



Calcium is cycled tightly in *Cryptomeria japonica* stands on soils with low acid buffering capacity



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ABSTRACT

Calcium (Ca) is a key cation in the soil acidification process and in the fertility of forest soils. We investigated total concentrations of Ca, magnesium (Mg), and potassium (K) in both soil and fresh leaf litter in eight plantation stands of *Cryptomeria japonica* divided into two groups of highly contrasting soil acid buffering capacity (ABC). In addition to conventional methods, we used a double-crystal high resolution X-ray fluorescence (HRXRF) spectrometer to determine Ca fractions in soils with low and high ABC. Total Ca had accumulated in the 0–10 cm depth horizons of the high-ABC soils, but not in the low-ABC soils. The exchangeable Ca concentration was correlated with total Ca concentration in the 0–10 cm depth of the low-ABC soils. Calcium- $K\alpha_{1,2}$ emission spectra of the 0–10 cm soils obtained by HRXRF spectrometry separated the total Ca into two fractions, suggesting that the predominant Ca constituents had either passed through biological processes or were derived from weathering hydration in both the high- and low-ABC soils. Calcium concentration in the fresh leaf litter was significantly lower in the stands with low-ABC soil than in those with high-ABC soil and was positively correlated with soil exchangeable Ca concentration in the low-ABC stands; no such correlation was observed in the high-ABC stands. Exchangeable Ca pools in the 0–10 cm depth range were equal to or less than the annual input of Ca via leaf litterfall in the low-ABC stands, but were seven times the annual input in the high-ABC soils. These results suggested that the accumulation of Ca in the upper soil occurs via the process of Ca circulation within *C. japonica* ecosystems, and that sufficient supply of Ca for tree needs was accompanied by the large accumulation of available Ca by *C. japonica* in the high-ABC soils. Nevertheless, it is likely that only small amounts of Ca were circulated in the stands on low-ABC soil. The lack of Ca accumulation in the surface soil and the accompanying tight circulation of Ca in the plant–soil system may explain the observed positive feedback in which ABC is gradually reduced in low-ABC soils.

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Abbreviations: ABC, acid buffering capacity; CEC, cation exchange capacity; EC, electric conductivity; FWHM, the full width at the half of the maximum intensity of the peak; HRXRF, high-resolution X-ray fluorescence; XRF, X-ray fluorescence spectroscopy.

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1. Introduction

Soil acidification is a form of soil degradation that affects soil functions related to the provision of ecosystem services such as water, food, and energy security; biodiversity protection; and climate change abatement (McBratney et al., 2014). Soil acidity is potentially influenced by soil parent materials through their weatherable minerals (Van der Perk, 2013) and by biological processes of vegetation in forest ecosystems (De Schrijver et al., 2012). Most tree species enhance soil acidification through a variety of processes such as nutrient uptake, but some are known to

accumulate base cations such as calcium (Ca) in the topsoil, causing soil acidity to be reduced (Dijkstra and Smits, 2002; Finzi et al., 1998; Hamburg et al., 2003; Schaetzl and Anderson, 2005).

Many studies have indicated that trees act in only one direction, either soil acidification or base cation enrichment, as they grow (e.g., Hallbäck and Tamm, 1986; Jobbágy and Jackson, 2003; Sawata and Kato, 1991, 1993, 2007; De Schrijver et al., 2012; Reich et al., 2005). However, we previously reported on a tree species, *Cryptomeria japonica*, that can act in either direction, depending on soil conditions (Table 1; Tanikawa et al., 2014a). We found that *C. japonica*, a major tree plantation species in Japan, accumulates exchangeable Ca in surface horizons of acidic soils with high acid buffering capacity (ABC) but does not do this in low-ABC soils. That is, *C. japonica* plays a positive feedback role in an acidic environment such that ABC increases in soil with high ABC and decreases in soil with low ABC. In that study, ABC was defined by two criteria according to Takahashi et al. (2001): the concentration of exchangeable base cations and the concentration of exchangeable aluminum (Al). The definition was based on the fact that soil acidification leads to a decrease in base cations and to an increase in potentially phytotoxic ions such as Al (Ulrich, 1994). One possible reason that *C. japonica* lost its ability to accumulate exchangeable Ca in the soils may be the small amount of Ca circulation in the plant–soil system on low-ABC soils.

Calcium uptake by trees is governed by the amount of water transported by evapotranspiration (Clarholm and Skjyllberg, 2013). Japanese foresters have conventionally planted *C. japonica* on lower hillslope positions where soil is moderately moist, because the species grows slower on drier upper slopes. Furthermore, the Ca content of leaf litter is known to be high in *C. japonica* (Sawata and Kato, 1993; Ohta et al., 2014). Sawata and Kato (1993) reported that the Ca input into soil via litterfall was about 4–5 times larger for *C. japonica* than for the other common plantation tree species of Japan, *Chamaecyparis obtusa*. Ohta et al. (2014) also indicated that the concentrations of both total Ca and water-extractable Ca in soils were higher in *C. japonica* forest than in evergreen broad-leaved forest. Nagakura et al. (2004) showed that transpiration rates in *C. japonica* are approximately 1.2 times as high as those in *C. obtusa* in well-watered conditions. Thus, we speculate that *C. japonica* intrinsically uses large amounts of Ca because of its higher evapotranspiration. Nevertheless, this intrinsic property may not be exhibited in low-ABC soils where soil Ca concentration is inherently low. For a species' inherent trait to become manifest, suitable soil conditions for the species may be required, i.e., a base-cation cycling species that accumulates base cations in soil (Schaetzl and Anderson, 2005) may require a large base cation supply in the soil to develop a desirable plant–soil interaction. To test this idea, it is necessary to confirm whether a difference exists in the circulation of base cations (especially Ca) between *C. japonica* forests on soils with contrasting ABC. Further, the conditions and processes of soil fertility that govern Ca accumulation or depletion should be evaluated for sustainable management of *C. japonica* plantations.

Speciation of nutrient elements in soil helps to elucidate their sources, biogeochemical stability, and accumulation processes (e.g., Tanikawa et al., 2013, 2014b,c). However, published reports on Ca fractionation in species other than exchangeable Ca are scarce, and information about the ratio of exchangeable Ca to total Ca is also limited. X-ray fluorescence (XRF) spectroscopy with a single-crystal spectrometer, using the energy and intensity of fluorescence X-rays to specify and quantify chemical elements, has often been applied to soils and rocks (Hou et al., 2004). High resolution X-ray fluorescence (HRXRF) spectroscopy with a two-crystal spectrometer has an energy resolution two orders of magnitude greater than XRF and can determine energy position and shape of

different spectra of an element with greater precision (Tochio et al., 2002).

HRXRF spectroscopy has been used for analyzing industrial products (e.g., Okada et al., 2001) as well as biological sulfur and phosphorus in leaves, bones, and human hair (e.g., Qi et al., 1987a,b). Bai et al. (1985b) determined the shifts of the Ca- $K\alpha_{1,2}$ emission spectra (characteristic spectra of Ca) of pure Ca reagents by HRXRF spectroscopy, and used these spectra to differentiate between CaO and Ca(OH)₂. Maruyama et al. (1992) proposed a basic model that can be applied to explain the spectral difference between Ca-O and Ca-O-H. Therefore, here, we tried to use double-crystal HRXRF spectroscopy to determine the speciation of soil Ca through the energy changes, called “chemical shifts”, of the Ca spectra and to evaluate whether HRXRF spectroscopy could characterize the fractionation of the Ca components of low- and high-ABC soils. By enabling us to characterize Ca circulation in *C. japonica* forests with low- and high-ABC soils, this method can lead to new insights into the bioavailability and retention pathways of Ca in soil.

The aims of this study were to characterize Ca circulation in *C. japonica* forests with low- and high-ABC soils, and to attempt speciation of soil Ca based on chemical shifts in the Ca- $K\alpha_{1,2}$ emission spectra using double-crystal HRXRF spectroscopy. To achieve these objectives, we investigated whether total Ca concentrations in both soils and fresh leaf litter were related to soil exchangeable Ca across the ABC gradient reported in our previous study (Tanikawa et al., 2014a). We also determined the total contents of other base cations (Mg and K), the compositions of which are related to soil ABC.

2. Materials and methods

2.1. Study sites and soils

The study utilized four *C. japonica* plantation stands with low-ABC soil (Aibano, Anji, Kuta, Hyakusaiji) and four stands with high-ABC soil (Yamasaki, Kasumi, Wachi, Takarazuka) reported in our previous studies (Table 1; Hirano et al., 2017; Tanikawa et al., 2014a). All soils were classified as Brown Forest Soil (Forest Soil Division, 1976) and Cambisol (FAO-UNESCO, 1990). The mean annual wet deposition of Ca was calculated from the datasets of the Japan Acid Deposition Survey 20 (Japan Environmental Sanitation Center, Acid Deposition and Oxidant Research Center, 2004). The deposition rates in the study areas were 4.6–7.0, 4.7–15.1, and 5.1–17.2 meq m⁻² year⁻¹ during 1988–1992, 1993–1997, and 1998–2002, respectively. There was no difference in the Ca deposition rates between the two groups of stands. Wet deposition rates of magnesium (Mg) and potassium (K) were also similar for the two ABC groups (data not shown). Neither was any difference in acid deposition found between the two groups (Tanikawa et al., 2014a).

The forest study sites had been established in the 1990s as part of a national forest health monitoring network, and we set up a 10 × 10 m plot within the boundaries of each study site for intensive soil and root studies (Hirano et al., 2017; Tanikawa et al., 2014a). Soil samples were collected from the depth ranges of 0–10, 10–20, and 20–40 cm in the four soil profiles during the previous study in 2009 (Tanikawa et al., 2014a), comprising 96 samples derived from 8 stands × 4 replicates × 3 depths. Thus, all soil chemical analyses were performed in four replications for each depth in each stand. The data are shown in Table 1. We also measured the thickness of the F-H layer in the four soil profiles. Because *C. japonica* leaf litter of the L layer is firm, keeps a three-dimensional shape, and includes a large amount of air space, it was difficult to accurately evaluate the L layer thickness in the C.

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