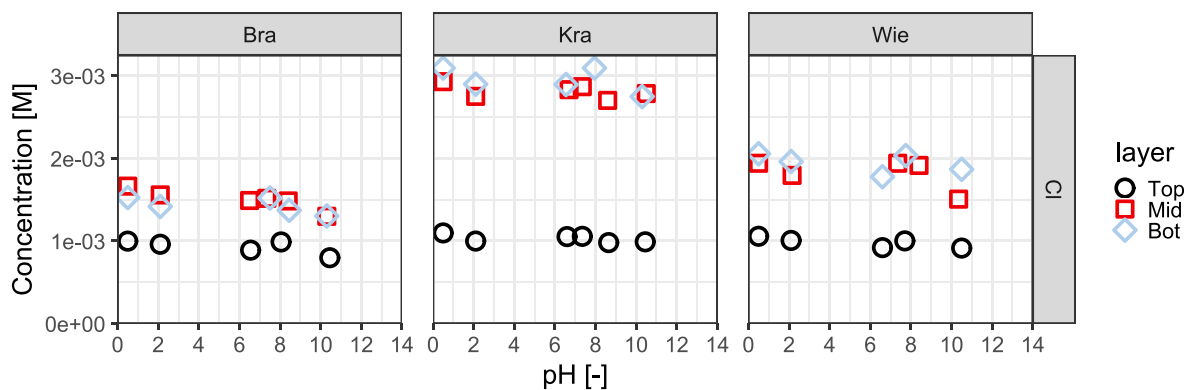


Fig. 1. Concentration of chloride as a function of pH for all three landfills and all three depth intervals, measured in a preliminary experiment in which HNO_3 instead of HCl was used as acid for pH control.

dynamics of OM for these layers, as described below, enhancing competition with P for binding sites and solubilizing organic P (Curtin et al., 2016; McLaren et al., 2015; Yan et al., 2023). For As the model inadequately predicts a cationic behaviour (with a minimum solubility around pH 8) with solely binding to Fe/Al (hydr)oxides (Fig. A11), instead of the observed anionic behaviour. This cationic behaviour was previously ascribed to the possibly incomplete description of competitive adsorption by other ions such as phosphate and fulvic acids (Dijkstra et al., 2009), the latter being even more likely in these highly organic landfill environments with correspondingly high fulvic acid concentrations, as described below.

3.2.3. Cationic behaviour

Cationic behaviour can be observed for Al, barium (Ba), calcium (Ca), cadmium (Cd), cobalt (Co), chromium (Cr), copper (Cu), Fe, magnesium (Mg), manganese (Mn), nickel (Ni), Pb, silicon (Si), Sn, Strontium (Sr), V, and zinc (Zn) (Fig. 2 and A3). The pH at which the concentration minimum occurs is variable; the cationic contaminants in Fig. 2 and A3 are ordered from top to bottom based on the pH at which the solubility minimum is observed. The position of this minimum is the net result of the affinity of the metal for OM, tending to push the solubility minimum to lower pH to follow the pH-dependent solubility of OM (Fig. 4), as described below, and its tendency to form metal hydroxyl species and (hydr)oxide minerals, pushing the solubility minimum to a higher pH. This apparent interplay is supported by the geochemical model (Fig. 3), where for example the higher pH solubility minimum of Mg compared to Cu corresponds with a predicted reduced binding of Mg to OM and a higher prevalence of inorganic hydroxy and carbonate species in the dissolved phase. The influence of OM is further corroborated when comparing the different landfills; the solubility minimum occurs for most elements at significantly lower pH for the Kra landfill compared to the other landfills (e.g. for Cu: pH 7 for Kra, pH 8 for Wie and Bra), related to the relatively high content of OM in the Kra. The Bra landfill, which has a relatively low content of reactive surfaces, instead shows a relatively large importance of mineral dissolution/precipitation, e.g. for Cu (Fig. A6), which is reflected in the solubility minimum occurring at relatively high pH. The large variation in OM content and OM solubility between the samples in this study allows for a distinction between elements based on their relative OM binding affinity and hydrolysing potential within the cationic group.

The geochemical model mostly predicts the pH dependent leaching behaviour of the elements showing cationic behaviour adequately (Fig. 2 and A3), including the corresponding shift in solubility minimum with changing OM affinity. Chromium, known to strongly bind to OM when present as Cr(III) (Fukushima et al., 1995; Gustafsson et al., 2014; Milne et al., 2003; Wen et al., 2018), i.e. the expected form in the reduced conditions and high OM contents of a landfill (Choppala et al., 2018; Jardine et al., 2011, 2013; Rai et al., 1989), has a solubility

minimum around pH 6 (Fig. 2). The pH dependent behaviour of Cr is, however, poorly predicted by the model. Poor model descriptions for Cr (III) have been observed previously and have been attributed to inadequate OM binding parameters (Koopmans and Groenenberg, 2011). These currently available binding parameters have been derived by linear free energy relationships based on the first hydrolysis constant of Cr, rather than from actual binding experiments (Carbonaro and Di Toro, 2007; Milne et al., 2003) and are currently considered to overestimate the binding affinity (Koopmans and Groenenberg, 2011). While improved binding parameters have been derived for the Stockholm Humic Model (Gustafsson et al., 2014), an adequate Cr binding parameter set for NICA-Donnan does not yet exist. Dijkstra et al. (2009) found an adequate model description of Cr leaching from soil samples, by assuming 50 % of DOM consisted of reactive humic substances and assuming that both reactive DOM and particulate OM could be represented by “generic” HA in the model. Contrary to the work of Dijkstra et al. (2009), in the current study the reactivity of DOM is based on measured concentrations of HA and FA. Introducing both HA and FA was previously found to result in an overestimated Cr binding dominated by FA (Koopmans and Groenenberg, 2011). This may also explain our poorer model description compared to (Dijkstra et al., 2009). The adequate modelling of other elements preferentially binding to OM, as discussed later, further emphasizes the specific inadequacy of the used Cr-FA binding parameters.

Cu shows a concentration minimum at a pH of around 7, slightly higher than for Cr, which is adequately predicted for Bra and Wie by the model yet slightly overestimated for Kra (Fig. 2). The predicted solid phase speciation shifts from solely binding to solid OM (SOM) towards binding to metal (hydr)oxides around neutral pH and the precipitation of mineral forms ($\text{Cu}(\text{OH})_2$) at high pH (Fig. 3 & A6). The relative contribution of SOM is highest for Wie; while the SOM content is higher for Kra than Wie, the higher reactive Cu concentration of Kra has likely resulted in a higher SOM site occupancy and shift towards lower-affinity sites, which, together with the relatively higher oxide content of Kra, result in the increased contribution of metal (hydr)oxides. Further modelling with a 10 times lower Cu concentration for Kra resulted in a shift toward a higher contribution of SOM binding (data not shown), confirming the significant effect of Cu loading to the relative contribution of these two reactive surfaces. In the dissolved phase, binding to DOM prevails above pH 6 for all landfills (Fig. 3 & A6), and remains key in controlling the pH dependent solubility.

A concentration minimum around pH 8 can be observed for Fe, which in the solid phase is predicted to be predominantly present in the mineral form ferrihydrite and in the dissolved phase predicted to be bound to DOM with inorganic hydroxide species dominating > pH 10 (Fig. A7). The previously described shift of minimum solubility towards lower pH for Kra is well-pronounced for Fe, owing to its high binding affinity specifically for FA (Hiemstra and van Riemsdijk, 2006). Ni binds

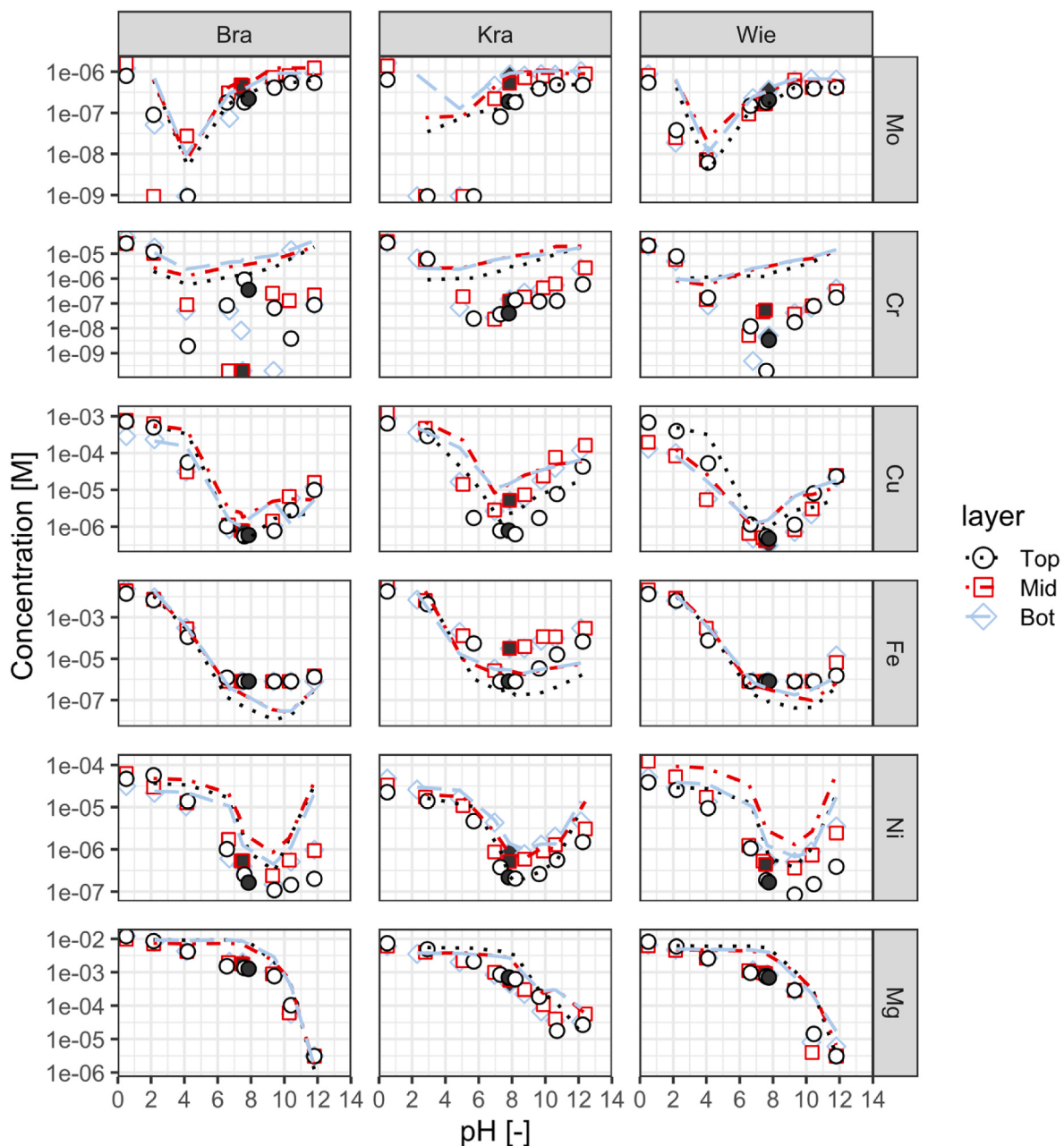


Fig. 2. Concentration of selected major and trace elements as a function of pH for each landfill and depth interval, measured experimentally (markers) and as calculated through multi-surface geochemical modelling (lines). Samples without pH adjustment are coloured dark grey.

to Fe/Al (hydr)oxides across the pH range in the solid phase, while in solution DOM binding is only of importance for Kra and the other landfills show predominantly inorganic hydroxide species formation (Fig. A8). In the presence of the higher amounts of DOM and SOM in Kra, the prediction of Ni at high pH is substantially better than in Wie and Bra. Mg shows only limited binding to the solid phase, instead precipitating as the mineral magnesite above pH 8 and brucite above pH 9; in the dissolved phase Mg is predominantly present as free ion (Fig. A9), except above pH 9–10 where inorganic hydroxide, carbonate, and sulphate species are becoming more important, as well as binding to dissolved HA and FA for Kra. The decreasing binding affinity to OM of these elements again fully reflects the shift in the solubility minimum in Fig. 2.

3.2.4. Organic matter

The pH dependent solubility of OM and its reactive fractions also show anionic behaviour (Fig. 4 and A26). With the reactive fractions of

OM having a variable negative charge, the charging behaviour of metal (hydr)oxides and (de)protonation of OM, resulting in decreasing binding with increasing pH, largely explain the observed behaviour. The deprotonation of OM at high pH further results in an increased repulsion between constituent molecules in particulate OM and consequently an increased solubility (Weng et al., 2002), while the low calcium (Ca) concentrations at high pH (Fig. A3) reduce OM coagulation rates (Kloster et al., 2013; Wall and Choppin, 2003). The increased solubility at high pH is paired with an absolute increase in the

“reactive” components of the DOM (Hayes and Swift, 2020; Olk et al., 2019) for Kra, becoming particularly enriched in HA. For Bra and Wie the relative composition of DOM remains rather invariant across the entire pH range except at pH 2, with a slight enrichment in dissolved HA at high pH for Wie. The relative enrichment in HA at high pH is related to its lower charge density compared to FA (Milne et al., 2001; Van Zomeren and Comans, 2009) and its higher susceptibility to Ca-induced

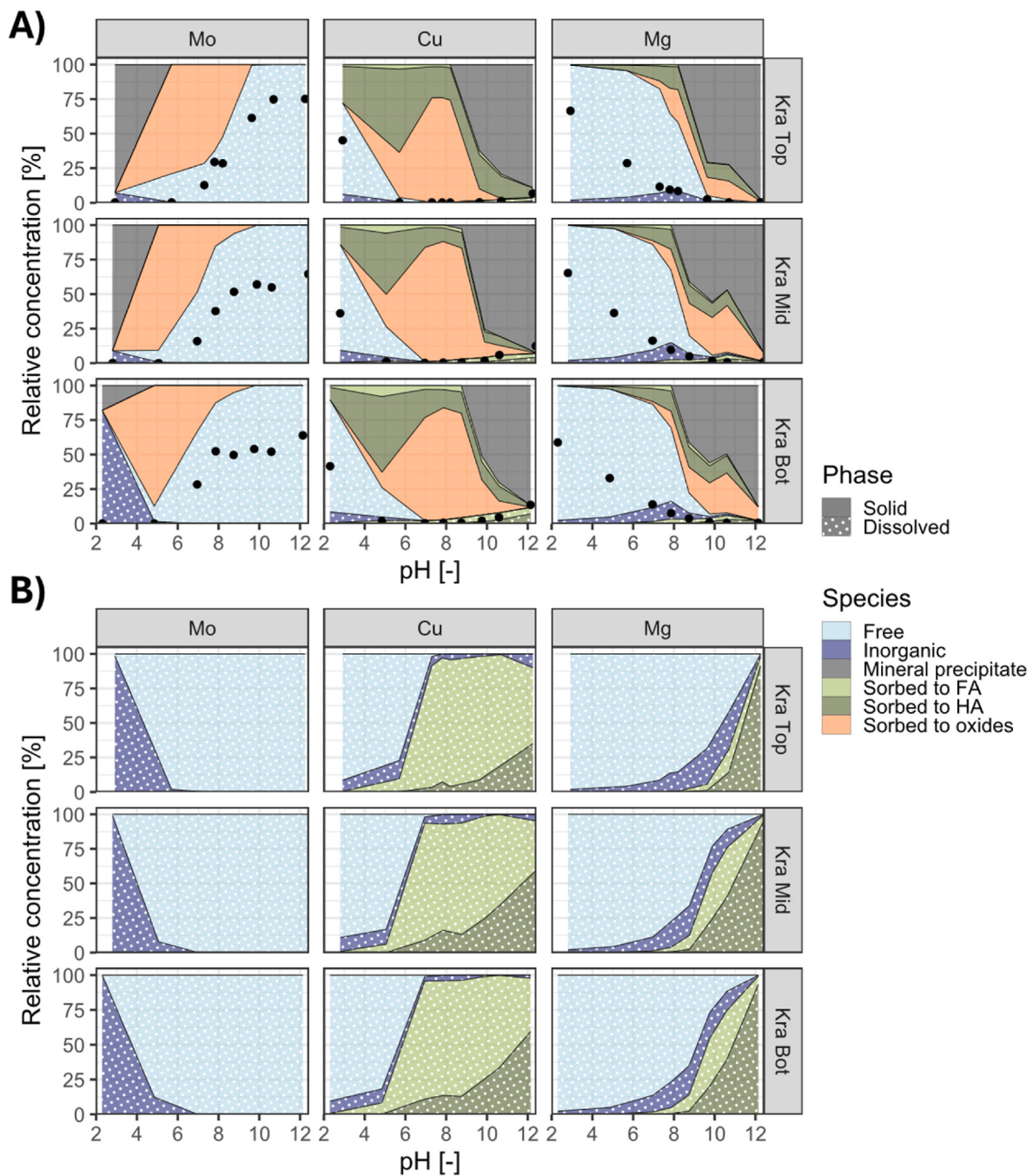


Fig. 3. A) Model calculated solid and dissolved phase speciation of molybdenum (Mo), copper (Cu), and magnesium (Mg) as a function of pH for the different layers of the Kragge landfill. Measured dissolved concentrations are represented by the black circles; B) Dissolved phase speciation of Mo, Cu, and Mg as a function of pH for the different layers of the Kragge landfill. Solid colours represent solid species, white-dotted colours represent dissolved species. Shown mineral precipitates are PbMoO_4 (Mo), $\text{Cu}(\text{OH})_2$ (Cu), Magnesite (Mg; pH 9–10), and Brucite (Mg; above pH 10). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

coagulation relative to FA, Hy, and HON (Kloster et al., 2013; Weng et al., 2002). The increasing concentration of DOM from pH 4 to pH 2 are associated with metal (hydr)oxide dissolution for which HA and FA have a strong binding affinity (Bao et al., 2022; Xu et al., 2020). All landfills show significantly lower DOM concentrations in the top layer, again an indication of potential down-flushing and/or enhanced degradation due to air intrusion.

The observed pH-dependent solubility of OM fractions allows further insight into the validity of often used assumptions for geochemical modelling, e.g. 50 % of DOM being HA (Dijkstra et al., 2009) or 100 % of DOM being FA (Gustafsson et al., 2014; Uddh-Söderberg et al., 2024).

The relatively constant amount of reactive fractions across all pH levels of 40 % (Bra) or 50 % (Wie) closely follows the assumption of 50 % of DOM being reactive, yet considering this reactive fraction to be HA leads to the application of erroneous parameter values as FA was found to be the most prevalent reactive fraction (Milne et al., 2003). Assuming 100 % of DOM to consist of FA would attribute correct parameter values yet instead overestimate DOM reactivity for these samples. Furthermore, the share of reactive fractions is significantly lower for all samples at pH 2, while for Kra at higher pH the share of reactive fractions is significantly higher and dominated by HA. Hence, assuming an invariant reactivity percentage of DOM when modelling across such an extensive

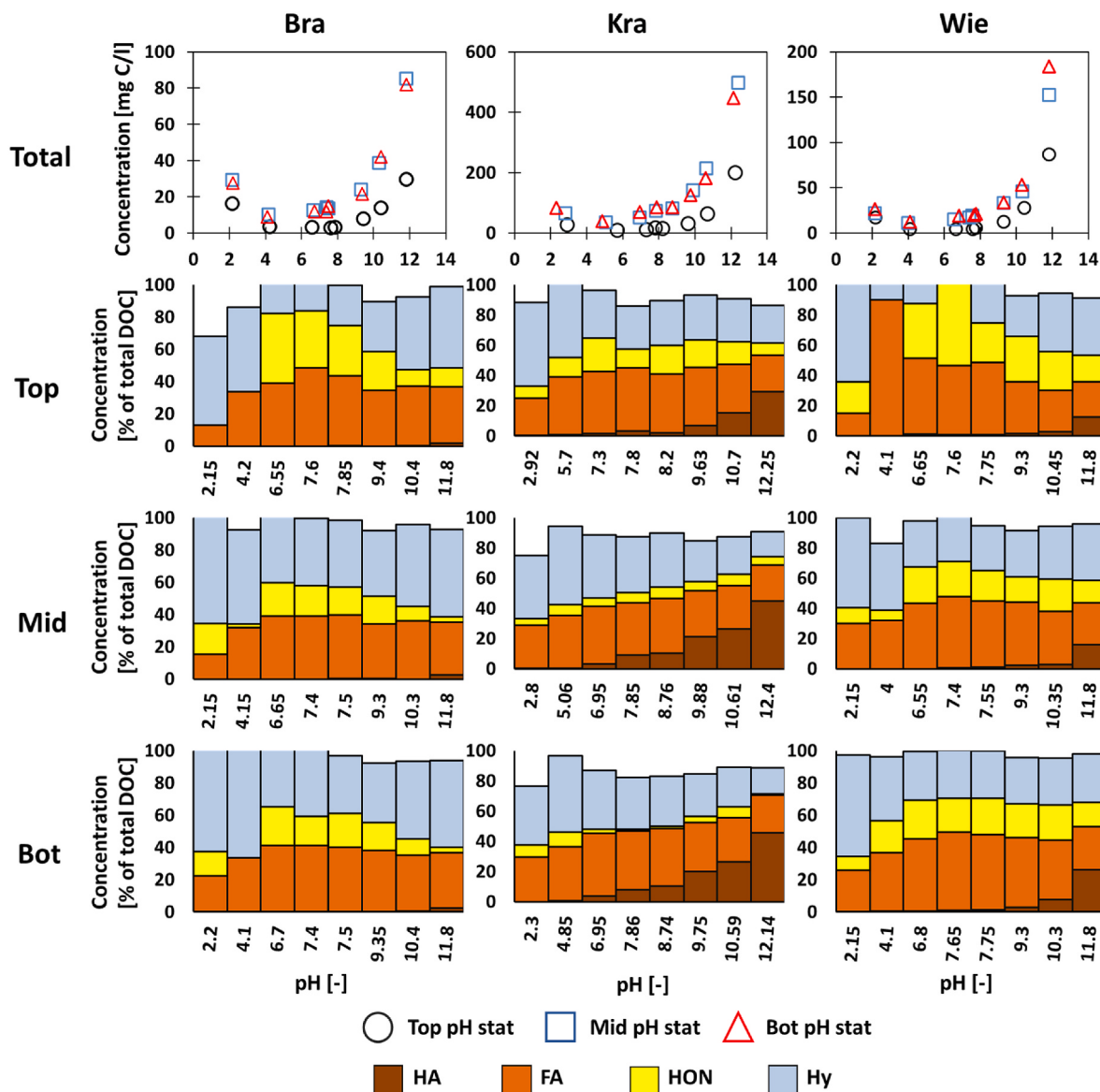


Fig. 4. Concentration of dissolved organic matter (upper panels) and of its subfractions (as a percentage of total DOC) as a function of pH for each landfill and each depth interval. Note the difference in y-axis scale in the upper panels for total DOC concentrations in the three landfills. Also note that the sum of all DOC subfractions for some samples exceeds 100 % relative to separately measured total DOC, or in certain cases is considerably less than 100 %; in both cases this is likely due to analytical uncertainty.

pH range is invalid, particularly for more organic rich samples. The implications of using these simplified assumptions are further investigated through two additional modelling scenarios: 1) assuming measured reactive SOM is all HA, and assuming 50 % of measured DOM is HA; 2) assuming measured reactive SOM is all HA, and assuming 50 % of measured DOM is FA. Results for Cr, Cu, and Pb are highlighted in Fig. 5, with results for all elements listed in Fig. A27. The assumption of 50 % HA, without consideration of Cr binding to FA, significantly improved predictions of Cr solubility (Fig. 5), consistent with the observed overestimated Cr binding to FA discussed above. Nevertheless predictions remain poor relative to Dijkstra et al. (2009), which may be related to the different nature of HA in these waste samples relative to HA in soils. Limited effects were found for Cu (Fig. 5), and for most other elements (Fig. A27). Assuming 50 % of DOM to be HA resulted in a lower Cu solubility at high pH; assuming 50 % of DOM to be FA instead resulted in overestimations. Finally an assumption of 50 % of DOM being HA resulted in significantly worsened predictions for Pb (Fig. 5), as is also the case for Cd and Zn (Fig. A27); assuming 50 % of DOM to be

FA instead showed little effects. While the often used assumptions are thus shown to have limited effects only for most elements, for specific elements considerable impacts can be observed. Additionally, assumptions can have consequences on the calculated speciation, e.g. a predicted larger portion of Cu being bound to reactive surfaces around neutral pH when assuming 50 % of DOM is HA compared to assuming 50 % of DOM is FA (Fig. A28).

3.2.5. Ammonium

The pH dependent leaching of NH_4^+ is akin to that of a reactive cation and shows a concentration minimum of generally around pH 7, and around pH 10 for the middle and bottom layer of Kra (Fig. 6). The most organic layers, i.e. the middle and bottom layer of Kra, have significantly higher NH_4^+ concentrations than Bra and Wie. The top layers of all three landfills have consistently lower concentrations, a sign of down flushing of NH_4^+ or nitrification due to air intrusion. While a clear pH dependence is observed experimentally, the model predicts non-reactive behaviour (Fig. 6). Monovalent ions such as NH_4^+ in soils are predominantly

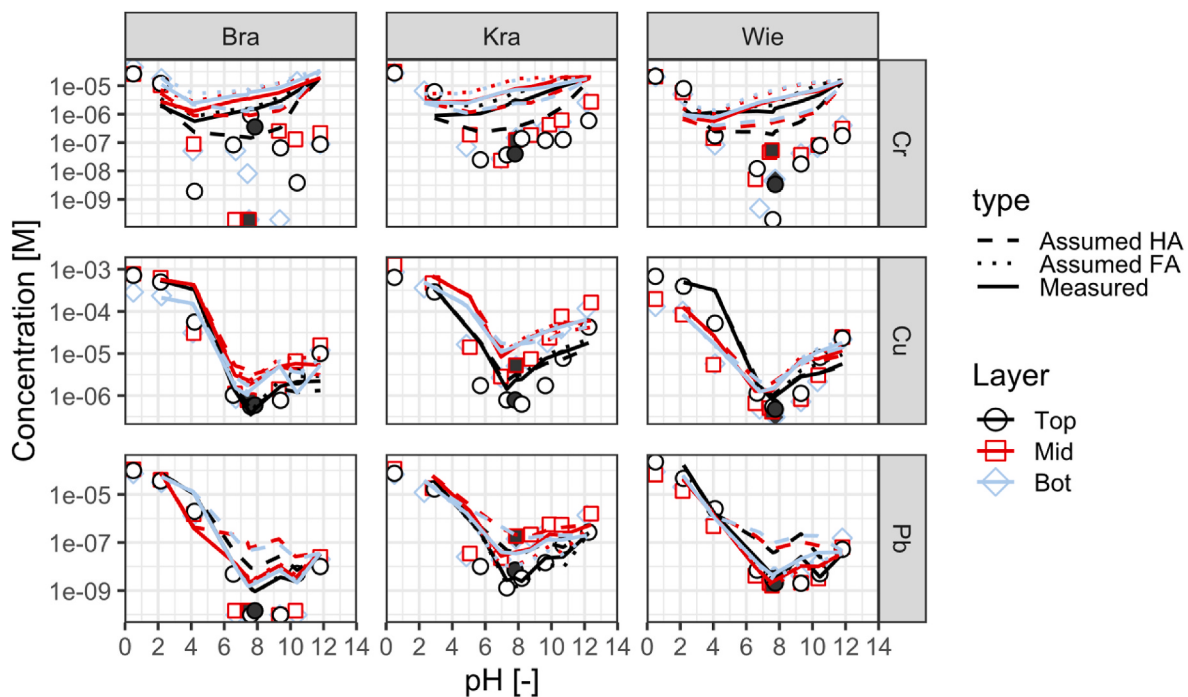


Fig. 5. Concentrations of chromium, copper, and nickel as a function of pH for each landfill and each depth interval, measured experimentally (markers) and as calculated through multi-surface geochemical modelling using measured dissolved and solid HA and FA as input (solid lines), or assuming 50 % of dissolved OM is HA (dashed lines) or FA (dotted lines). Samples without pH adjustment are coloured dark grey.

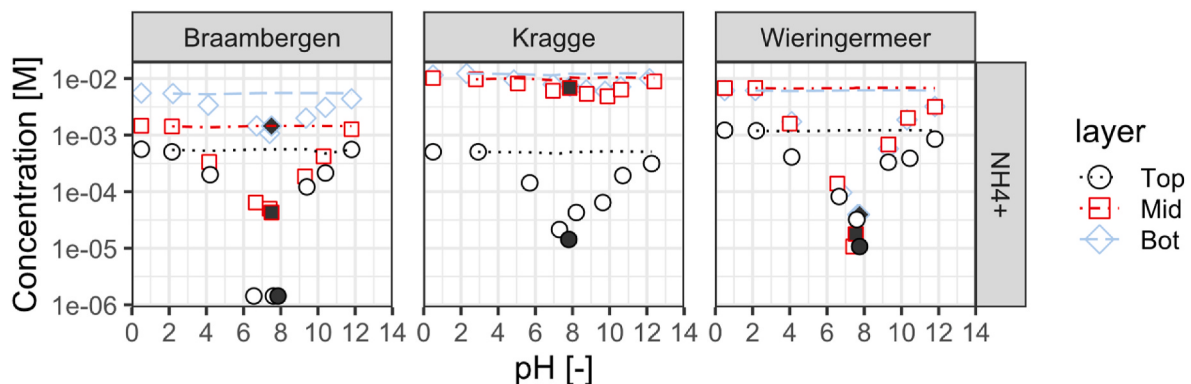


Fig. 6. Concentration of ammonium as a function of pH for each landfill and each depth interval, measured experimentally (markers) and as calculated through multi-surface geochemical modelling (lines). Clay was not considered in the modelling calculations.

associated with clay minerals (Alshameri et al., 2017; Nieder et al., 2011; Nommik and Ahtras, 1982). While electrostatic binding to clay mineral surfaces is not taken into account in the present model, a hypothetical clay content of 20 % was found to have little effect on the predicted pH dependent behaviour of NH₄⁺ in preliminary model calculations that included electrostatic binding to clay minerals (Fig. A1).

The discrepancy between model and experiment indicates that processes explaining the NH₄⁺ behaviour are not sufficiently incorporated in the model. This can particularly concern potential binding to OM (Nommik, 1970; Nommik and Ahtras, 1982; Zhang et al., 2016), selective binding to the frayed edges of 2:1 clay minerals such as vermiculite or illite (Bruggenwert and Kamphorst, 1979; Evangelou and Lumbanraja, 2002; Nieder et al., 2011), or precipitation of minerals other than struvite which was already considered in the current modelling approach (Kim et al., 2007; Kumar and Pal, 2015). Furthermore, nitrification may play a role, as this process has a maximum potential around pH 8 in soils (Sahrawat, 2008; Shammass, 1986) coinciding with the observed concentration minimum (Fig. 6). Nevertheless, NO₃ + NO₂

concentrations remained constant over the entire pH range (data not shown), suggesting that nitrification during the leaching test is unlikely. The potential role of nitrification was further investigated through a time-dependent leaching test (section S1 in the SI) of which the results form a further indication that the experimentally observed pH dependent leaching pattern is more likely to be the result of sorption processes than of nitrification (Fig. 7). If nitrification is the cause of the pH-dependent behaviour, constantly decreasing concentrations with time are expected at neutral pH while concentrations are expected remain constant at high and low pH where nitrification is inhibited. Instead, NH₄⁺ concentrations significantly increased at the upper and lower ends of the pH range, and remained relatively unchanged around neutral pH (Fig. 7). This observation may indicate that rather than nitrification, sorption processes control the pH dependent solubility of NH₄⁺ and are currently significantly underestimated by the model. Desorption from the solid phase is observed at conditions unfavourable for sorption, i.e. low and high pH, and the observed increase is consistent with time-dependent concentration patterns previously observed by

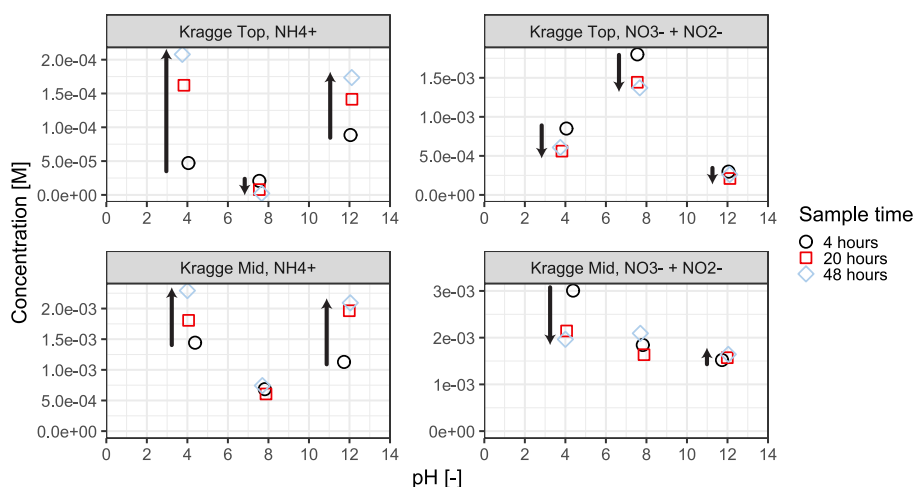


Fig. 7. Concentrations of ammonium and nitrate + nitrite as a function of pH for the top and middle layer of the Kragge landfill, measured as a function of pH (markers). Arrows indicate the change in concentrations with time at the specific pH levels.

(Dijkstra et al., 2006a).

3.3. Implications for active treatment of landfill waste

The elaborate characterization of in-situ sampled waste from three different depth intervals in the three pilot landfills of this study was conducted to explain observed differences in leaching behaviour between landfills, and identify underlying mechanisms controlling leaching of individual elements. As such, this study is meant to provide a baseline that can give direction on how active treatment can be most effectively used to decrease contaminant concentrations in the leachate. The three categories previously identified in different environmental media, i.e. non-reactive behaviour, anionic behaviour, and cationic behaviour, were also identified here for landfill waste. In general, the two treatment approaches, i.e. active treatment by leachate recirculation or aeration, are expected to affect these three contaminant categories differently. Leachate recirculation promotes the throughflow of water and flushing of non-reactive salts and DOM with associated cations from the major flow paths in the waste body. Observable lower concentrations in the top layer of these elements (Fig. 1) already seem to confirm this flushing effect through decades of rain water infiltration prior to sampling and the start of active treatment. Furthermore, both aeration and leachate recirculation are expected to stimulate degradation of particularly easily degradable OM, being most effective during aeration (Ritzkowski and Stegmann, 2012), and ultimately resulting in more stable and humic-like OM with a relative enrichment in FA and particularly HA and a consequently enhanced reactivity of SOM towards metal binding and reduced DOM concentrations (Olk et al., 2019). Additionally, the enhanced aerobic conditions of the aeration treatment likely result in further Fe(II) oxidation and (neo-)formation of Fe/Al (hydr)oxides (Schwab and Lindsay, 1983). Consequently, both treatments are expected to increasingly stimulate the retention of elements that strongly adsorb to reactive organic and metal (hydr)oxide surfaces in the solid matrix, while their mobilization through adsorption to and competition with DOM (Weng et al., 2011) is reduced e.g. Mo (Fig. A4), Cu (Fig. A6), Ni (Fig. A8), and Pb (Fig. A17). The magnitude of active treatment effects may differ between landfills based on the relative importance of these reactive surfaces as observed in the present study. The observed ratio of SOM to DOM in the investigated landfills may already represent different maturation stages, where more mature waste is considered to have a relatively high amount of SOM compared to DOM. Based on our results, Kra can be considered the least mature waste, followed by Wie and Bra. The magnitude of active treatment effects may thus decrease in this order, where particularly for contaminants with a high affinity for OM leachate concentrations may decrease

proportionally to the achieved enhancement of the SOM/DOM ratio.

Interpreting these results, particularly in the context of active treatment, does require consideration of the used methodology. In the present study, waste was sampled from the landfill and directly homogenized. While this has proven to be an effective tool to identify geochemical mechanisms, translating results to the field requires consideration of waste heterogeneity and preferential flow paths in the landfill. Assessment of actual in-situ effects of active treatment requires additional temporal monitoring of landfill leachate and pore water composition (Aharoni et al., 2017; Dahan et al., 2009).

4. Conclusions

Landfilled solid waste samples – sampled from three landfills of different waste origins, at three different depth intervals, and homogenized to acquire representative samples – were characterized on their geochemical leaching behaviour. pH static leaching experiments showed reactive elements, including ammonium, had maximum solubility at highest and lowest pH. The pH of the solubility minimum varied for cationic elements, which resulted from the interplay between the binding affinity for organic matter (OM) and the tendency of the element to form hydroxy species and mineral precipitates. This effect was emphasized for more organic-rich landfills. Increasing pH resulted in increased OM solubility, where the dissolved OM of the most organic landfill became increasingly dominated by humic substances and particularly humic acid with increasing pH. The geochemical model mostly adequately predicted this behaviour, however arsenic and ammonium were poorly predicted, presumably due to binding mechanisms not captured by the model. Poor predictions were also obtained for chromium, due to limitations of the currently available generic binding parameters for this metal in the NICA-Donnan model.

The results of this research confirm that a reduction in the concentration of specific contaminant groups require different treatment strategies for different landfills. Non-reactive contaminants can only be removed from the waste body by enhanced flushing with water, which may be a relevant treatment technique to lower the leaching potential of soluble salts for all of the investigated landfills. Our modelling of leaching processes shows that for contaminants that (strongly) associate with reactive organic and/or mineral surfaces, enhancing the reactivity of organic matter and Fe/Al (hydr)oxides in the solid phase, and reduction of dissolved organic matter in the leachate by aeration of the waste body, can be an effective strategy to reduce the leaching of reactive contaminants from landfill waste. This treatment is anticipated to be most effective in less mature landfills characterized by a low ratio of solid OM to dissolved OM and particularly for contaminants with a

high affinity for OM binding. Overall, this study has shown that landfill-specific combinations of proposed treatment strategies (aeration, leachate recirculation) may be necessary to reduce the leaching of specific contaminant groups to below limit values.

CRedit authorship contribution statement

Frank van Raffe: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Joris J. Dijkstra:** Writing – review & editing, Supervision, Methodology. **Rob N.J. Comans:** Writing – review & editing, Supervision, Funding acquisition.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Frank van Raffe reports financial support was provided by Dutch Research Council. If there are other authors, they 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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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.apgeochem.2025.106503>.

Data availability

Data will be made available on request.

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