



Using isotopes to trace freshly applied cadmium through mineral phosphorus fertilization in soil-fertilizer-plant systems



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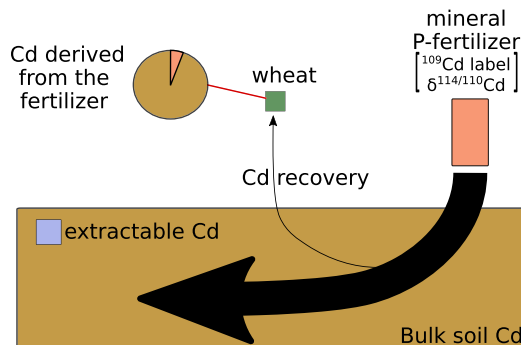
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HIGHLIGHTS

- Radio and stable isotope techniques were applied to trace Cd that was applied with P fertilizer in soil-wheat systems
- The radio isotope techniques provided more robust results than the stable isotope approach at natural abundance
- Less than 3% of the Cd applied with mineral P fertilizer reached the plant shoot and more than 97% of Cd remained in the soil.
- We hypothesize that past fertilizer applications lead to a build-up of a residual Cd pool in soils in the long-term

GRAPHICAL ABSTRACT



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ABSTRACT

Applications of mineral phosphorus (P) fertilizer can lead to cadmium (Cd) accumulation in soils and can increase Cd concentrations in edible crop parts. To determine the fate of freshly applied Cd, a Cd source tracing experiment was conducted in three soil-fertilizer-wheat systems by using a mineral P fertilizer labeled with the radio isotope ^{109}Cd and by exploiting natural differences in Cd stable isotope compositions ($\delta^{114/110}\text{Cd}$). Source tracing with stable isotopes overestimated the proportion of Cd in plants derived from the P fertilizer, because the isotope ratios of the sources were not sufficiently distinct from those of the soils. Despite indistinguishable extractable Cd pools between control and treatments, the addition of P fertilizer resulted in a more negative apparent isotope fractionation between soil and wheat. Overall, the radio isotope approach provided more robust results and revealed that 6.5 to 15% of the Cd in the shoot derived from the fertilizer. From the introduced Cd, a maximum of 2.2% reached the wheat shoots, whilst 97.8% remained in the roots and soils. The low recoveries of the fertilizer derived Cd suggest that continuous P fertilizer application in the past decades can lead to a build-up of a residual Cd pool in soils.

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1. Introduction

The toxic element Cd occurs naturally in soils, but it has been additionally released into the environment through human activities. A major concern is posed by the unintended input of Cd into arable soils through mineral P fertilization (Jiao et al., 2012). Cadmium can substitute for Ca^{2+} in apatite ($\text{Ca}_{10-n}(\text{PO}_4)_6(\text{OH},\text{F})$, Fedoroff et al., 1999; Pan and Fleet, 2002; Sery et al., 1996). The latter is used to produce P fertilizer that contains, compared to the initial product, readily soluble Ca (H_2PO_4)₂ and/or CaSO_4 and unintentionally also soluble Cd (Fedoroff et al., 1999; Prochnow et al., 2008). In Switzerland, the average Cd concentrations of P fertilizers are about 22 times higher than in arable soils (Bigalke et al., 2016; FOAG, 2015). Consequently, application of P fertilizer can lead to Cd accumulation in arable top soils (Bigalke et al., 2016) and to an increase of Cd in staple food products (Grant et al., 2013). In human nutrition, Cd poses a threat since it has a long biological half-life of about 10 years and damages organs such as the kidney (Godt et al., 2006). In this context, the fate of fertilizer-derived Cd and its impact on Cd transfer from soil to edible plant parts needs to be well understood in order to characterize the risk of Cd transfer into the food chain.

Isotope source tracing techniques are commonly used to investigate the fate of nutrients that were applied with fertilizer (e.g., Bosshard et al., 2009; McBeath et al., 2013; Nanzer et al., 2014). Compared to nutrient mass balances, isotope tracing of freshly applied nutrients provides further insights into the fate of nutrients by not only taking into account inputs- and outputs of soil-fertilizer-plant systems but also the fluxes and dynamics within these systems. However, to our knowledge only one study used isotopes (the radio isotope ^{109}Cd) to trace fertilizer-derived Cd in soil-plant systems (Jensen and Mosbæk, 1990). It revealed that 4 to 16% of the Cd added to the soil-lettuce system with NPK-fertilizer reached the plants, such that the majority of the applied Cd remained in the soil. To date, no such study exists for wheat although it often receives Cd through mineral P fertilizers and wheat is one of the main Cd contributors in the diet of western European countries (Filippini et al., 2018; Vromman et al., 2010).

Recent advances in metal stable isotope analysis enabled the use of isotope ratios at natural abundance to trace sources of metals in the environment (Wiederhold, 2015). The application of isotope ratios at natural abundances is particularly interesting since the element sources of the investigated system do not need to be additionally processed for Cd labeling, so that one manipulation step that potentially creates artifacts can be skipped. Furthermore, the approach does not require the purchase of isotope spikes, which significantly increase costs depending on the size of the experiment and the scale of observation. However, the contribution of different sources (e.g., soil, fertilizer) to a sink (e.g., grain) can only be determined with confidence if (i) the sources have distinct isotope ratios, (ii) the extent of isotope fractionation that occurs in the system (e.g., during plant uptake and transport processes) is known or negligible relative to the isotopic difference between the sources and (iii) additional sources can be excluded. Two recent studies reported on the use of stable isotopes at natural abundance to determine the impact of past anthropogenic Cd inputs on the current Cd stocks in soils. Salmanzadeh et al. (2017) showed that the Cd stock of arable soils can be dominated by past applications of mineral P fertilizers. This finding is based on distinct isotope ratios of old and new mineral P fertilizers, which governed the Cd isotope compositions in the soils of a long-term field trial in New Zealand. Imseng et al. (2018) measured Cd isotope ratios for all relevant inputs- (atmospheric deposition, mineral and organic fertilizer, weathering), outputs (crop offtake, leaching) and sinks (soil) of three arable cropping systems. Cadmium mass balances and isotope ratios revealed that long-term processes such as soil weathering and soil-plant cycling determined the current distribution of Cd isotopes in soils whilst recent anthropogenic Cd inputs played a minor role.

Cadmium isotope ratios at natural abundance may also provide novel insights into the fate of freshly applied Cd in soil-fertilizer-plant

systems. Recent studies have shown that the Cd isotope ratios of mineral P fertilizers ($\delta^{114/110}\text{Cd}$) range between -0.15% and 0.15% (Imseng et al., 2018) while the plant-available Cd pool (obtained using $\text{Ca}(\text{NO}_3)_2$ extraction) of arable Swiss soils ranged from 0.12% to 0.54% (Wiggerhauser et al., 2016). Furthermore, no or only minor Cd isotope fractionation was observed between the plant-available Cd pool of the arable soils and whole wheat plants ($\Delta^{114/110}\text{Cd}_{\text{whole plant-extractable}} -0.21$ to 0.03%). Thus, a source tracing approach using stable isotopes at natural abundance would be applicable to selected soil-fertilizer-wheat systems, if the addition of soluble mineral P fertilizer shifts the plant-available Cd pool towards lighter isotopes.

In this study, Cd was freshly applied with a mineral P fertilizer and traced in soil-wheat systems. This was achieved both with the aid of a Cd radio isotope and stable isotope ratios at natural abundance, which were both employed in Cd mass balances calculations. We aimed to (i) test if the natural variation of Cd stable isotopes in the Cd sources of the plant (soil, fertilizer) can be used to trace the distribution of freshly applied Cd in a closed soil-fertilizer-wheat system and (ii) to quantify the fate of freshly applied Cd derived from mineral P fertilization in soil-wheat systems.

2. Material & methods

2.1. Soil-fertilizer-wheat systems

Three soils from Oensingen (Oen), Wiedlisbach (Wied) and Landquart (LQ) were selected to represent typical arable soils for wheat production in Switzerland. The soils have distinct properties (Table A1) such as pH, texture and C concentration which influence the size of the plant-available Cd pool (Smolders and Mertens, 2013). For two of the three soils (Oen, Wied), Cd mass balances at the field scale were determined recently (Imseng et al., 2018). The third soil from LQ was chosen because of its comparatively high pH (7.1). In Oen, we measured stable isotopes in soils and wheat that had received unlabeled mineral P fertilizer. The wheat cultivar (*Triticum aestivum* L., cv. "Fiorina") has been commonly cultivated in the past years in Switzerland. A commercially available mineral P fertilizer (triple super phosphate, TSP) was chosen with a Cd concentration of 19 mg kg^{-1} Cd per kg of dry weight (equivalent to 92 mg kg^{-1} P) which is at the upper end of Cd concentrations for mineral P fertilizers applied to Swiss agricultural soils (FOAG, 2015). We applied 500 mg mineral P fertilizer with $9.5 \mu\text{g Cd}$ to 1 kg of soil (Table A2). This represents a P fertilization of $230 \text{ kg ha}^{-1} \text{ y}^{-1}$, an amount of P that is about 10 times larger than the current average P-fertilization of arable soils in Switzerland. However, P fertilization in Switzerland peaked in the 1970s and was then reduced by 80% until 2005 (Spiess, 2011). Thus, the chosen model soil-fertilizer-wheat system can be considered as a worst-case scenario for Swiss farming practice in terms of unintended Cd addition to soils.

2.2. Fertilizer labeling

We adapted the protocol of Jensen and Mosbæk (1990) to label mineral P fertilizer with a radioactive Cd isotope. The P fertilizer (6.5 g) was dissolved in deionized water (130 mL, $>18.2 \text{ M}\Omega$) and continuously stirred. Afterwards, carrier-free ^{109}Cd (CdCl_2 in 0.5 M HCl, 6 MBq, radio isotope center POLATOM) was added to the mineral P fertilizer suspension, stirred for 10 min and evaporated slowly to dryness on a hotplate. The labeled fertilizer was then carefully ground and split into 13 portions, each containing 0.5 g of labeled fertilizer. Twelve portions were used for the pot experiment and one portion was used to measure the specific activity of the mineral P fertilizer added to each pot. For characterization, we compared the mineralogy of the inorganic crystallized species in the original mineral P fertilizer with the evaporated fertilizer using Powder X-ray Diffraction (XRD, Bruker AXS D8 Advance). The comparison revealed

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