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Dynamique comparée des éléments majeurs dans les humus de forme mull dans une hêtraie du Nord-Est de la France

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Résumé de la thèse

L'humus est le siège de processus clés pour la nutrition des arbres. Cependant, il reste un compartiment de l'écosystème encore peu étudié pour l'ensemble des nutriments et éléments bénéfiques autre que C et N. Ainsi, dans un contexte où les pressions exercées sur les forêts augmentent depuis quelques années, il est important de comprendre la dynamique comparée des éléments de leur arrivée dans l'humus à leur sortie dans le sol. Les objectifs de cette thèse sont (1) d'identifier les mécanismes et les facteurs responsables de la dynamique des éléments majeurs (C, N, P, K, Ca, Mg, Mn, S, Si, Al et Fe), (2) de déterminer les relations entre ces éléments et (3) de préciser la place de l'humus dans la nutrition de l'arbre. Pour répondre à ces objectifs, un modèle conceptuel basé sur l'identification des flux entrant et sortant de l'humus et des possibles mécanismes présents dans le niveau de l'humus a été élaboré.

Les résultats obtenus dans cette thèse suggèrent que la dynamique des éléments et leur disponibilité pour l'arbre à la sortie de l'humus dépendent d'un ensemble de processus et réactions accélérant ou retardant leur départ de l'humus. Les vitesses de libération des éléments dans le sol dépendent de leurs formes dans les entrées de l'humus (soluble, molécules organiques, biominéraux), de leur localisation dans les feuilles (limbe vs. nervure) et des tissus des végétaux plus ou moins facilement dégradables alimentant l'humus. La dynamique des éléments est également influencée par des réactions de recyclage à l'intérieur de l'humus, comme des précipitations biotiques ou abiotiques. Les éléments être utilisés par les amibes à thèques (Si, P, Ca et Mn), précipités autour des hyphes (Ca, Mn, P, et K) ou encore être précipité sous forme de plage amorphes (Si). Ces réactions contrôlent les temps moyens de résidence des éléments : les éléments les plus solubles, localisés dans des tissus plus dégradables et les moins recyclés seront lessivés plus rapidement et auront des temps de résidence plus bas.

La période libération des éléments sous l'humus intervient sur leur disponibilité pour l'arbre de ces éléments. En période de végétation où les éléments sont directement disponibles pour les arbres, moins de 25% de K et P sont libérés. La capacité du sol à retenir les éléments produits en période de sénescence et de dormance sera fondamentale pour assurer la pérennité de l'écosystème.

Lors de cette thèse, les particules en sortie de l'humus ont été quantifiées pour la première fois. Outre le rôle sur la pédogenèse en général et la dynamique du C, ce flux particulaire sous l'humus pourrait augmenter la capacité d'échanges cationique des sols et sa capacité à retenir ces éléments lessivés en période de dormance.

Mots clés : Humus; dynamique; bilan entrées/sorties; particules; formes des éléments; recyclage ; hêtraie

Summary

Humus is the place of key tree nutrition processes. However, it remains a compartment of the ecosystem still under studied for all major nutrients and beneficial elements other than C and N. Thus, in a context of increasing pressure on forests, it matters to understand the dynamics of the elements from their input into the humus to their output towards the soil.

The aims of this thesis are: (1) to identify the mechanisms and factors responsible of the major element dynamics (C, N, P, K, Ca, Mg, Mn, S, Si, Al and Fe), (2) to determine the relationships between these elements and (3) to specify the place of the humus in tree nutrition. To answer these objectives, a conceptual model based on the identification of humus inputs and outputs and the possible mechanisms present at the humus level was elaborated.

The results of this thesis suggest that the element output fluxes depends on processes and reactions accelerating or delaying their release from the humus. The element release rates in the soil depend on their forms in the humus inputs (soluble, organic molecules, biominerals) and their location in the plant tissues (leaf blade vs. vein) supplying the humus. The element dynamics is also influenced by recycling reactions within the humus, such as biotic or abiotic precipitation through testate amoebae (Si, P, Ca and Mn), precipitates around hyphae (Ca, Mn, P, and K) or amorphous Si precipitates. These reactions control the mean residence times of the elements and their leaching gradient below the humus: the most soluble and least immobilized elements will be leached more rapidly and will have lower residence time.

The release of these elements below the humus is another important factor that affects the availability of these elements to trees. During the growing season, when the trees upatke soil solution, less than 25% of K and P are released from humus. The ability of the soil to hold the elements produced during periods of senescence and dormancy is fundamental to ensure the sustainability of the ecosystem.

In this thesis, the particles leaving the humus were quantified for the first time. In addition to their role on pedogenesis and the dynamics of C, this particle flux below the humus could increase the cation-exchange capacity of the soil and its ability to hold these leached elements during the dormant period.

Key words: Humus; dynamic; input/output budget; particles; element forms; recycling; beech forest

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Chapitre I -

Introduction Générale

Avant-propos

L'humus, bien qu'étant le siège de processus clés pour la nutrition des arbres, reste un compartiment de l'écosystème encore peu étudié pour l'ensemble des principaux nutriments et éléments bénéfiques (P, K, Ca, Mg, Mn, S, Si), en dehors de C et N. Ainsi, dans un contexte où les pressions exercées sur les écosystèmes forestiers augmentent depuis quelques années, le recyclage des parties aériennes de l'arbre n'en est que plus important.

À travers un suivi des flux au sein de l'humus, d'une étude expérimentale sur la dégradation des litières et d'une étude de la co-évolution des éléments dans les flux entrants/sortants de l'humus, ce travail de thèse propose d'identifier et de quantifier les flux entrants et sortants de l'humus ainsi que de déterminer l'ensemble des mécanismes impliqués dans les interactions entre les éléments afin de comprendre l'impact de l'humus sur la dynamique des éléments et la nutrition des arbres. Ce manuscrit est composé de sept chapitres :

- Le chapitre 1 est une introduction générale du travail de thèse replaçant l'étude au regard des connaissances actuelles, expliquant les différentes notions et termes utilisés dans ce manuscrit et présentant les objectifs, les questions et les hypothèses de cette thèse.
- Le chapitre 2 traite du matériel et des méthodes employés durant cette thèse.
- Les chapitres 3, 4, 5 et 6 présentent et discutent les résultats obtenus sous forme d'articles. Le 3ème chapitre concerne le temps moyen de résidence des éléments majeurs dans l'humus et du rôle de la forme des éléments et du recyclage présent dans l'humus. Cet article a été publié en 2020 dans la revue *Soil, Biology and Biochemistry*. Le 4ème chapitre présente le flux annuel de particules à la sortie de l'humus et son importance dans les bilans entrées/sorties des éléments de celui-ci. Cet article a été accepté en juillet 2020 dans la revue *Biogeochemistry*. Le chapitre 5 traite de la dynamique saisonnière de la libération des éléments dans la solution de l'humus et des conséquences de cette dynamique pour la nutrition des arbres (article soumis à *Biogeochemistry*). Enfin le chapitre 6 est une comparaison entre une étude expérimentale de la dégradation des litières par la méthode des sachets de litières et les données de dégradation des humus obtenus dans les chapitres 3 à 5.
- Le chapitre 7 correspond à une synthèse générale de la thèse mettant en avant les relations entre les différents éléments à l'entrée, dans et en sortie de l'humus. Ce chapitre présente également les conclusions et les perspectives de ce travail.

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1 Contexte et enjeux

En France métropolitaine, les forêts couvrent 16.9 millions d'hectares, ce qui correspond à environ 31 % du territoire (Inventaire Forestier National, 2018). Parmi ces forêts, les peuplements de feuillus purs représentent 67% (9.8 millions d'hectares) (Figure 1). Le hêtre est la troisième essence feuillue de la forêt française, couvrant 1.4 millions d'hectares soit 10 % des surfaces françaises boisées. Cette essence est une espèce d'intérêt car elle joue un rôle socio-économique important de par son poids dans la filière bois. Le hêtre représente 23% du bois d'œuvre de feuillus et 10% du bois d'œuvre total. Dans le Grand Est, la forêt couvre 1.9 millions d'hectares dont 70% du volume sur pied sont des feuillus et 30% des résineux (Inventaire Forestier National, 2018).

Les écosystèmes forestiers fournissent des biens et des services économiques, sociaux et environnementaux (comme la production de biomasse, la filtration de l'eau, la régulation du climat par le stockage du carbone ou encore comme source de biodiversité). Cependant, depuis plusieurs années, les pressions exercées sur les écosystèmes forestiers augmentent, notamment par l'intensification de l'exportation des rémanents (petits bois résiduels suite à une éclaircie ou une exploitation totale) (Hartmann et al., 2012; Mendham et al., 2002) ou les pratiques de sylviculture à rotation plus courte (Heilman and Norby, 1998). De plus, les forêts ne sont généralement pas amendées et sont, soit présentes sur des sols acides avec une faible capacité de fourniture des nutriments par altération des minéraux (Badeau and Dambrine, 1999), soit sur des sols dits « riches » comme les sols calcaires qui peuvent présenter des carences en P et K (Sardans and Peñuelas, 2015; Zederer and Talkner, 2018). L'intensification des pratiques sylvicoles risque alors d'impacter la fertilité minérale de ces écosystèmes déjà fragiles. En plus de ces contraintes nutritionnelles, s'ajoute des contraintes climatiques. Les écosystèmes forestiers européens sont directement affectés par l'élévation de la température moyenne et les modifications des régimes de précipitations (Intergovernmental Panel on Climate Change, 2018), notamment par une augmentation de la saison de végétation et une accentuation des déficits hydriques en été.

Pour pallier aux différentes pressions exercées sur ces écosystèmes, des pratiques de sylviculture durables sont mises en place. Elles ont pour but de répondre à l'augmentation de la demande en biomasse tout en assurant la pérennité de l'écosystème forestier et de son bon fonctionnement. En effet, les écosystèmes forestiers dépendent en grande partie du recyclage des parties aériennes (feuilles, bois, branches, organes de reproductions) et des parties souterraines (racines fines) de l'arbre. Ainsi, l'humus, composé majoritairement des chutes annuelles des feuilles de l'arbre, est un compartiment clé dans ce recyclage. Il tient donc un rôle de protection du sol mais

également d'apport de matières organiques aériennes et d'éléments chimiques au sol. Plusieurs études ont également mis en lumière la nécessité de comprendre les interactions entre les peuplements, l'humus et les sols forestiers comme des prérequis pour la définition d'une sylviculture durable (Mahendrappa et al., 1986; Nambiar, 1996).

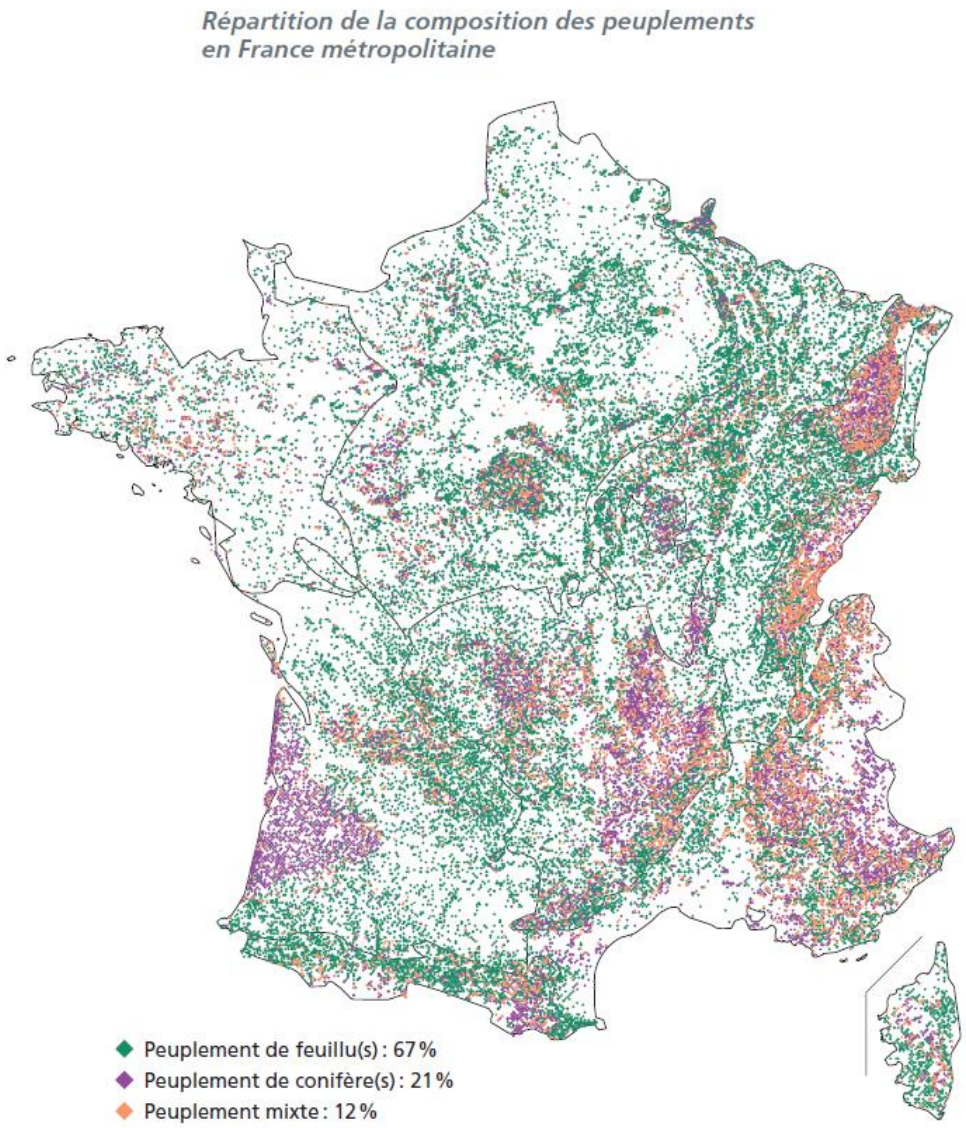

Figure 1 : Carte des répartitions des formations boisées en France par type d'essence (IGN, 2017)

2 L'humus

2.1 Définition de l'humus

En forêt, les débris végétaux (feuilles, branches, organes reproducteurs…) tombant sur le sol sont plus ou moins rapidement décomposés par l'activité biologique et l'action physique de l'eau. Cette dégradation progressive forme des horizons organiques successifs suivant la vitesse de décomposition des éléments présents. Ces horizons holorganiques (c'est-à-dire contenant essentiellement de la matière organique) sont divisés en trois horizons notés O_x :

- **OL (L = « Litière »)** : Il s'agit d'horizons constitués de débris foliaires, non ou peu évolués, et de débris ligneux (bois). La forme des débris est identifiable à l'œil nu. On distingue OLn (litière « nouvelle ») constituée des débris de l'année et OLv (« litière vieillie ») constituée de feuilles plus transformées mais toujours reconnaissables (Jabiol et al., 2007). L'épaisseur des horizons OL est maximale lors de la chute des feuilles et diminue plus ou moins rapidement au cours de l'année selon l'activité biologique et le climat.
- **OF (F=Fragmentation)** : Cet horizon est formé de résidus d'origine foliaire, plus ou moins fragmentés mais reconnaissables à l'œil nu, et de matière organique fine sous forme d'amas millimétriques résultant de l'accumulation de boulettes fécales plus ou moins remaniées par la mésofaune (Jabiol et al., 2007). La proportion de chaque partie peut varier et cet horizon peut être présent de manière discontinue.
- **OH (H= « Humification »)** : Cet horizon est composé à plus de 70% de matière organique fine composée d'amas de boulettes fécales et de micro-débris végétaux et mycéliens non reconnaissables à l'œil nu (Jabiol et al., 2007). Il s'agit d'un horizon sombre assez homogène qui peut parfois présenter des grains minéraux visibles à l'œil nu. Des racines mortes et vivantes peuvent être présentes en plus ou moins grande quantité.

L'ensemble de ces horizons et la description de l'horizon A (horizon organo-minéral) permettent de donner la forme de l'humus. Selon la présence ou non et l'épaisseur de chaque horizon, il est possible de définir trois formes principales de l'humus : MULL, MODER et MOR (Figure 2), qui peuvent être déclinées en formes intermédiaires (par exemple OLIGOMULL, EUMODER ou DYSMODER) (Jabiol et al., 2007).

La forme d'humus mull (OL ; A grumeleux) montre une utilisation rapide des éléments et un turn-over rapide. Le moder (OL ; OF ; OH ; A non grumeleux) présente une transition progressive entre OH et A. Il est le témoin d'une utilisation ralentie des

éléments. Enfin, la forme d'humus mor (OL ; OF ; OH ; horizon A voir horizon minéral) a une limite inférieure très nette. Le turn-over de la matière organique est très lent, la litière est très peu décomposée et les éléments sont immobilisés.

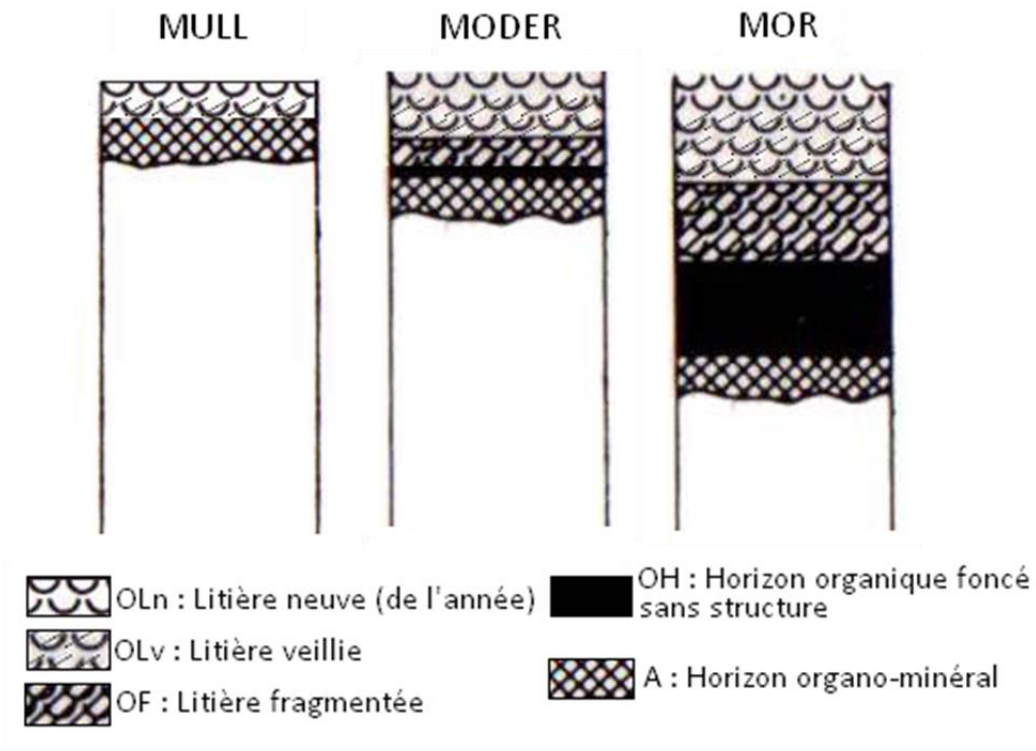

Figure 2 : Schématisation des principales formes de l'humus et représentation des différents horizons holorganiques d'après Jabiol et al., 2007.

Même si d'autres définitions existent, dans la suite de ce texte, nous utiliserons le terme « humus » comme l'ensemble des couches holorganiques. Nous utiliserons le terme de « litière » pour décrire les entrées issues de la chute des parties annuelles de l'arbre (feuilles, petites branches, fruits). Le terme de « rémanents » correspond ici aux résidus de petit bois laissés au sol après chaque éclaircie.

2.2 Place de l'humus dans les cycles biogéochimiques

Comprendre le fonctionnement des écosystèmes forestiers est une clé pour définir une sylviculture durable. Une des méthodes pour comprendre ce fonctionnement est l'étude des cycles biogéochimiques dans les écosystèmes forestiers. Ces cycles décrivent la circulation permanente des éléments dans l'écosystème par différents flux et un recyclage interne par la végétation (Ranger and Turpault, 1999).

Les cycles biogéochimiques sont divisés en trois sous-cycles reprenant les flux et les transferts d'éléments dans l'écosystème (Figure 3):

- **Le cycle géochimique** : ce cycle correspond aux échanges aux limites de l'écosystème c'est-à-dire aux entrées et aux sorties de l'écosystème, qu'elles soient naturelles ou anthropiques. Les entrées correspondent aux dépôts atmosphériques (particules <0.45 µm arrivants par temps humide ou sec et poussières >0.45µM) et à l'altération des minéraux du sol. Ce cycle intègre aussi les pertes d'éléments par drainage et l'exportation de la biomasse.

- **Le cycle biologique** : c'est le cycle interne à l'écosystème qui comprend le recyclage des parties annuelles aériennes (litière et petites branches) et souterraines (racines) ainsi que des parties pérennes non exploitées de l'arbre (rémanents). Ce cycle correspond à la remise en circulation (ou recyclage) des éléments entre l'humus, le sol et l'arbre. Les travaux de cette thèse s'inscrivent dans ce cycle.

- **Le cycle biochimique :** il correspond au cycle interne à l'arbre, c'est-à-dire à la circulation des éléments dans la plante elle-même. Il s'agit par exemple du phénomène de translocation des éléments des organes les plus vieux vers les plus jeunes.

Dans cette thèse nous avons utilisé le découpage de l'écosystème proposé par (Kirchen, 2017) qui divise les différents compartiments de l'écosystème en 4 niveaux distincts: l'atmosphère, la canopée, le sol et l'humus.

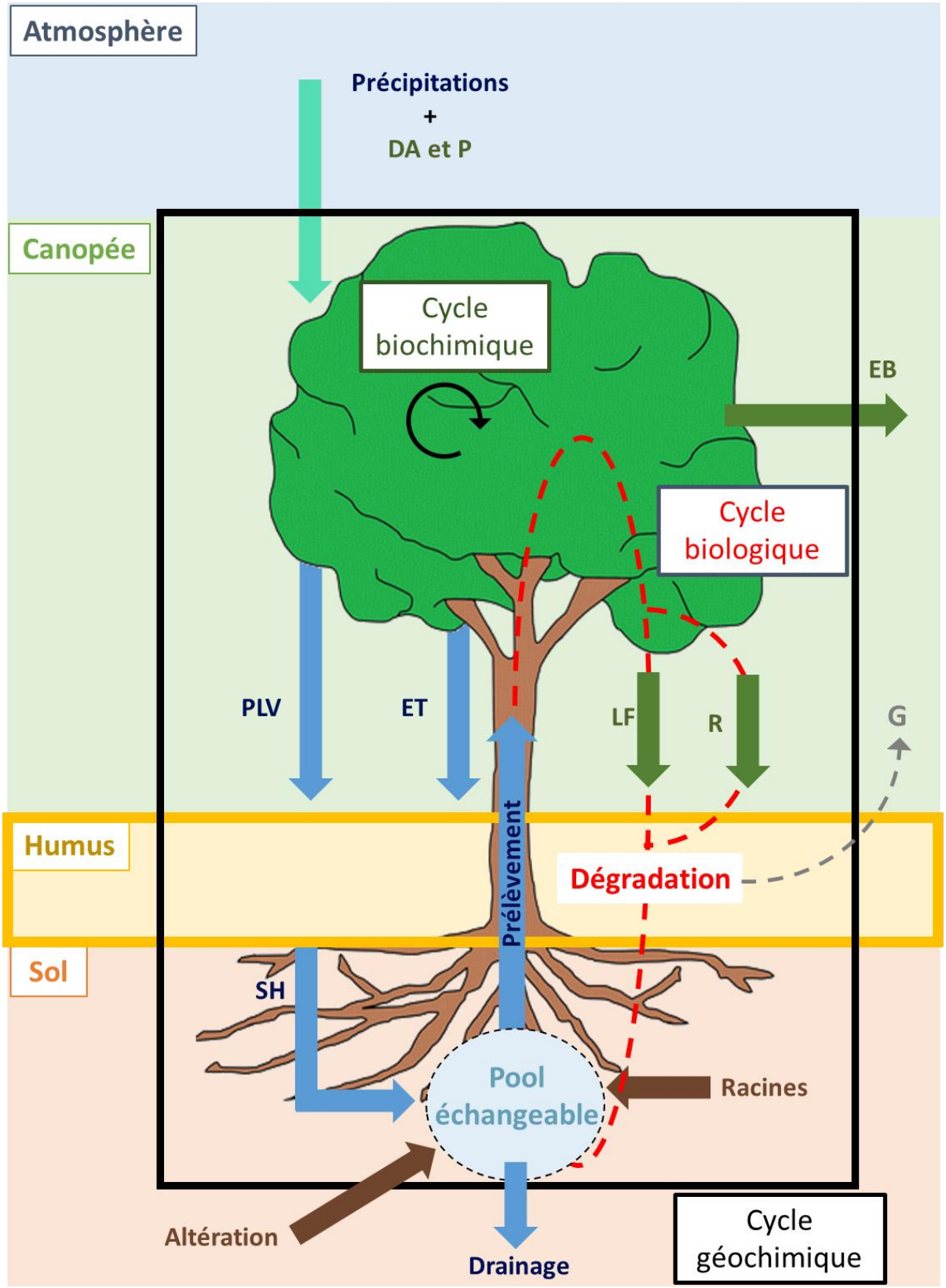

Figure 3 : Représentation schématique des cycles biogéochimiques et des flux dans un écosystème forestier. Les flux en solution sont représentés par des flèches bleues et les flux solides par des flèches vertes. DA= Dépôts Atmosphériques humides et secs <0.45µm ; P = Poussières (>0.45µm) ; EB = Exportation Biomasse ; PLV = Pluviolessivat ; ET = Écoulement de Tronc ; SH = solution humus ; LF = Litière ; R = rémanents ; G = Gaz

Dans ces cycles, l'humus occupe une place centrale. En plus d'avoir un rôle dans la protection du sol, il est une partie inhérente du cycle des nutriments et du carbone (Sayer, 2006). En effet, dans un contexte où les pressions augmentent sur les écosystèmes forestiers, l'humus à travers la dégradation et le recyclage des parties annuelles de l'arbre, est l'une des principales sources d'éléments nutritifs et bénéfiques pour la végétation (Aber and Melillo, 1991; Attiwill and Adams, 1993; Bartoli, 1983; Jacob et al., 2009; Sayer, 2006; Schlesinger, 1997). Ce processus est d'autant plus important que pour la plupart des forêts tempérées, il a été montré une prédominance du cycle biologique sur le cycle géochimique (Ponette et al., 2001; Ranger et al., 2002; van der Heijden et al., 2017). Cependant, pour comprendre la place de cet humus dans l'écosystème et son rôle dans la nutrition de l'arbre, il est nécessaire de connaître les stocks, les entrées et les sorties des éléments de ce compartiment.

3 Les entrées d'éléments dans l'humus

3.1 Les entrées en solution

L'eau de pluie entre dans l'écosystème forestier au niveau de la canopée. Dans la canopée, les éléments contenus dans les feuilles peuvent être échangés avec l'arbre (par résorption et accrétion) et avec la solution traversant la canopée (absorption foliaire et lessivage des éléments labiles) (Figure 4)(Kirchen, 2017). Ces échanges entre la feuille, l'arbre et la solution traversant la canopée peuvent également modifier les concentrations en éléments, notamment pour K, dans les solutions entrants dans l'humus (Pelster et al., 2009; Talkner et al., 2010).

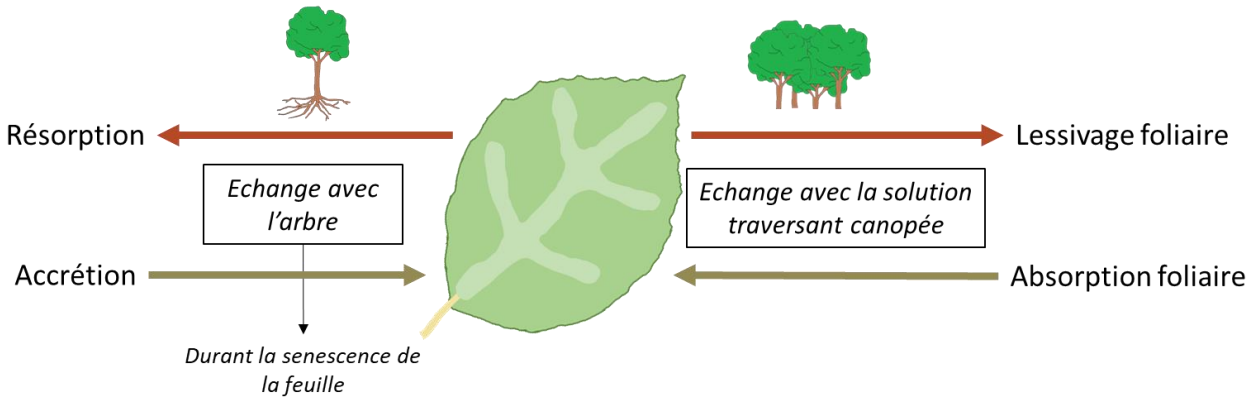

Figure 4 : Échanges d'éléments possibles entre les feuilles de l'arbre, l'arbre et la solution traversant la canopée.

Après le passage dans la canopée, les solutions arrivent au sol selon deux voies de transfert d'eau : le pluviolessivat (eau traversant la canopée) et l'écoulement de tronc (ruissellement de l'eau le long des troncs d'arbres) (Figure 3). En général, la majorité des études utilisent uniquement les solutions de pluviolessivats, les écoulements de tronc étant considérés comme un flux négligeable d'entrée d'éléments dans le sol forestier (Draaijers et al., 1996). Pour le hêtre, les écoulements de troncs peuvent représenter jusqu'à 14-20% des précipitations totales (Baba and Okazaki, 1999). Ce flux est d'autant plus important dans la nutrition des arbres qu'il s'écoule directement du tronc à la sphère radiculaire (André et al., 2008; Forgeard et al., 1980).

L'étude des solutions de pluviolessivats et de d'écoulements de troncs permet de déterminer la quantité d'entrée des éléments en solution dans l'humus (Draaijers et al., 1996). Cependant, la période d'arrivée et de sortie de ces solutions est très importante pour la nutrition de l'arbre. En effet, les besoins en nutriments des arbres dans les milieux

tempérés ont lieu essentiellement durant la période de végétation (printemps/été) (Helcoski et al., 2019; Li et al., 2019; Lovelock et al., 2007; Soper et al., 2018). En automne et en hiver, le prélèvement des arbres étant limité, si le sol n'a pas la capacité de retenir les éléments, ceux-ci sont lessivés et perdus pour l'écosystème et la nutrition de l'arbre.

3.2 Les entrées solides

Les entrées sous formes solides dans l'humus peuvent provenir de trois sources : les entrées par poussières atmosphériques, la chute de litière et les rémanents.

Les poussières sont actuellement peu documentées. Il s'agit de dépôts atmosphériques supérieurs à 0.45 µm. De récentes études menées par (Lequy et al., 2014, 2013, 2012) ont mis en évidence l'importance de ce flux d'entrée dans l'écosystème forestier. Il pourrait contribuer jusqu'à 30% des dépôts atmosphériques des écosystèmes forestiers et être l'entrée principale de P dans certains écosystèmes (Lequy et al., 2014).

Les chutes de litières ont longtemps été considérées comme les seules entrées solides de l'humus, du fait de la quantité importante d'éléments apportés à ce compartiment (Kavvadias et al., 2001; Ukonmaanaho et al., 2008; Whittaker et al., 1979). Les litières sont composées majoritairement de feuilles, de petites branches et d'autres débris végétaux comme les organes reproducteurs ou des bourgeons (Tardif, 2013).

Les rémanents sont des résidus d'exploitations (petits bois) laissés sur le sol après la récolte du bois (éclaircie ou coupe rase) et correspondent à des apports des parties pérennes de l'arbre au sol (Jensen, 1984). Le plus souvent, ce flux d'entrée n'est pas pris en compte bien que sa participation soit aujourd'hui reconnue comme non négligeable (de Dieu Nzila et al., 2002; Laclau et al., 2010).

4 Décomposition des litières et éléments dans l'humus

4.1 La décomposition des litières

La **décomposition ou dégradation** de la matière végétale (la litière) est le processus de dégradation physique et chimique des tissus de plantes (Tardif, 2013). La transformation de cette matière organique morte aboutit à une libération d'eau, de dioxyde de carbone et d'éléments minéraux dans le sol, sous une forme disponible (c'est-à-dire assimilable tout de suite) pour les végétaux (Begon et al., 2005; Chapin et al., 2002). Cette décomposition est un processus clé pour les écosystèmes (Cadisch and Giller, 1997; Swift et al., 1979) car c'est un facteur majeur influençant le cycle du carbone (Canadell et al., 2007; Prentice et al., 2001) ainsi que la disponibilité en nutriments et éléments bénéfiques pour la végétation. Elle influence donc directement la nutrition des plantes (Bardgett, 2005; Wardle, 2013). Les arbres et végétaux du sous-bois participant à la litière affectent également le cycle des nutriments dans l'écosystème à travers le prélèvement et l'utilisation des nutriments par les végétaux, les interactions avec la rhizosphère et la qualité de la litière (Berg and McClaugherty, 2014; Eviner and Chapin III, 2003; Hobbie, 1992).

Ce processus de décomposition, en plus de constituer une source importante de nutriment pour l'arbre, contribue à l'émission de gaz à effet de serre et en particulier de CO_2. La décomposition de la matière organique est donc un processus au cœur des problématiques actuelles (IPCC et al., 2005).

4.2 Vitesse de décomposition des litières

Les débris végétaux de la litière sont composés de **constituants organiques** mais également de **biominéraux** (Krieger et al., 2017). Les biominéraux les plus communs retrouvés dans les plantes sont les oxalates de calcium, la silice amorphe et les carbonates de calcium (Bauer et al., 2011).

La plupart des constituants chimiques organiques des litières correspondent à des composés hydrosolubles. Parmi ces composants, trois sont particulièrement présents : la cellulose et l'hémicellulose qui sont des carbohydrates labiles en polymères, et les lignines composées de cycles aromatiques, plus difficiles à décomposer (Neher et al., 2003; Tardif, 2013; Williams and Gray, 1974). La cellulose représente 20-30 % de la masse totale des litières, l'hémicellulose 30-40% et les lignines 15-40% (Killham, 1994). Cependant, ces valeurs peuvent varier selon le type de plante, l'âge et l'environnement (Berg and McClaugherty, 2014). Généralement, la décomposition des litières comprend deux étapes (Aerts, 2006) :

- **La fragmentation** de la litière par les détritivores (macro et microfaune) qui peut durer de quelques mois à quelques années.
- **La minéralisation** de la litière, c'est-à-dire la transformation chimique des fragments en molécules inorganiques de base (ammonium, phosphate, dioxyde de carbone, eau, etc…) (Cadisch and Giller, 1997; Swift et al., 1979). En général, l'hémicellulose est minéralisée avant la cellulose et les lignines.

Les temps de décomposition moyens des composants principaux de la litière varient de 14 jours pour l'hémicellulose à 500 jours pour les lignines (Killham, 1994). Ainsi, avec l'avancement de la décomposition, la concentration en lignines va augmenter et va ralentir la vitesse de décomposition de la litière.

La vitesse de décomposition de la litière est également influencée par des facteurs abiotiques et biotiques dont les trois principaux sont : l'environnement physique (le climat, la géographie, le sol…), la qualité de la litière (caractérisée par des rapports comme C/N, N/lignine…) et les organismes présents (la micro, méso et macrofaune) (Figure 5). Ces différents facteurs influencent différemment la décomposition en fonction de son stade d'avancement (Berg and McClaugherty, 2014) mais également en fonction de l'échelle spatiale : le climat aura un effet prédominant à l'échelle régionale alors que la qualité des litières sera plus importante à une échelle locale (Aerts, 1997; Meentemeyer, 1984). Cependant, certains auteurs ont hiérarchisé ces facteurs par ordre décroissant d'importance : climat > qualité des litières > organismes décomposeurs (Aerts, 2006; Lavelle et al., 1993). Nous pouvons supposer que la présence plus ou moins importante de biominéraux dans les tissus végétaux pourrait également jouer sur la vitesse de décomposition, ces formes étant potentiellement plus difficiles à dégrader.

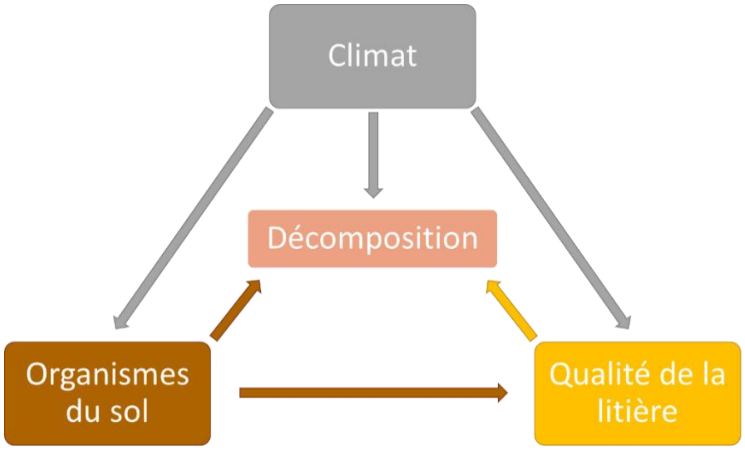

Figure 5 : Principaux facteurs contrôlant la décomposition de la litière (adaptée de Aerts (2006))

4.3 Perte de masse des litières

Le processus de décomposition se traduit par une perte de masse des litières liée à l'action associée de la fragmentation, de la minéralisation et du lessivage de la litière. Cependant, comme décrit précédemment, les principaux composants des litières ne se dégradent pas à la même vitesse et la perte de masse n'est pas constante dans le temps (Figure 6) (Tardif, 2013).

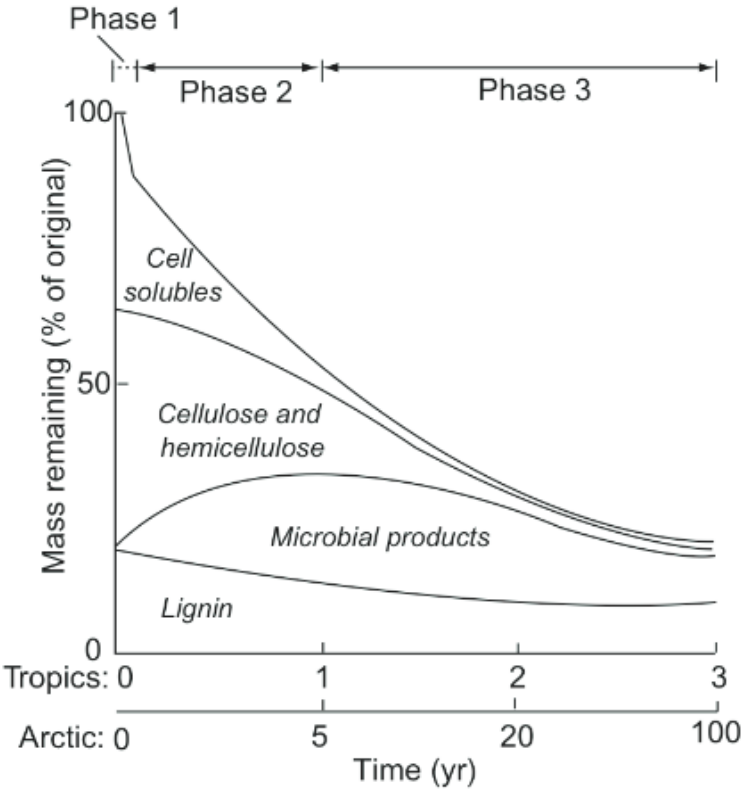

Figure 6 : Représentation schématique de la masse restante (% de la masse initiale) de litière foliaire en fonction du temps de décomposition (années) en climats tropical et arctique. Les principaux types de constituants chimiques sont présentés (composés cellulaires solubles, cellulose, hémicellulose, produits microbiens, lignines), ainsi que la dynamique des 3 phases majeures (tiré de Chapin et al., 2002)

Cette perte de masse pourrait être divisée en trois phases (Figure 6) (Chapin et al., 2002) :

1) **Le lessivage des composés cellulaires solubles**. En effet, ces composés peuvent représenter 7% à 30% de la masse foliaire pour des aiguilles de Pin sylvestre et d'Aulne blanc respectivement (Berg and McClaugherty, 2014). Le lessivage de ces composés solubles a lieu dans les premières 24h et peut représenter jusqu'à 5% de la perte de masse initiale de la feuille sénescente (Chapin et al., 2002).

2) **La fragmentation et la minéralisation de la litière**. Cette seconde phase, moins rapide est dépendante de l'action des organismes (macro-, méso- et micro-organismes). Cette phase peut durer quelques mois à quelques années.

3) **La très lente réduction de la masse des litières**. Cette phase, la plus lente, correspond à des litières qui évoluent peu chimiquement tout en s'incorporant au sol (Aber et al., 1990; Melillo et al., 1989). Les litières sont à ce moment constituées essentiellement de composés difficiles à dégrader comme des lignines, des parois cellulaires microbiennes ou encore des biominéraux.

De nombreux modèles mathématiques ont été élaborés pour décrire le processus de perte de masse des litières (Berg and McClaugherty, 2014; Moorhead et al., 1996). Le modèle le plus utilisé est celui d'Olson (1963) décrivant la décomposition de la litière selon une loi exponentielle négative :

$$M_t = M_0.e^{-kt}$$

avec M_t la masse de litière restante au temps t, M_0 la masse de litière initiale et k le taux de décomposition.

4.4 Temps de résidence dans l'humus

Bien qu'essentielles dans le cycle biologique de l'écosystème forestier, les études sur les éléments autres que C et N dans l'humus sont plutôt limités. Le plus souvent, les éléments sont étudiés dans les litières à travers la perte de masse par des études par sachets de litières (Albers et al., 2004; Berg and McClaugherty, 2014; Berger et al., 2009). Les études centrées sur l'humus auront tendance à se concentrer sur les vitesses de minéralisation de la matière organique et les temps de résidence des éléments notamment de C, N et parfois P (Campo et al., 2014; Gosz et al., 1976; Kavvadias et al., 2001; Prescott et al., 1989; Vogt et al., 1983). Dans ces différentes études, le temps de résidence des éléments peut être calculé pour des humus stables, c'est-à-dire que le stock d'éléments présents dans l'humus n'évolue pas. Ce temps est calculé à partir de la formule (Gosz et al., 1976) :

$$T = H / L$$

avec T= temps de résidence, H = stock d'un élément dans l'humus et L= élément dans la litière entrante.

Le calcul de ce temps de résidence présente cependant des limites car il s'agit d'un temps moyen de résidence pouvant intégrer des processus très contrastés. En effet, il ne tient généralement compte que d'un seul type d'entrée solide dans l'humus, négligeant des entrées comme les rémanents qui pourraient influencer le temps de résidence de certains éléments. De plus, la présence de particules de sol dans l'humus (liée notamment à de la bioturbation) pourrait augmenter la quantité de certains éléments dans le stock d'éléments présents.

Dans l'humus, certains éléments peuvent également être immobilisés par des processus biotiques et abiotiques. Ces processus ont été plus majoritairement étudiés pour Si et Ca. Plusieurs auteurs ont montré que Si pouvait être immobilisé par certains types d'amibes qui utilisent le Si présent dans l'humus pour construire leurs thèques (Sommer et al., 2013; Wilkinson and Mitchell, 2010). Le Ca, parfois combiné avec P, K ou Mn a été observé sous forme d'oxalate de calcium précipitant autour d'hyphes de champignons quand il était présent en grande quantité dans l'humus (Burford et al., 2003). Cependant, peu d'études présentent l'immobilisation d'autres éléments, processus qui pourrait influencer leurs différents temps de résidence.

5 Les sorties d'éléments dans l'humus

Dans la littérature, les solutions à la sortie immédiate de l'humus sont très rarement étudiées et concernent seulement C, N et P (Spohn and Sierra, 2018). La plupart du temps, seules les sorties de solutions drainées hors du sol (en sortie de l'écosystème) sont considérées (Likens et al., 2002; Liu et al., 2003; Manderscheid et al., 1995). Ce manque de données sur les solutions issues de l'humus pourrait résulter de la difficulté matérielle à capter, prélever et échantillonner ce type de solution (Chapitre II - 2.1).

D'autres sorties de l'humus ont été identifiées comme les sorties sous forme de gaz, notamment pour C et N lors de la minéralisation de la matière organique (Batjes, 1996; Berg and McClaugherty, 2014; Marklein et al., 2016). Lorsque les racines sont présentes dans l'humus, les éléments peuvent être directement prélevés par l'arbre. Les éléments peuvent également être prélevés par des hyphes mycéliens, prolongement des racines de l'arbre dans ce niveau (Mikola, 1990; Mousain et al., 1997).

Cependant, même en quantifiant ces prélèvements, les bilans entrées/sorties de l'humus calculés pour un humus stable révèlent un déséquilibre avec des sorties inférieures aux entrées (Spohn and Sierra, 2018), indiquant qu'il existe une ou plusieurs autres sorties encore non identifiées.

L'identification et la quantification de ces différents flux de sortie permettraient de caractériser les relations existants entre les éléments dans les entrées et dans les sorties et ainsi de mieux comprendre la dynamique des éléments dans l'humus et leur évolution au cours du temps.

6 Objectifs, questions et hypothèses

À ce jour, il n'existe que peu d'études recensant l'ensemble des mécanismes liés à la dynamique des éléments (autres que C et N) et leurs relations dans l'humus ainsi que l'impact de l'humus dans la nutrition des arbres. **Les objectifs de cette thèse sont donc d'identifier les mécanismes et facteurs influençants la dynamique de ces éléments dans l'humus et de déterminer les relations entre les éléments ainsi que la place de l'humus dans la nutrition de l'arbre.**

Pour réussir à répondre à ces objectifs, nous avons élaboré un modèle conceptuel basé sur l'identification des flux entrants et sortants de l'humus et des possibles mécanismes présents au sein de l'humus (Figure 7). En effet, les études centrées sur l'humus prennent rarement en compte tous les types d'entrées et de sorties d'éléments et ne tiennent pas compte des mécanismes et processus présents à l'intérieur de celui-ci. Ce modèle conceptuel entrées/sorties des éléments, basé sur l'état stationnaire de l'humus, pourrait permettre d'avoir une vision plus globale de la dynamique des éléments et des conséquences associées à ces mécanismes (comme la vitesse de dégradation) pour la nutrition de l'arbre.

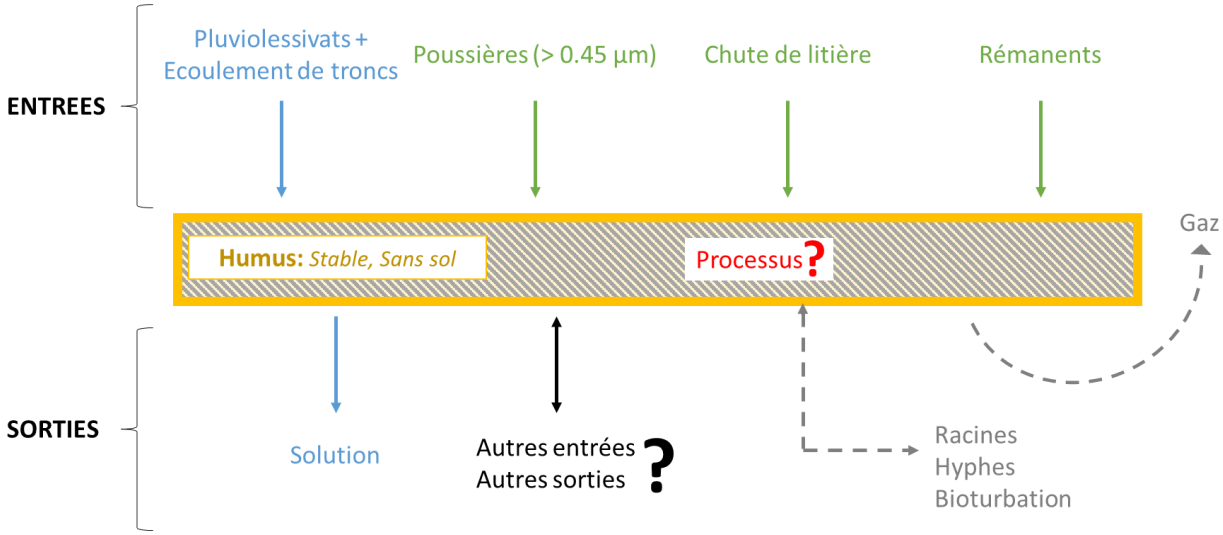

Figure 7: Modèle conceptuel de l'étude de la dynamique des éléments basé sur les flux d'entrées et de sorties de l'humus, en bleu les flux en solution et en vert les flux solides. Les processus correspondent aux processus ayant lieu dans l'humus non ou peu documentés. La double flèche noire correspond aux entrées et sorties non ou peu documentées.

Pour répondre au mieux à ces objectifs, cette thèse est divisée en 5 grandes parties (les chapitres 3, 4, 5, 6 et 7) (Figure 8) qui répondront aux questions et hypothèses posées ci-dessous :

⇨ **Question 1** : À partir des entrées solides et du stock des éléments dans l'humus, quels sont les temps moyens de résidence des éléments dans les trois humus étudiés et quels sont les mécanismes associés ?

Hypothèse 1 - La forme des éléments dans les entrées solides influence leur temps moyen de résidence dans l'humus.

Hypothèse 2 - La localisation des éléments dans les végétaux influence leurs temps moyens de résidence dans l'humus.

Hypothèse 3 - Le recyclage biologique et abiotique influence le temps moyen de résidence des éléments dans l'humus.

⇨ **Question 2 :** Tous les flux entrants et sortants de l'humus sont-ils identifiés et quel est l'importance de chacun des flux ?

Hypothèse 1 - Il existe un flux de particules solides en sortie de l'humus, comme il en existe un en entrée, qui pourrait permettre de boucler les bilans des éléments dans l'humus.

Hypothèse 2 - La contribution des flux d'entrées et des flux de sorties diffère selon l'élément.

⇨ **Question 3** : À quelle période sont libérés les éléments dans la solution de l'humus et concorde-t-elle avec la période de prélèvement de l'arbre ?

Hypothèse 1 - Les éléments sont libérés dans la solution lors de la période de végétation.

Hypothèse 2 - La libération des éléments en solution est influencée par plusieurs facteurs dont les précipitations atmosphériques.

⇨ **Question 4** : Une étude expérimentale à l'aide de sachets de litière peut-elle refléter la réalité de la dégradation de la litière en forêt ?

Hypothèse 1 - Des facteurs expérimentaux comme les variations interannuelles ou la taille des mailles des sachets peuvent influencer la vitesse de dégradation des litières.

Hypothèse 2 - Le type de sol peut influencer la vitesse de dégradation des litières.

Hypothèse 3 - Les vitesses de dégradations expérimentales sont similaires aux temps moyens de résidence de l'humus et la méthode par sachets de litière est un bon indicateur des vitesses des humus.

⇨ **Question 5** : Quelles sont les co-évolutions entre les éléments à l'entrée, à la sortie et dans l'humus ? Quels sont les mécanismes et les processus influençant ces évolutions ?

Hypothèse 1 - La co-évolution des éléments est influencée par la source de ces éléments (végétaux, poussière, solution).

Hypothèse 2 - La forme des éléments influence la co-évolution des éléments (soluble, non soluble) dans les entrées, les sorties et dans l'humus.

Pour répondre aux questions posées et vérifier nos hypothèses, différentes approches ont été développées sur le site expérimental de Montiers (55), essentiellement par des **approches d'observations** directes à l'aide d'une quantification *in situ* des flux et des stocks d'éléments afin d'avoir un suivi en conditions réelles et par l'observation au microscope électronique à balayage des litières, des poussières atmosphériques et des particules en sortie de l'humus. Une **approche expérimentale** a également été développée pour étudier la dégradation de la litière sur plusieurs années et sous différentes conditions expérimentales par la méthode des sachets de litières. Une dernière **approche théorique** a été mise en oeuvre afin de déterminer les relations entre les éléments et de créer un schéma de synthèse de la dynamique des éléments dans l'humus.

Cette thèse prend le parti de traiter principalement la dynamique des éléments autres que C et N qui sont habituellement étudiés. Les résultats obtenus pour ces éléments sont toutefois présentés aussi souvent que possible même si l'absence des flux gazeux ne permet pas de raisonner en terme de bilan.

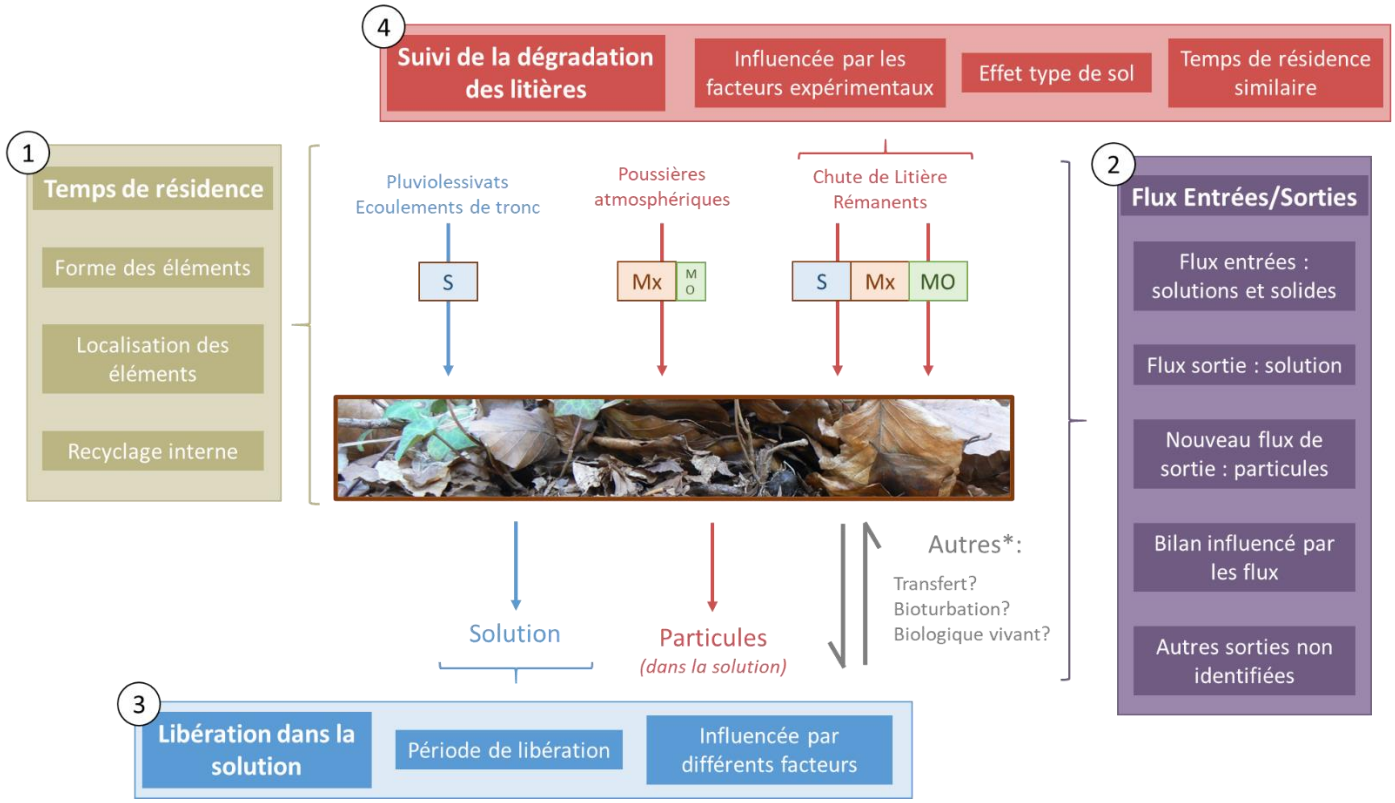

Figure 8 : Schéma des différentes questions traitées dans ce projet de thèse. Mx : minéraux, MO : matière organique, S : soluble. [Temps de résidence] Question 1 (chapitre 3) ; [Bilan entrées/sorties] Question 2 (Chapitre 4) ; [Dynamique de libération des éléments en solution] Question 3 (Chapitre 5) ; [Suivi expérimental de la litière] Question 4 (Chapitre 6) ; [Relations stœchiométriques dans l'humus] Question 5 (Chapitre 7).

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II-

Chapitre II -

Matériels et Méthodes

1 Le site expérimental de Montiers

1.1 Description générale du site

1.1.1 Généralités

Cette thèse a été réalisée grâce aux données collectées sur le site expérimental du massif de Montiers situé dans la partie Nord-est de la France dans le département de la Meuse (5° 16' 8'' E / 48° 31' 55'' N), à l'est du village de Montiers-sur-Saulx (Figure 9). La mise en place de ce site en 2011 résulte d'un projet commun entre l'INRAE-BEF de Nancy (Institut National de Recherche pour l'Agriculture, l'Alimentation et l'Environnement - Laboratoire Biogéochimie des Ecosystèmes Forestiers) et l'ANDRA (Agence Nationale pour la gestion des Déchets Radioactifs). Le site de Montiers fait également parti du réseau national AnaEE (Analyses et Expérimentations pour les Ecosystèmes). Le but de ce site est de suivre sur le long terme les cycles biogéochimiques des éléments majeurs et mineurs dans une hêtraie mature. Ce site a été choisi selon les critères suivants :

- Un même peuplement (même âge et sylviculture représentative de la région)
- Un même climat
- Une surface de site importante (133 ha sur deux toposéquences)
- Toposéquence de 3 sols représentatifs de la région

Le site expérimental de Montiers est composé de 3 stations expérimentales situées entre 340 et 386m d'altitude, d'une tour à flux et de deux dispositifs sécheresses (toits automatisés). Le climat est semi-continental (hivers froid et été chaud). Entre 2012 et 2018 la température moyenne annuelle était de 9.4°C et les précipitations moyennes de 1081 mm.

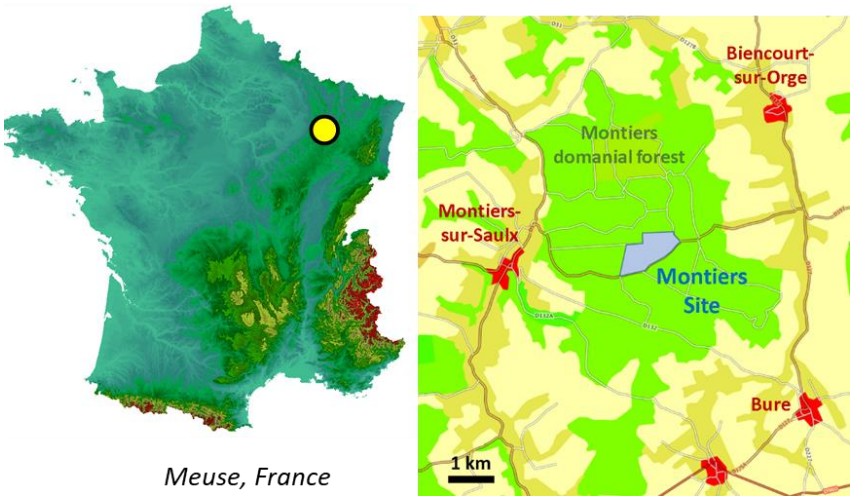

Figure 9 : Localisation géographique du site de Montiers

1.1.2 Peuplement

Le site expérimental de Montiers est localisé dans la forêt domaniale de Montiers-sur-Saulx, classée comme forêt depuis au moins 1830, indiquant qu'il n'y a pas eu de changement majeur de pratique durant les 200 dernières années. La forêt est gérée principalement en futaies de hêtre, celui-ci constituant 88% du peuplement. Des éclaircies ont lieu tous les 7 ans. Les dernières ont eu lieu en 2011 et en 2018 afin que le peuplement soit homogène sur les 3 stations (Figure 10). L'âge des hêtres a été estimé en 2009 à 45 ans sur S1, 54 ans sur S2 et 57 ans sur S3 (Genet et al., 2011). Leur hauteur dominante est de 27m. En terme d'espèce, le peuplement est composé de 88% de hêtres (*Fagus sylvatica*), 6% d'érables (*Acer pseudoplatanus*), 1.7% d'aliziers (*Sorbus torminalis*), 1.4% de frênes (*Fraxinus excelsior*), 1.2% de chênes (*Quercus robur L.*), 1% de charmes (*Carpinus betulus*), 0.4% de merisiers (*Prunus avium*) et 0.3% de noisetiers (*Corylus avellana*). Le peuplement présent sur le site est géré de la même manière que le reste de la forêt domaniale par l'Office National des Forêts (ONF).

Les indices foliaires annuels (LAI -*Leaf Area Index*) des couverts des trois stations mesurés en 2013 sont de 8.87 ± 1.82 pour S1, 9.55 ± 1.71 pour S2 et 8.64 ± 1.73.

1.1.3 Géologie

La géologie du site de Montiers est composée de la superposition d'un calcaire du Portlandien par des sédiments détritiques du Valanginien (Figure 10, Table 1)(Kirchen et al., 2017).

Le calcaire du Portlandien inférieur (-145 Ma) s'est formé lors de la sédimentation du Bassin Parisien. Il est composé principalement de carbonate de calcium (78% de la masse de la roche mère), de quelques minéraux de quartz (<5% masse totale de la roche) et d'environ 3.4% de minéraux argileux (Kirchen, 2017). Par le biais de l'altération du calcaire, une couche d'argile de décarbonatation s'est formée à la surface de la couche.

Les sédiments détritiques du Valanginien (-135 Ma) de type arénite-lutite surmontent le calcaire. Ces dépôts transgressifs côtiers sont composés d'argiles, de limons, de sables grossiers plus ou moins ferrugineux et de nodules de fer (composés majoritairement de goethite et de quartz)(Calvaruso et al., 2017).

Table 1 : Composition chimique des matériaux parents du site de Montiers d'après (Calvaruso et al., 2017) Calcareous bedrock = calcaire du Portlandien ; Detrial sediments = sédiments détritiques du Valanginien.

Parent Material		Element (in % of dry soil)								
		SiO_2	Al_2O_3	Fe_2O_3	K_2O	MgO	CaO	P_2O_5	Na_2O	MnO
Calcareous bedrock	Total	2.34	0.68	0.25	0.14	0.51	54.63	0.05	0.04	0.01
	Clay minerals	63.13	17.10	4.86	3.87	1.66	0.29	0.21	0.12	0.01
Detrial sediments		52.91	13.37	23.64	0.80	0.49	0.14	0.41	0.13	0.20

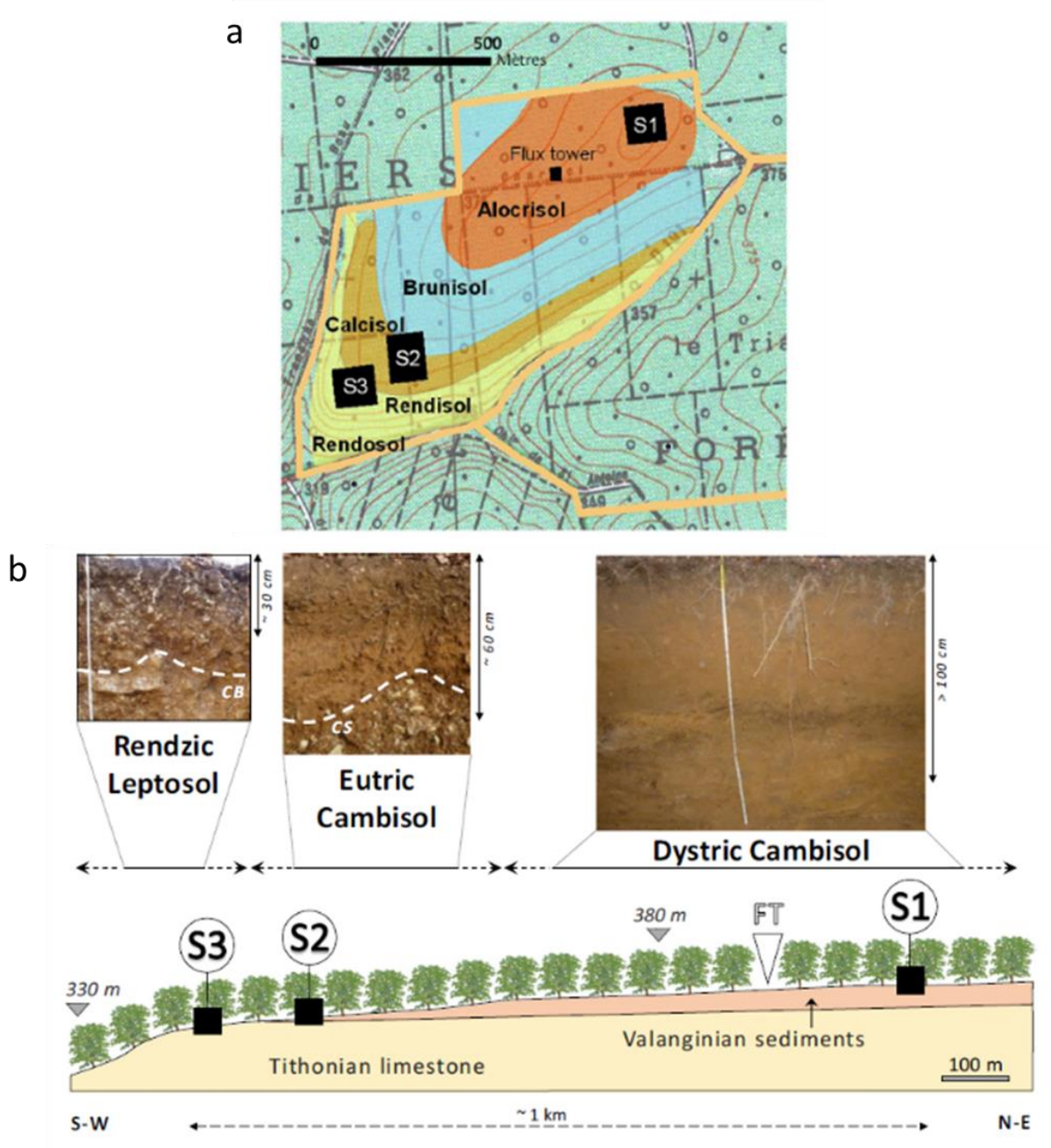

Figure 10: Schéma de la géologie et pédologie du site expérimental de Montiers. a) Localisation des trois stations expérimentales (S1, S2 et S3) et de la tour à flux (Fux tower). b) Représentation schématique de la toposéquence du site de Montiers et photo des 3 sols. FT= tour à flux ; Thithonian limestone = calcaire du Portlandien inférieur ; Valanginian sediments = sédiments détritiques du Valanginien

1.1.4 Pédologie

Le site de Montiers est localisé sur 3 sols suivant une toposéquence allant de l'alocrisol (Dystric Cambisol) en haut de pente, au calcisol (Eutric Cambisol) puis au rendisol (Rendzic Leptosol) en bas de pente (FAO, 2006; Kirchen et al., 2017). Les trois stations expérimentales ont été positionnées sur ces trois sols (Figure 10).

Table 2 : Profils racinaires dans les différents sols

	Apparition de la roche mère calcaire	Profondeur max. des racines fines (observation)	Proportion du système racinaire
DC	/	-170 cm	*-60 cm* 98% des racines fines
EC	85 cm	-140 cm	*-60cm* 93% des racines fines
RL	44 cm	Enracinement dans la roche mère ; profondeur d'observation max. : -120 cm	/

Le Dystric Cambisol (DC, station S1), s'est développé sur la couche de sédiment Valanginien sur une profondeur de 2m en moyenne et présente une texture limoneuse en surface et plus argileuse en profondeur. Lors de l'étude de ce sol, la roche mère n'a pas été atteinte, la profondeur maximale correspond donc à la profondeur de disparition des racines. Sa réserve utile est de 204.5 mm et le pH_{eau} de ce sol relativement peu acide est inférieur à 5 dans les horizons supérieurs. Sa capacité d'échange cationique (CEC) est <6.7 $cmolc.kg^{-1}$ dans les premiers 60 cm et son taux de saturation se situe entre 26 et 64% avec Ca^{2+} et Al^{3+} comme cations échangeables dominants (Table 3). Les racines fines sont présentes jusqu'à -170cm mais 98% de ces racines sont présentes dans les 60 premiers centimètres du sol (Table 2).

L'Eutric Cambisol (EC, station 2), est un sol intermédiaire entre l'alocrisol et le rendisol car il s'est formé sur une couche de sédiment plus fine que pour l'alocrisol. Il fait en moyenne 60cm de profondeur (présence de la roche mère) et présente une réserve utile de 83.9 mm. En surface, sa texture est limono-sableuse puis argileuse en profondeur. Le pH_{eau} est ici moins acide car il est supérieur à 5 tout le long du profil. La CEC est comprise entre 7.7 et 17.8 $cmolc.kg^{-1}$ avec un taux de saturation entre 59 et 83% dont Ca^{2+} est le cation échangeable dominant (Table 3). Les racines fines pénètrent ici directement dans la roche et sont présentes jusqu'à -180cm : les racines fines sont donc présentes tout le long du profil (93% dans les premiers 60cm de sol) (Table 2).

Le Rendzic Leptosol (RL, station 3) est situé directement sur le calcaire bien que quelques alluvions du Valanginien puissent être retrouvées. C'est un sol peu épais (30 cm en moyenne) avec un pH_{eau} aux alentours de 6. Sa réserve utile est la plus faible avec

57.46mm et sa CEC est comprise entre 20 et 25 cmolc.kg^{-1} avec un taux de saturation >94% représenté très majoritairement par les cations Ca^{2+} (Table 3). Les racines sont présentes très profondément dans la roche et donc tout le long du profil (Table 2).

1.1.5 Les humus

Sur le site de Montiers, les humus (ensemble des couches holorganiques) sont composés d'une couche de litière (OL) sur les trois sols et sur DC (S1) et EC (S2) d'une couche très discontinue de matière organique fragmentée (OF) (Figure 11). Les humus du site sont en moyenne de 3 cm d'épaisseurs et aucune racine n'a été observée. En tenant compte de l'horizon A, ces humus sont classés comme des formes d'humus mull (Brethes et al., 1995), et plus précisément comme des mulls acides sur DC et EC et un mull eutrophe sur RL. Ils sont caractérisés par la disparition rapide de la litière sous l'influence du climat, par l'homogénéisation de la matière organique humifiée avec des particules minérales au sein de macro-agrégats par des animaux fouisseurs/décomposeurs (par exemple les vers de terre) et des pourritures blanches (Ponge, 2003).

Figure 11: Coupe de l'humus du mull acide (DC), la ligne jaune représente la limite avec l'horizon organo-minéral.

La différence de forme de mull entre DC, EC (mull acide) et RL (mull eutrophe) (S3) est due à la différence de pH entre les 3 humus. Cependant, le stock en éléments dans les trois humus reste très similaire et n'a statistiquement pas varié entre 2010 et 2018 (Table 4).

Table 3: Propriétés physico-chimiques des trois sols étudiés sur le site de Montiers. Sont présentés la densité apparente (B. density), la répartition des textures (clay= argile : <2 µm ; F.silt= limons fins : 2-20 µm ; C. silt = limons grossiers, F. sand = sable fins : 50-0.2mm ; C.sand = sables grossiers : 0.2-2mm), le volume total de roche (RV), la capacité de rétention de l'eau du sol (SWHC), le pH eau du sol, la quantité de matière organique (OM), la capacité d'échange cationique (CEC) et le taux de saturation du sol (S/CEC avec S la somme des cations basiques) (Kirchen et al., 2017).

	Depth	B. density	Clay	F. silt	C. silt	F. sand	C. sand	RV	SWHC	pH_{water}	OM	CEC	S/CEC
	cm	g cm^{-3}	g kg^{-1}					%	mm		g kg^{-1}	cmol + kg^{-1}	%
S1 Dystric Cambisol	0–5	0.98	255	281	160	185	121	1.4	8.2	4.9	68	6.7	64
		0.12	*25*	*24*	*17*	*36*	*19*				*22*	*3.0*	*23*
	5–15	0.94	245	276	162	184	131	1.4	16.5	4.8	43	4.2	35
		0.17	*26*	*29*	*17*	*40*	*24*				*16*	*2.2*	*21*
	15–30	1.23	268	280	161	170	115	1.8	22.7	4.8	26	3.5	26
		0.22	*28*	*31*	*21*	*44*	*31*				*9*	*0.9*	*14*
	30–45	1.36	306	262	150	161	119	2.3	22.6	4.9	15	4.3	36
		0.18	*65*	*45*	*27*	*47*	*32*				*5*	*1.6*	*16*
	45–60	1.45	355	229	126	166	141	3.6	18.1	5.1	10	5.7	55
		0.15	*100*	*45*	*31*	*49*	*39*				*2*	*2.6*	*22*
S2 Eutric Cambisol	0–5	1.03	242	242	143	290	83	2.3	9.2	5.4	73	10.1	83
		0.11	*52*	*16*	*13*	*36*	*24*				*26*	*5.4*	*14*
	5–15	0.93	241	246	145	287	82	3.1	18.2	5.2	45	7.8	59
		0.13	*65*	*17*	*13*	*45*	*24*				*29*	*7.3*	*24*
	15–30	1.23	294	234	136	273	64	7.6	19.1	5.3	27	7.7	61
		0.19	*83*	*23*	*17*	*55*	*11*				*13*	*3.9*	*23*
	30–45	1.35	420	188	107	214	71	29.0	14.7	5.3	17	13.2	68
		0.18	*141*	*43*	*31*	*63*	*20*				*8*	*6.9*	*27*
	45–60	1.32	523	154	85	176	63	40.3	10.3	5.4	11	17.8	76
		0.23	*136*	*42*	*32*	*57*	*31*				*4*	*8.8*	*17*
S3 Rendzic Leptosol	0–5	0.88	449	227	123	119	41	2.3	9.8	5.7	109	24.9	98
		0.14	*80*	*54*	*26*	*39*	*15*				*27*	*8.3*	*5*
	5–15	0.98	430	224	114	123	59	4.9	19.2	5.7	71	20.0	94
		0.12	*82*	*56*	*36*	*37*	*21*				*23*	*7.9*	*7*
	15–30	1.06	516	169	77	102	63	36.4	12.5	6.0	42	23.2	99
		0.22	*81*	*50*	*38*	*42*	*24*				*10*	*6.4*	*5*

Table 4 : Stock moyen des éléments et de la masse sèche dans les humus des trois sols de Montiers en 2010 et 2018. L'écart-type est donné entre parenthèses. Il n'y a pas de différence significative entre les 2 années. Les différentes lettre représentent les différences significative dans un même sol (Test de Kruskal-Wallis, p-value<0.05). La différence significative entre les sols est donnée par une astérisque. La méthode utilisée est présentée en 2.3.

		Stock (kg. ha^{-1})												
		Masse sèche	C	N	Ca	Si	Fe	Al	K	Mn	S	Mg	P	Na
S1	2010	11139 (1900)^{a}	4940 (847)^{a}	178 (27)^{b}	168 (24)^{b}	148 (37)^{b}	55 (39)^{bc}	19 (5)^{cd}	18 (2)^{cd}	15 (4)^{cd}*	14 (2)^{cd}	13 (1)^{d}	11 (2)^{d}	1.4 (0.2)^{e}
	2018	11952 (1448)	5118 (668)	159 (29)	187 (15)	132 (28)	25 (32)	14 (7)	16 (1)	20 (3)	nd	15 (3)	14 (2)	1.2 (0.3)
S2	2010	9601 (1432)^{a}	4280 (609)^{a}	134 (25)^{b}	160 (24)^{b}	82 (19)^{b}	20 (9)^{bc}	14 (2)^{cd}	16 (1)^{c}	11 (2)^{cde}*	10 (2)^{de}	11 (1)^{cde}	9 (2)^{e}	1.2 (0.1)^{f}*
	2018	9108 (438)	3898 (326)	89 (7)	141 (23)	74 (21)	10 (20)	12 (5)	12 (1)	9 (1)	nd	9 (0)	9 (0)	0.8 (0.3)
S3	2010	8758 (1519)^{a}	3855 (679)^{a}	116 (22)^{b}	195 (36)^{b}	85 (14)^{b}	13 (4)^{cd}	20 (11)^{cd}	16 (3)^{c}	4 (1)^{e}	10 (2)^{d}	11 (2)^{ce}	8 (2)^{de}	1.0 (0.1)^{f}*
	2018	7744 (1710)	3474 (740)	82 (22)	155 (35)	76 (21)	4 (1)	11 (3)	11 (2)	3 (1)	nd	7 (2)	7 (2)	0.8 (0.2)

2 Échantillonnage

2.1 Dispositifs des stations pour les échantillonnages

Le site de Montiers est un site de suivis biogéochimiques qui a pour but de déterminer l'impact du type de sol sur le fonctionnement biogéochimique d'une même hêtraie. Afin de réaliser ce suivis, 3 stations ont été installées le long de la toposéquence sur les trois types de sols étudiés (Figure 10) : Dystric Cambisol - Station 1 ; Eutric Cambisol - Station 2 ; Rendzic Leptosol - Station 3.

Chaque station couvre 1ha de forêt et est divisée en quatre sous-stations (2500 m^2 chacune): 3 sont équipées pour le suivi biogéochimique et la dernière est laissée comme réserve (Figure 12). En tout, 9 sous-stations sont instrumentalisées: S11, S12, S13, S21, S22, S23, S31, S32, S33. Chacune de ces sous-stations est équipée des mêmes instruments (Figure 13) :

- 4 gouttières pour collecter les pluviolessivats
- 6 colliers collecteurs des écoulements de troncs
- 8 fluteaux à chaque profondeur du sol pour collecter les eaux gravitaires (litière, -10cm, -30cm, -60cm, -90 cm et -120cm)
- 4 bougies poreuses à chaque profondeur de sol pour collecter les eaux liées (-10cm, -30cm, -60cm, -90 cm et -120cm)
- 3 pièges à litières pour collecter la chute des litières
- 4 sondes TDR pour suivre l'humidité et la température du sol à chaque profondeur (10cm, -30cm, -60cm et -90 cm)
- Des dendromètres répartis équitablement pour suivre la croissance des arbres

Les relevés pour les pluviolessivats, les écoulements de troncs, l'eau du sol (gravitaire ou liée) et les dendromètres sont effectués toutes les 4 semaines. Le relevé des litières se fait 7 fois par an. Depuis fin de 2011, l'humidité du sol est relevée toutes les 12h et la température toutes les heures. Les feuilles fraîches sont récoltées annuellement sur des arbres situés à proximité de chaque station en août depuis 2011..

Une tour à flux de 45m de haut a été installée proche de la station S1. Son but est de pouvoir collecter les dépôts atmosphériques, les précipitations hors couverts et les paramètres climatiques. Trois collecteurs de pluies sont disposés au sommet de la tour à flux. Il s'agit d'entonnoirs circulaires de 0.24 m^2 de surface réceptrice et reliés à des bidons de 50L en polyéthylène. Afin de relever en continu les principaux paramètres météorologiques, plusieurs capteurs sont installés en haut de la tour à flux. Les données sont enregistrées toutes les 30 min. En complément des données récoltées sur site, une demande d'information météorologique est effectuée auprès de Météo France dans le cadre de la convention de recherche entre Météo France et l'INRAE.

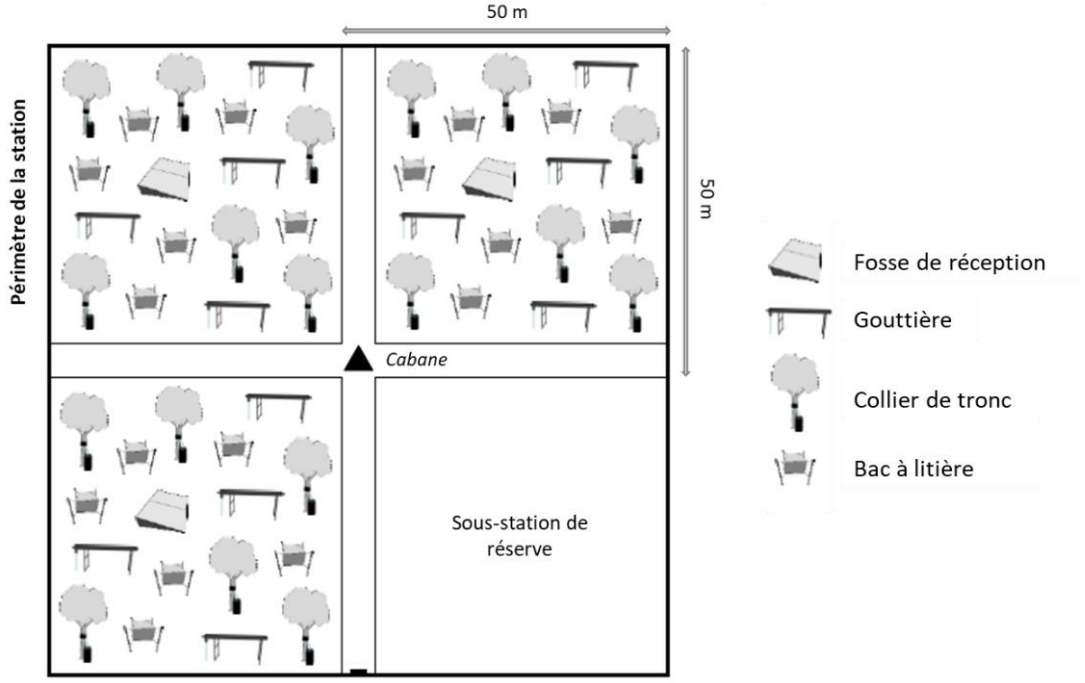

Figure 12: Schéma de la disposition des quatres sous-stations d'une station expérimentale du site de Montiers (Kirchen, 2017).

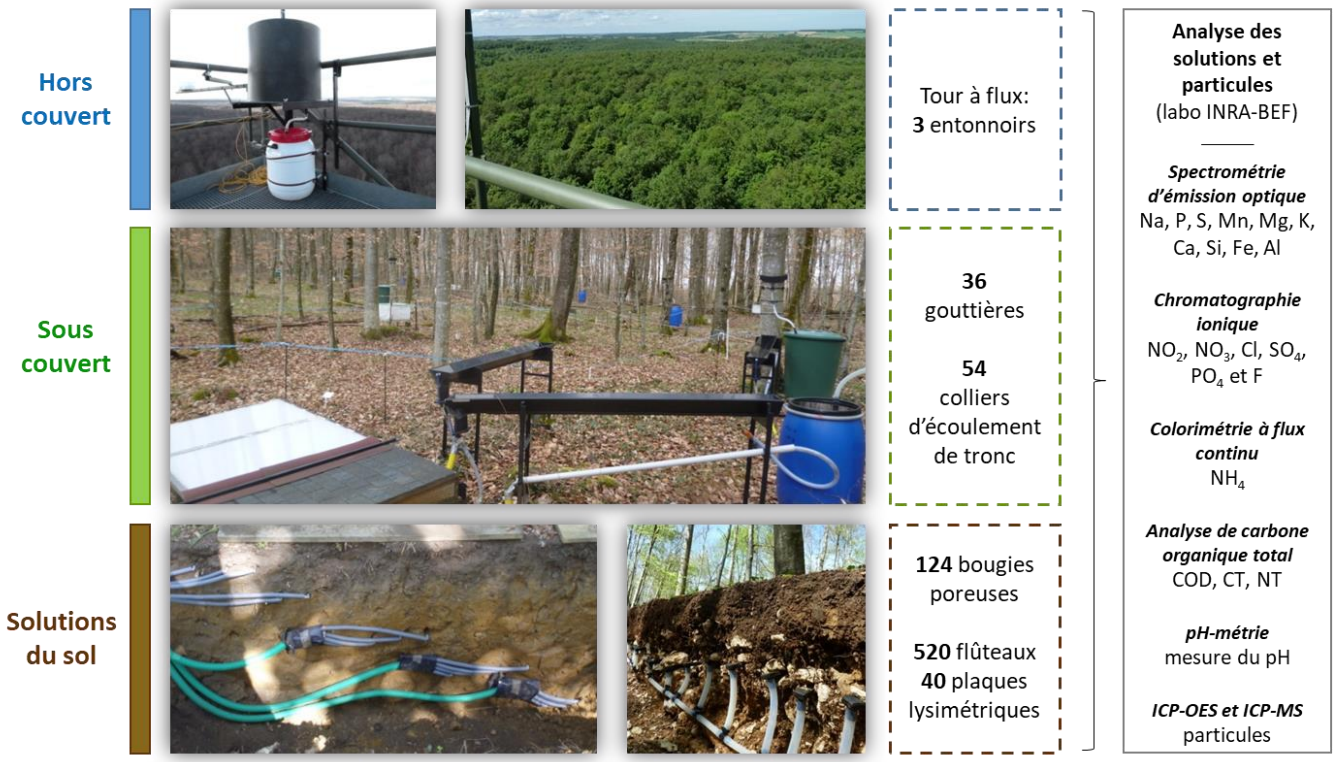

Figure 13: Représentation du nombre d'instruments présents sur le site de Montiers pour collecter les solutions et les particules et les différentes analyses associées.

2.2 Poussières et particules

Toutes les poussières atmosphériques (>0.45µm) ont été collectées dans les solutions hors couverts à partir de 2012. Les particules dans les solutions à la sortie de l'humus ont été collectées en 2017 et 2018 dans S1 et S3. Les solutions récoltées au-dessus du couvert sur la tour et dans les fluteaux sous l'humus sont centrifugées pendant 40 minutes à 3500 $tr.min^{-1}$ pour séparer la phase solide (particules) des solutions (Lequy et al., 2014).

2.3 Suivi de l'humus

Afin de suivre l'évolution du stock d'éléments dans les humus des trois stations, deux campagnes d'échantillonnage ont eu lieu en 2010 et en 2018 (à chaque fois, 27 réplicas par station soit 81 échantillons en tout) avec la même personne présente pour expliquer la méthodologie et le calage. L'échantillonnage à la main de l'humus a été effectué à l'aide d'un cadre calibré de 0.1 m^2 suivant une grille systématique (Figure 14).

Figure 14: a- Cadre calibré de 0.1 m^2 délimitant la zone de prélèvement; b- prélèvement de l'humus en triant au maximum le sol; c- zone du cadre après le prélèvement

2.4 Expérience de dégradation des litières

Entre 2013 et 2019, 5 manipulations de sachets de litières ont été réalisées. En automne 2013 et 2018, des feuilles de litières ont été collectées grâce à des filets étendus sous des hêtres dans chaque station (Figure 15). Les feuilles collectées en 2013 ont été séchées à 60°C puis triées à la main afin de garder uniquement les feuilles de hêtres. Elles ont ensuite été séparées pour 3 manipulations : M1, M2 et M3. Pour M1, 2.0 g de feuilles séchées ont été placées dans des sachets de mailles de 0.5cm et de surface 20x20 cm et collectées entre décembre 2013 et décembre 2016 (Figure 15). Pour M2 et M3, 6,0 g de feuilles séchées ont été placées dans les mêmes sachets et récoltées entre décembre 2014

et juin 2018 pour M2, et entre décembre 2015 et janvier 2018 pour M3 (Figure 16). Pour ces 3 manipulations, 324 sachets ont été placés sur les différentes stations.

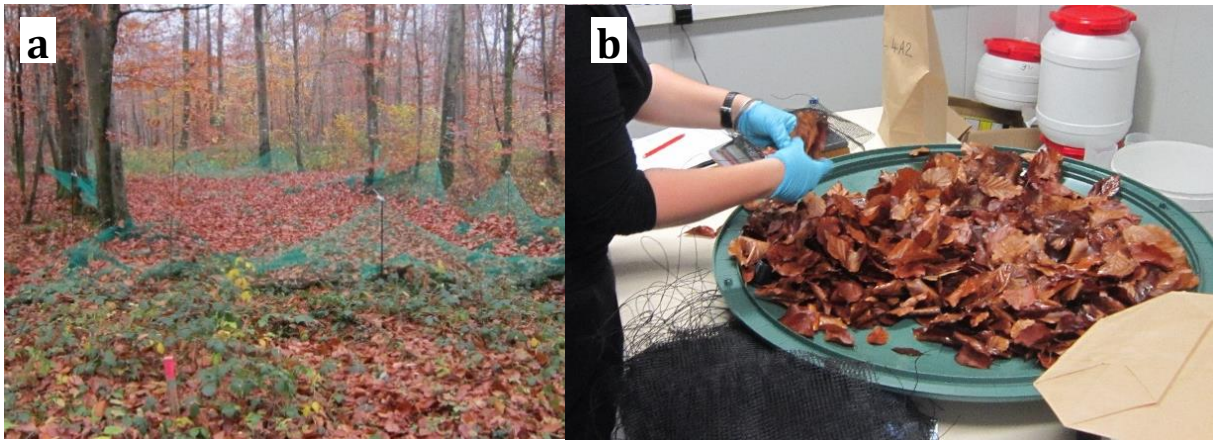

Figure 15 : a - Collecte des feuilles de hêtre tombées dans le filet de récupération des litières ; b - Tris et pesées des feuilles afin de réaliser les sachets de litières

Les feuilles de hêtre échantillonnées en 2018 ont été utilisées pour les manipulations M4a et M4b. Pour M4b, les feuilles sont séchées à 60°C et 10g sont placés dans des sachets de mailles de 1cm et de 22 x 22cm de surface. Au total, 72 sachets sont placés dans les différentes stations. Pour M4a, 40g de feuilles de hêtre humides sont placés dans 126 sachets (les même que pour M4b) et sont répartis sur les 3 stations. Les sachets de litières de M4a et M4b sont collectés entre novembre 2018 et mars 2020 (Figure 16).

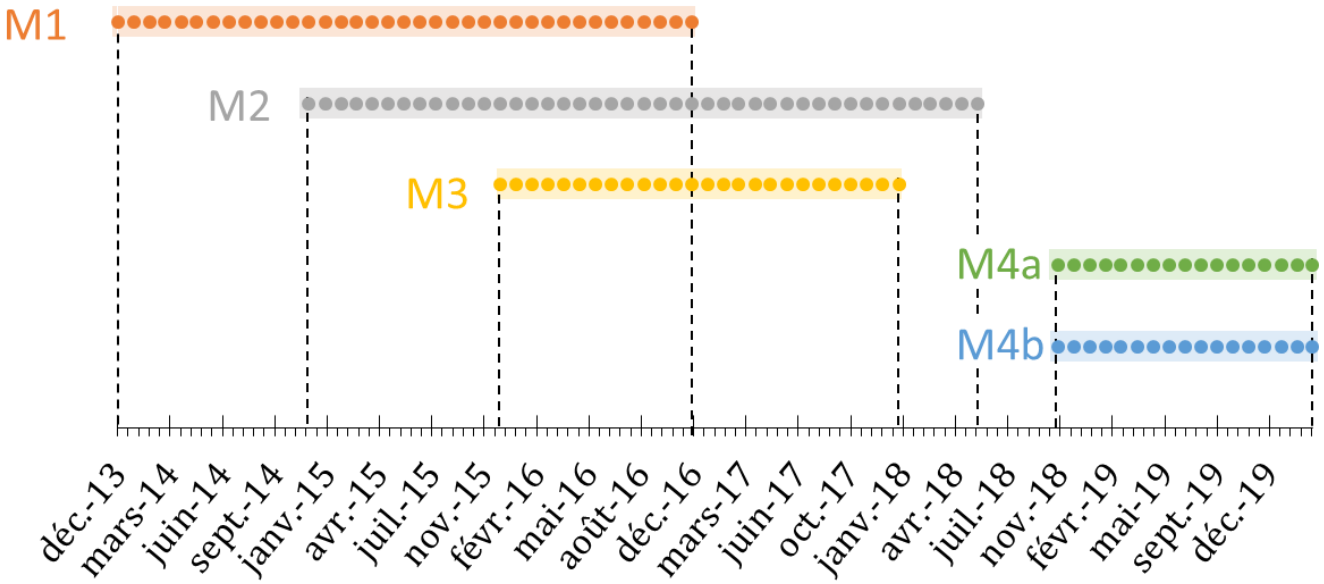

Figure 16: Date de début et de fin des différentes manipulations par sachets de litières

3 Méthodes analytiques

3.1 Analyses des solutions

Les échantillons de solutions du sol et hors-sol sont placés dans des flacons en polyéthylène et sont immédiatement transportés au laboratoire de l'unité de Biogéochimie des Ecosystèmes Forestiers de l'INRAE de Champenoux. Ils sont ensuite filtrés sous vide à 0.45 µm par filtration sur membrane puis analysés le jour même. Les paramètres analysés sont les suivants (Figure 13) :

- Le pH à l'aide d'un pH-mètre (DL70 ES, METTLER TOLEDO). L'incertitude de mesure est de 0.05 unité de pH.
- Les teneurs en Na, P, S, Mn, Mg, K, Ca, Si, Al, et Fe par un spectromètre d'émission optique (700 Series ICP-OES, AGILENT TECHNOLOGIES).
- Les anions NO_2^-, NO_3^-, Cl^-, SO_4^{2-}, PO_4^{3-} et F^- par chromatographie ionique (ICS 2100, DIONEX).
- L'ammonium (NH_4^+), mesuré par colorimétrie à flux continu (spectromètre d'absorption moléculaire San^{++}, SKALAR).
- Le carbone organique dissous (DOC), le carbone total (TC) et l'azote total (TN) par analyseur de carbone organique total (TOC-L, SHIMADZU).

Les incertitudes de ces mesures sont toutes inférieures à 10 %.

3.2 Analyse des particules

Les particules, après la centrifugation, sont séchées à 35°C puis broyées. Les particules atmosphériques et les particules de l'humus ont été regroupées par années. Elles ont ensuite été analysées par le laboratoire CNRS (SARM Nancy) en utilisant la spectrométrie d'émission optique à plasma inductif (ICP-OES) pour les éléments majeurs et la spectrométrie de masse à plasma inductif (ICP-MS) pour les éléments traces (Carignan et al., 2001).

3.3 Analyse des végétaux

Les échantillons de végétaux sont séchés à 60°C durant 3 jours, puis broyés dans un broyeur mécanique. Chaque poudre obtenue est ensuite compactée en pastilles de 0.3g qui sont analysées par un spectromètre à fluorescence X (X fluorescence sequential spectrometer S8 TIGER 1kW, BRUKER). Le carbone et l'azote total sont dosés par un analyseur CHN (EA/NA 1110, THERMO QUEST).

3.4 Analyses par microscopie électronique à balayage

Afin d'étudier les différentes formes de recyclage des éléments dans l'humus, des échantillons de végétaux alimentant l'humus et des fragments d'humus ont été analysés par microscopie électronique à balayage (MEB). Les échantillons sont, dans un premier temps, montés sur des lames en verre en utilisant des bandes conductrices en carbone à double revêtement puis recouverte d'une fine couche de carbone. L'observation et l'analyse qualitative des échantillons sont réalisées avec un MEB Hitachi S-4800 (Figure 17) équipé d'un spectromètre à rayons X à énergie dispersive (EDX) et contenant un détecteur au silicium enrichi au lithium. Les analyses MEB ont été effectuées en utilisant une tension d'accélération de 3 ou 15 kV avec une distance de travail de 15 mm. Les images obtenues sont réalisées par électrons rétrodiffusés ou électrons secondaires.

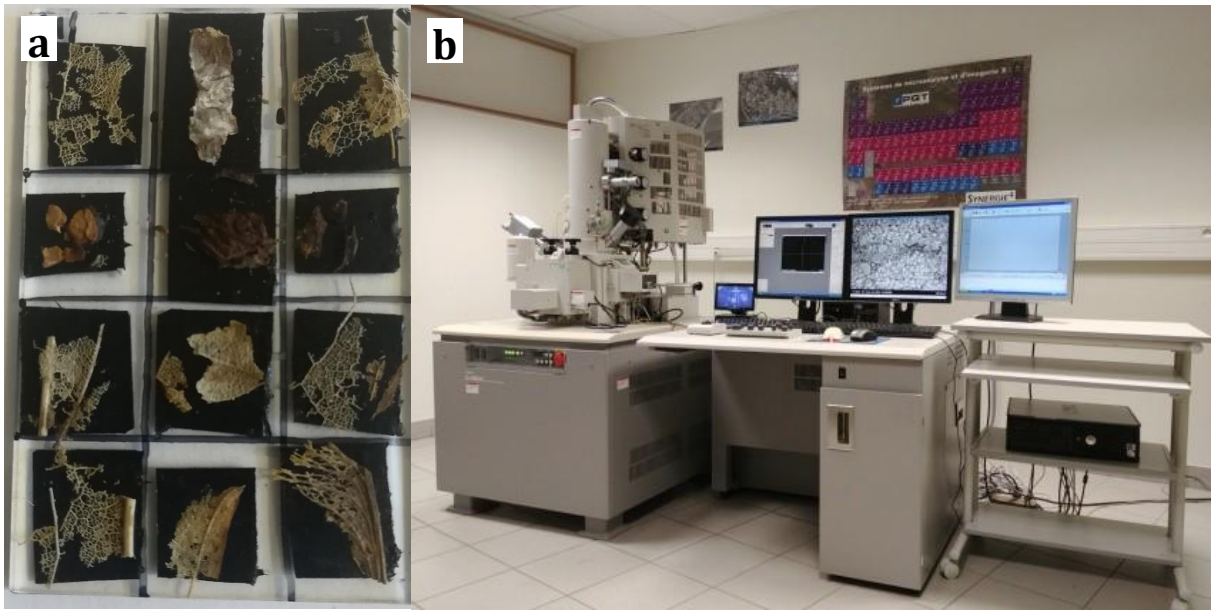

Figure 17: a- Exemple d'une lame préparée pour une analyse au MEB b- MEB utilisé pour les analyses de litières et humus ©Georessource

4 Calculs, modèles et statistiques

Les différents calculs et modèles utilisés sont décrits en détail dans la partie maétriels et méthodes des chapitres III à VI de la thèse. Les statistiques réalisées y sont également détaillées.

III

Chapitre III -

Major element residence times in humus from a beech forest: the role of element forms and recycling

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Ce chapitre présente une étude basée sur les flux entrants solides et le stock des éléments dans les humus. Il tend à répondre à la première question de recherche : À partir des entrées solides et du stock des éléments dans l'humus, quels sont les temps moyens de résidence des éléments dans les trois humus et quels sont les mécanismes associés ?

Résumé

Cette étude présente la relation entre les entrées d'éléments solides (en particulier les biominéraux), les mécanismes de recyclage et les temps moyens de résidence des éléments majeurs dans des mulls.

Dans les écosystèmes forestiers où les entrées de nutriments sont généralement faibles, la décomposition de la litière est une source importante de nutriments pour le sol. Alors que les processus et les vitesses de libération des éléments comme C, N et P sont bien déterminés lors de la dégradation de la litière, la dynamique dans l'humus des éléments tels que K, Na, Ca, Si, S, Mg, Mn, Fe ou Al, dont certains sont essentiels à la nutrition des arbres, sont moins bien connus.

Les objectifs de cette étude sont de déterminer les temps moyens de résidence de ces éléments dans des humus de forme mull dans trois sols différents (Dystric Cambisol (S1), Eutric Cambisol (S2) et Rendzic Leptosol (S3)) dans une même forêt de hêtres du Nord-Est de la France et d'identifier les principaux mécanismes contrôlant ces temps moyens de résidence.

Pour atteindre ces objectifs, les approches utilisées sont : 1) l'observation au microscope électronique à balayage de l'évolution et du recyclage des éléments pendant la dégradation de la litière ; 2) la quantification des entrées solides totales et de leur forme soluble/insoluble dans la litière ainsi que de l'apport des résidus d'exploitation ; 3) la quantification et évolution des stocks de litière; et 4) le calcul et la comparaison du temps de résidence des éléments en fonction de leur forme.

Le calcul des entrées et des stocks d'éléments dans l'humus a permis d'évaluer le temps moyen de résidence de chaque élément dans l'humus. Les temps moyens de résidence sont compris entre 58,4 et 13,1 ans pour Fe et Al ; 3,3 et 1,6 ans pour Si, N, S et Ca ; 2,2 et 1,2 ans pour Mn, Mg, Na, P et C ; et 0,6 et 0,8 an pour K. Les résultats sont similaires pour les trois sols étudiés, à l'exception du stock et des entrées de Mn qui sont plus faibles dans S3 et des entrées de Si qui ont diminué de S1 à S3.

Les résultats de cette étude indiquent que les temps moyens de résidence de K, P, Na, Mg et S diminuent avec le pourcentage de forme soluble. À l'inverse, ces temps augmentent quand les éléments sont principalement sous forme de biominéraux (Si, Ca), dans les tissus végétaux plus résistants (nervures) ou dans des molécules organiques (N). Ils augmentent également lorsque les éléments sont repris dans des mécanismes de (i) sorption (Al ; Fe), (ii) de recyclage biotique, comme les amibes à thèques (Si, Ca, P, Mn) ou les précipités autour des hyphes (Ca, Mn, P), ou (iii) de recyclage abiotique (précipitation amorphe de Si).

Major element residence times in humus from a beech forest: the role of element forms and recycling

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1 Introduction

Decomposing leaf litter is an important source of nutrients and beneficial elements, such as Si, in natural forests (Attiwill and Adams, 1993; Bartoli, 1983; Jacob et al., 2009). The aerial litter is composed of the annual litter falls of the tree aerial compartments (leaves, buds, beechnuts) and naturally falling branches, as well as the branches remaining on the ground during forest exploitation, which are called exploitation residuals (Jensen, 1984). This return of organic matter to the soil indicated a powerful biological cycle that is dominant in comparison to the geochemical one. For example, the stocks of Ca and Mg have been demonstrated to be larger in fresh litter in comparison to soil horizons at the experimental site of Breuil-Chenue in the Morvan Mountains in France (van der Heijden et al., 2017). The degradation of litter in the humus results in rapid nutrient release into mull-type humus (Ponge, 2003), which may be delayed by internal litter recycling mechanisms (Ponge, 2013; Sommer et al., 2013).

Litter degradation has been extensively studied and is controlled by abiotic and biotic factors. Abiotic factors correspond to climate, geography, and soil properties. Biotic factors include litter quality, e.g., lignin, N, P and other nutrients, C/N, lignin/N and biological activity of micro and macro fauna (Berg et McClaugherty, 2014). One of the most well-known macro litter decomposers is the earthworm. Indeed, in terms of macro fauna biomass, this organism represents 100 to 1500 kg. ha^{-1} compared to protozoa which represent 20 to 200 kg. ha^{-1} (Brady and Weil, 2002). They have an important role in the decomposition of organic matter and its transfer to the soil (Sanderman and Amundson, 2003).

Because C and N are closely linked to organic molecules and constitute the main plant tissues, studies of the rate of litter degradation and quality have mainly focused on these two elements (Albers et al., 2004; d'Annunzio et al., 2008; García-Palacios et al., 2016; Melillo et al., 1982). Other elements, such as P in nucleic acids, S in certain proteins, or Mn in several enzymes, are partially present in organic matter. It is now accepted that elements in plant tissues are present in three forms: organic molecules (unlignified substances and lignin), biominerals and soluble molecules (Berg et McClaugherty, 2014; Fink, 1991; Krieger et al., 2017). Biominerals are the products of the selective uptake of elements from the local environment and their incorporation into functional structures under strict biological control e.g. calcium oxalate (Mann, 2001). Organic matter and biominerals, correspond to the insoluble form.

Although the proportions of the forms for all elements in different plant tissues is not known, it seems to be dependent on the elements. For example, in tree leaves, C and N are mainly present in organic molecules, and K can be found in soluble form (Schreeg et al., 2013). Ca is mainly found in insoluble form, either associated with pectin and other

organic molecule, not in covalent bond, but as an ionic pair (Jones et Lunt, 1967) or as CaOx crystals located primarily in leaf veins when Ca is strongly present in the soil (Krieger et al., 2017). The proportion of Ca in the form of CaOx should not be neglected in fresh and degraded litter. Dauer and Perakis (2014) found values between 34 and 70% of Ca as CaOx in annual litterfall and between 23 and 41% in the forest floor. Silica is also present in amorphous mineral form. It can line the cell walls in the leaf and veins (Krieger et al., 2017; Turpault et al., 2018). Thus the average residence time or dynamics of the elements in the litter will depend on their forms, distributions in plant tissues and solubilization speeds.

Biotic and abiotic recycling of nutrients and beneficial elements through the production of organism shells or precipitation of minerals in litter during degradation may delay their availability for trees. For example, it was shown that Si could be immobilized by testate amoebae to form their shell (Sommer et al., 2013; Wilkinson and Mitchell, 2010). CaOx crystals, sometimes combined with P, K or Mn, can precipitate around mycelial hyphae (Burford et al., 2003). As a consequence, there is a need to identify these recycling mechanisms and determine their potential roles in the residence time of the elements in litter.

The co-dynamics of elements other than C, N and P have been little studied until now (Fältmarsch et al., 2007; Laskowski et al., 1995; Ukonmaanaho et al., 2008). The challenge of this study was to highlight the dynamics of the major elements other than C, N and P and to determine their main control mechanisms in three mull-type humuses, which are more or less acidic and characterized by rapid turnover allowing for a short period of study.

The objectives of this study were to determine (1) the form of the elements (insoluble or soluble) in the inputs of the humus, (2) the evolution and recycling of the elements during the mineralization of aerial litter in a mull-type humus, (3) the average residence time of the elements in the humus, and (4) the dynamics of the major elements during the degradation of the humus. To achieve these objectives, two approaches were used. The first one was qualitative and consisted of the observation of biominerals and solid forms recycled in fresh plant tissues and degraded litter using the scanning electron microscope (SEM) in the secondary scanning of electrons (EGS). The second approach was quantitative and consisted of the determination of the fluxes of total inputs (litter and exploitation residuals) and the form of elements in these inputs (soluble and not soluble), as well as the assessment of the stock of elements in the humus and its evolution over 8 years. The average residence times of the elements were also calculated to determine the main mechanisms controlling the element dynamics.

2 Materials and methods

2.1 Experimental site

The study was carried out in the "Montiers" beech forest experimental site built in 2011 and co-managed since 2012 jointly by INRA-BEF (French National Institute for Agricultural Research – Biogeochemical Cycles in Forest Ecosystems research unit) and ANDRA (French National Radioactive Waste Management Agency). The Montiers site is part of the international research network AnaEE (https://www.anaee.com/). This temperate forested area is located in Northeastern France in the Meuse department (48° 31′ 54″ N, 5° 16′ 08″ E).

The climate is semi-continental (cold winters and hot summers) with a mean annual rainfall of 1069 mm and mean temperature over the last 20 years of 9.8°C (Météo France). In winter, soil can temporarily freeze over a depth of a few centimetres and occasionally be covered by snow. The Montiers site is covered by the same homogeneous forest: same age (~50 years old in 2010), same management and same species. The stand is mainly composed of beech (89%) and other species (11%), i.e., sycamore maple (*Acer pseudoplatanus*), ash (*Fraxinus excelsior*), pedunculate oak (*Quercus robur L.*), European hornbeam (*Carpinus betulus L.*) and wild cherry (*Prunus avium*). Every 7 years, the ONF (French National Forest Office) carries out a stand thinning and above-ground biomass (approximately 40 x 103 kg. ha-1) is cut in each of the three soils. Wood with a diameter >7 cm is exploited, and the exploitation residues <7 cm are left on the soil of the soils. The occupation of the Montiers forest has remained the same since at least 1830. The management carried out by the ONF has been homogeneous across the forest since the 1960s (with a cut every 7 years).

The Montiers site geology is composed of two overlapping soil parent materials: an underlying Tithonian limestone surmounted by acidic Valanginian detrital sediments. Due to the differences in thickness of the sediment layer, the site is composed of three different soil types: Dystric Cambisol (DC), Eutric Cambisol (EC) and Rendzic Leptosol (RL) (FAO, 2016). A schematic representation of the soil profiles and their locations, as well as the main characteristics of these different soil types, is presented by Kirchen et al. (2017). Humus type is a eutrophic mull for RL and EC and an acidic mull for DC. No roots were found in the humus of the 3 soils. The great abundance of earthworms observed in humus also highlights the high biological activity of humus on the three soils, especially in S3.

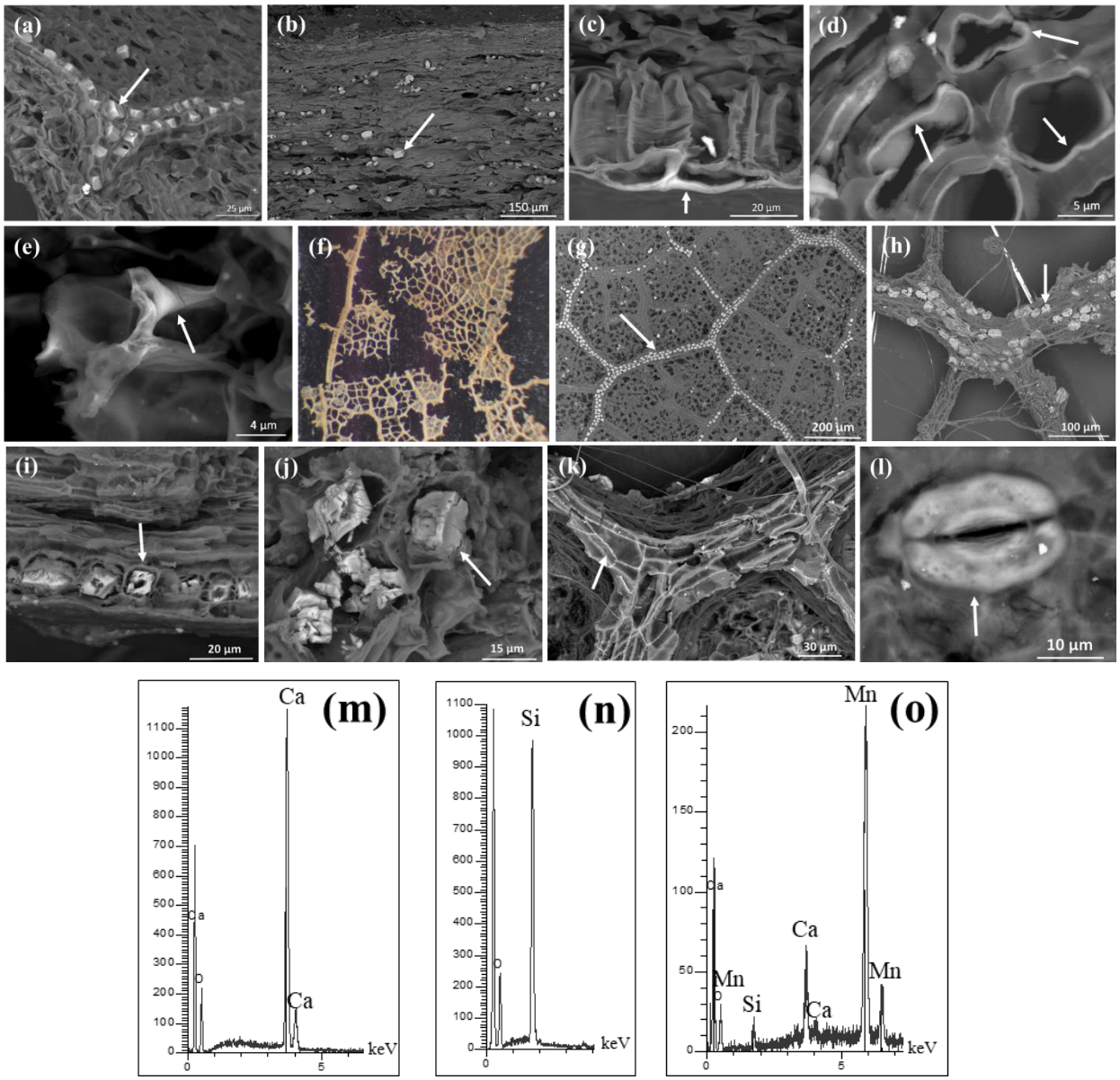

Figure 18 : Primary biominerals observed in leaves and wood (a to e) and in leaf litter (f to l) and EDX spectra of biominerals (m to o). (a) CaOx aligned in a leaf vein, (b) CaOx in the branch, (c) cell wall of leaf lower epidermis covered with Si, (d) cell wall of branch covered with Si, (e) cell wall of leaf covered with Mn, (f) degraded leaf in the humus, (g) primary CaOx aligned in leaf veins at the beginning of blade degradation, (h) primary CaOx in a vein without the blade (which are totally degraded), (i) primary CaOx aligned in a vein and traces of completely degraded oxalates (sectional view), (j) residues of CaOx in a degraded small bark branch, (h) details of the residues of branch CaOx, (k) silicified cell walls in a vein, (l) details of silicified stoma in blade, (m) EDX spectrum of CaOx, (n) EDX spectrum of amorphous Si, and (o) EDX spectrum of amorphous Mn.

Three soils (S1: DC; S2: EC: S3: RL), with an area of 1 ha each and separated by 300 to 1200 meters, were equipped to monitor water, element fluxes and tree growth. Three replicates for each soil had the same monitoring for collecting the above-ground and below-ground solutions at different depths, soil at different depths, organic horizons (3 lysimetric collectors per replicate), litterfall (4 gutters per replicate), tree compartments (stem flow and throughfall, 6 collectors per replicate) and for the standing above-ground

(6 litter traps per replicate) and below-ground biomasses. Additionally, a 45-m flux tower was placed between the soils to collect rainfall and atmospheric deposits. The data from the flux tower are not yet exploited and for the moment only one year has been studied. For more information, see Calvaruso et al. (2017), Kirchen et al. (2017) et Turpault et al. (2018).

2.2 Sampling

2.2.1 Humus and litterfall sampling

On the Montiers site, the humus layer is composed of the organic horizons Oln, Olv and Of, which discontinue above the organo-mineral horizon (Ah). Only on DC (S1) is the organic horizon Oh found discontinuously. The organic horizons were collected in June 2010 and May 2018 using the same protocol and the same operator to determine whether the elemental concentrations in the humus were stationary or had changed. Sampling was conducted with a calibrated metal frame (0.1 m^2). In the three soils (S1, S2 and S3) consisting of 3 replicates, nine samples were collected for a total of 162 samples. To avoid soil contamination as much as possible, the samples were collected very carefully.

For each replicate, litterfall was collected throughout the year between 2012 and 2015 using six litter-traps of 0.34 m^2 placed 40 cm above the forest floor. The content of the litter-trap was collected seven times per year for a total of 1 512 samples in 4 years. They were separated according to three fractions: (i) leaves, (ii) branches falling naturally from the trees and (iii) buds, beechnut and fruit capsules. The leaves and buds, beechnuts and fruit capsules belonged to annual tree compartments (recycling each year), while small branches belonged to perennial compartments. We consider that atmospheric deposits are deposited on the leaves and included in the analysis of leaf litter.

2.2.2 Tree compartments and fresh leaves

In each soil in 2009, three beech trees were harvested to collect wood, stem bark, branches and branch bark. The branches were separated into 3 classes based on their diameters: <4; 4-7; and >7 cm. The detailed procedure for collecting the samples has been described by Calvaruso et al. (2017). In August 2013, fresh leaves were collected from trees developed on different soil types by shooting off small branches with a shotgun.

2.3 Analytical methods

2.3.1 Microscopic analysis

Four litterfall and humus, fresh branch wood and fresh leaves samples were randomly selected from each replicate in 2018. The samples were mounted on glass plates using double-coated carbon conductive tabs and covered with carbon. Organic matter, degradation, fauna, microorganism colonization, fresh biominerals and their evolution in these samples, were observed and analysed using a Hitachi S-4800 scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDX) and containing a lithium-drifted silicon detector. SEM analyses were carried out using an acceleration voltage of 3 or 15 kV with a working distance of 15 mm. The obtained images consisted of backscattered electrons, excluding the images of bacteria, which consisted of secondary electrons.

2.3.2 Elements in litterfall, exploitation residuals and humus

Samples from the litterfall, humus and residual exploitation (wood left on the soil after a cut) were dried in a stream air-drier (65 °C) and then ground and encapsulated for analysis. The total Ca, K, Mg, P, Al, Mn, Na, S, Si and Fe contents were assessed by X fluorescence using an X fluorescence sequential spectrometer S8 TIGER 1 kW (Bruker). The total C and N was assessed using a CHN analyser (EA/NA 1110, THERMO QUEST).

2.3.3 Soluble form

To assess the soluble fractions of Ca, Si, Fe, K, Mg, P, Mn, S, Al and Na, an extraction with water was carried out for the litterfall and small branch fraction for the years 2012–2015. The samples were dried at 60 °C and crushed. To perform the extraction, 0.1 g of sample was weighed, suspended in 50 ml of ultrapure water and then shaken overnight. The samples were centrifuged at 20 °C for 10 min at 3000 rotations per minute. To determine the element concentration, the supernatant was then analysed using an inductively coupled plasma–atomic emission spectrometer (ICP-AES Agilent Technologies 700 type ICP-OES, Santa Clara, USA).

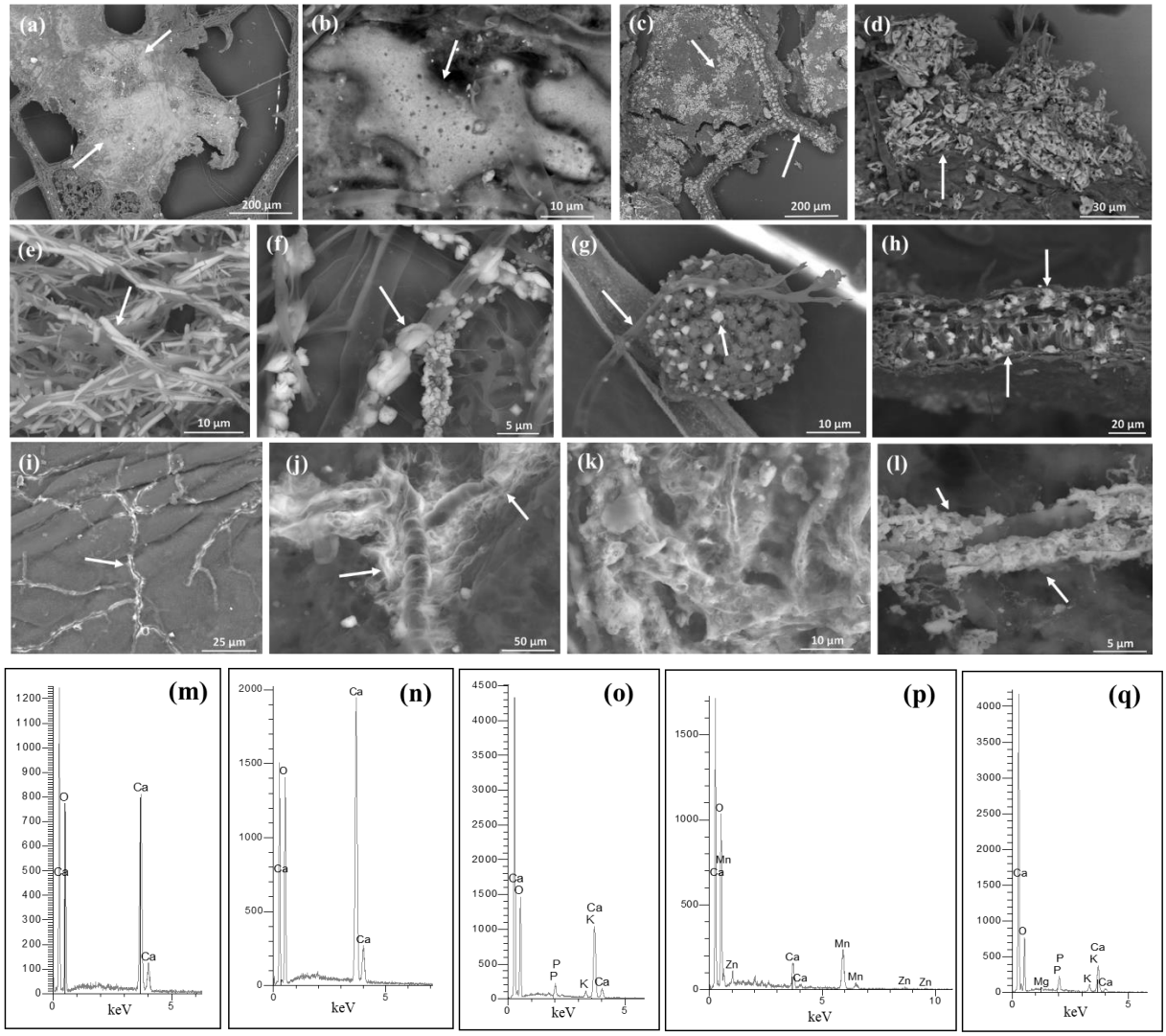

Figure 19 : Secondary minerals in leaves and small wood from litter of beech forest (a to l) and EDX spectra of biominerals (m to q). (a) Amorphous Si deposit in a large area on the leaf blade and vein, (b) Si deposit in the form of root grain, (c) vein with aligned and degraded primary oxalates and clusters of secondary oxalates on the blade, (d) cluster of secondary CaOx bordering a blade close to hyphae, (e) hyphae on the leaf with ovoid CaOx, (f) hyphae on the branch with elongated oxalates, (g) cubic CaOx crystals on the spore, (h) amorphous Mn in cells of the lower epidermis of the leaf, (i, j, k) hyphae surrounded by amorphous Mn, (l) hypha enveloped by Mn crystals, (m) EDX spectrum of secondary CaOx (e), (n) EDX spectrum of secondary CaOx (f), (o) elongated crystals of P and Ca (g), (p) EDX spectrum of amorphous Mn and Ca (i), and (q) EDX spectrum of the crystals surrounding a hypha (l).

2.4 Calculation of the elements concentrations forms and residence times

2.4.1 Element stocks in organic horizons

Since the lowest part of the organic layer is in direct contact with the soil, it is very difficult to perform the sampling without soil contamination. The percentage of soil mixed with the organic layer was calculated using the equation described by Turpault et al. (2018), taking into account the titanium content:

$$\text{Soil \%} = [\,[(Ti_{Hb} - Ti_{Hp}) / (Ti_{s} - Ti_{Hp})]$$

where Ti_{Hb} is the concentration of Ti in the organic horizons, Ti_{Hp} is the concentration of Ti in the pure organic horizons, and Ti_S is the mean concentration of Ti in the 0–5 cm horizon of soil for each soil. The fraction of each element introduced by soil contamination was deducted to obtain the real content in the organic horizons.
Turpault et al. (2018) observed that the soil contamination of the organic layer was very low, i.e., <5%.

2.4.2 Element concentrations and forms in the inputs

For a selected element, the total input was calculated using Eq. (1):

$$It = Lt + Rt \qquad (1)$$

where It is the total input in kg. ha-1. y-1, Lt is the total litter input in kg. ha-1. y-1 and Rt is the total exploitation residual input in kg. ha-1. y-1.

Lt and Rt are each composed of a soluble (s) and an insoluble (i) fraction. It can be calculated following Eq (2):

$$It = (Li + Ls) + (Ri + Rs) \qquad (2)$$

where Li and Ri are the amounts of insoluble element in Lt and Rt in kg. ha-1. y-1, and Ls and Rs are the amounts of soluble element in Lt and Rt in kg. ha-1. y-1.

It can also be calculated using Eq (3):

$$It = (\alpha L \times Lt + \beta L \times Lt) + (\alpha R \times Rt + \beta R \times Rt) \qquad (3)$$

where αL is the proportion of the insoluble form of an element in the litter, βL is the proportion of the soluble form of an element in the litter, αR is the proportion of the insoluble form of an element in the exploitation residuals, and βR is the proportion of the soluble form of an element in the exploitation residuals

with $\alpha L + \beta L = 1$ et $\alpha R + \beta R = 1$

and $\alpha L \times Lt = Li$; $\beta L \times Lt = Ls$; $\alpha R \times Rt = Ri$ and $\beta R \times LR = LR$

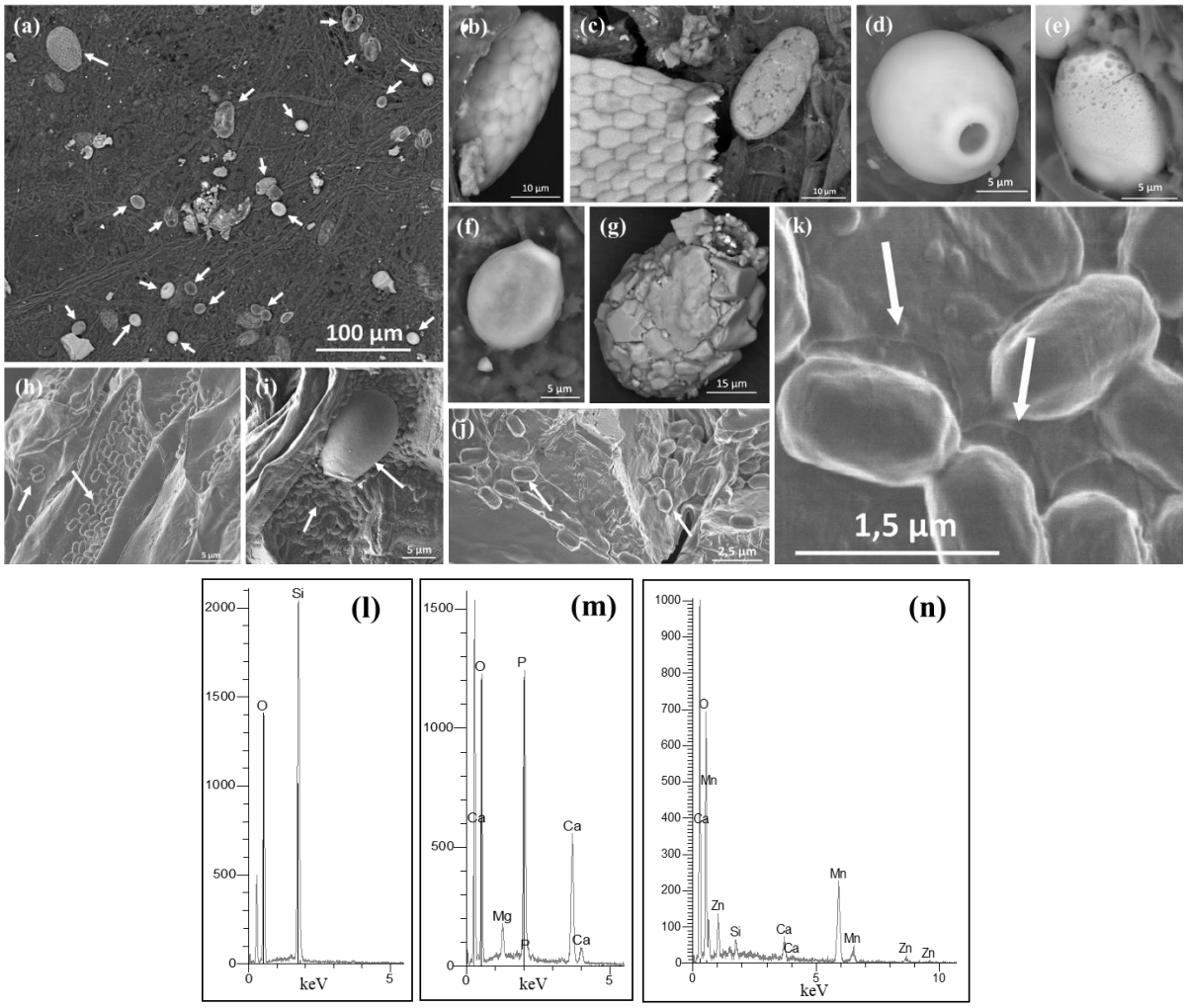

Figure 20 : Testate amoebae and bacteria observed in leaves from a litter of beech forest (a to k). EDX spectra of testate amoebae (l to m). (a) Testate amoebae with different chemical compositions on the leaf blade, (b) Testate amoeba mimicking Trinema complanatum idiosomic taxa (Sommer et al., 2013), (c) Two testate amoebae mimicking (to the left) Trinema lineare xenosomic taxa (Sommer et al., 2013) and (to the right) Euglypha rotunda Idisonic taxa (Sommer et al., 2013), (d) Testate amoeba mimicking Cryptodifflugia oviformis (Ogden and Hedley, 1980), (e) Testate amoebae with a test composed of Mn, (f) testate amoeba mimicking Crypodifflugia oviformis (Ogden and Hedley, 1980) with dissolution traces, (g) testate amoebae composed of exogenous elements (Delaine et al., 2017), (h) vein area with numerous bacteria, (i) bacteria surrounding a testate amoebae mimicking Cryptodifflugia oviformis (Ogden and Hedley, 1980), (j) bacteria on CaOx, (k) bacteria on CaOx, with the arrow indicating a trace of oxalate alteration, (l) EDX spectrum of testate amoeba (b), (m) EDX spectrum of testate amoeba (d), and (n) EDX spectrum of testate amoeba (f).

2.4.3 Exploitation residuals and harvest

As the quantity of the cut biomass depends on the aboveground biomass, these data were integrated into the calculation of exploitation residuals and harvest. Based on the data concerning the proportion of the different tree compartments in the total aboveground biomass at the Montiers site, the biomass of residues (branches <4 and 4-7 cm in diameter) and exports (branches > 7 cm in diameter, stem wood and bark) for this thinning were determined for each soil. Since thinning in the region's forest is generally performed every 7 years, the annual quantities of returned and exported elements were obtained by dividing the total exploitation residue by seven. For more details, see Calvaruso et al., 2017.

2.4.4 Mean residence time

For an element, the mean residence time (RTt) during litter decomposition was calculated using the equation (Eq. (4)) described by Vogt et al. (1986):

$$RTt = stock / I_t \qquad (4)$$

where RTt is the mean residence time in years, and stock is the mass of the element in the humus in 2010 in kg. ha^{-1}. However, to be able to apply this calculation, it will be necessary to verify the following: i) the stability of the stock and inputs, ii) the absence of input/output not related to the decomposition of aerial organic matter such as root biomass or soil input and iii) the absence of root uptake (Derrien and Amelung, 2011). For these reasons, we have chosen to study roots non-colonized mull, not excluding ectomycorrhizal fungi hyphae low uptake.

As the plant tissues of the inputs are composed of soluble and insoluble forms, it is possible to calculate a mean residence time of the 2 insoluble (RTi) and soluble (RTs) forms according to the following equation (Eq. (5)):

$$RTt = \alpha\, RTi + \beta\, RTs \qquad (5)$$

and $$\alpha = I_i/I_t = (\alpha_L \times L_t + \alpha_R \times R_t) / L_t + R_t$$

$$\beta = I_s/I_t = (\beta_L \times L_t + \beta_R \times R_t) / L_t + R_t$$

where α is the proportion of the insoluble form of the element, β is the proportion of the soluble form of the element, I_i the total input of the insoluble form of the element and I_s the total input of the soluble form of the element.

In addition, if RTs = 0, then $RTt = \alpha\, RTi$ or $RTi = RTt/\alpha$

Table 5 : Mean element stocks in the humus of the three 3 soils at Montiers in 2010 and 2018. SD values are indicated in brackets. There was no significant difference between the elements over the 2 years. Values with different letters were significantly different in one soil according to the Kruskal-Wallis test at the threshold P value level of 0.05. A significant difference between soils is indicated by an asterisk.

		Stock (kg. ha^{-1})												
		Dry Mass	C	N	Ca	Si	Fe	Al	K	Mn	S	Mg	P	Na
S1	2010	11139 (1900)^{a}	4940 (847)^{a}	178 (27)^{b}	168 (24)^{b}	148 (37)^{b}	55 (39)^{bc}	19 (5)^{cd}	18 (2)^{cd}	15 (4)^{cd}*	14 (2)^{cd}	13 (1)^{d}	11 (2)^{d}	1.4 (0.2)^{e}
	2018	11952 (1448)	5118 (668)	159 (29)	187 (15)	132 (28)	25 (32)	14 (7)	16 (1)	20 (3)	nd	15 (3)	14 (2)	1.2 (0.3)
S2	2010	9601 (1432)^{a}	4280 (609)^{a}	134 (25)^{b}	160 (24)^{b}	82 (19)^{b}	20 (9)^{bc}	14 (2)^{cd}	16 (1)^{c}	11 (2)^{cde}*	10 (2)^{de}	11 (1)^{cde}	9 (2)^{e}	1.2 (0.1)^{f}*
	2018	9108 (438)	3898 (326)	89 (7)	141 (23)	74 (21)	10 (20)	12 (5)	12 (1)	9 (1)	nd	9 (0)	9 (0)	0.8 (0.3)
S3	2010	8758 (1519)^{a}	3855 (679)^{a}	116 (22)^{b}	195 (36)^{b}	85 (14)^{b}	13 (4)^{cd}	20 (11)^{cd}	16 (3)^{c}	4 (1)^{e}	10 (2)^{d}	11 (2)^{ce}	8 (2)^{de}	1.0 (0.1)^{f}*
	2018	7744 (1710)	3474 (740)	82 (22)	155 (35)	76 (21)	4 (1)	11 (3)	11 (2)	3 (1)	nd	7 (2)	7 (2)	0.8 (0.2)

Table 6 : Mean annual input of dry matter and elements for litterfall and forest exploitation residues year in the three soils. SDs are shown in brackets. Values with different letters are significantly different between soils according to the Kruskal-Wallis test at the threshold P value of 0.05.

		Input kg. ha^{-1}.y^{-1}												
		Dry mass	C	N	Ca	Si	Fe	K	Mg	P	Mn	S	Al	Na
Litterfall	S1	5238 (182)	2452 (85)	52 (2)	63 (6)	45 (2)^{a}	0.9 (0.1)	20 (3)	6.1 (0.8)	5.7 (0.3)	6.8 (0.4)^{a}	4.9 (0.2)	0.8 (0.1)^{a}	0.6 (<0.1)
	S2	5964 (474)	2690 (98)	55 (2)	78 (6)	39 (4)^{b}	1.0 (0.1)	23 (2)	7.1 (0.2)	6.1 (0.1)	6.4 (0.4)^{a}	5.5 (0.3)	0.9 (0.1)^{b}	0.7 (<0.1)
	S3	5663 (37)	2617 (147)	55 (1)	90 (7)	25 (1)^{c}	0.9 (0.1)	23 (3)	7.5 (0.7)	5.4 (0.8)	2.1 (0.3)^{b}	5.9 (0.9)	0.8 (<0.1)^{a}	0.7 (<0.1)
Exploitation residuals	S1	1276	592	3.3	3.6	0.4	0.1	2.3	0.5	0.7	0.2	0.4	0.1	0.1
	S2	1413	652	3.7	4.8	0.4	0.1	2.7	0.5	0.7	0.2	0.4	0.1	0.1
	S3	1117	515	3.2	6.4	0.3	0.1	2.2	0.5	0.6	0.1	0.3	0.1	0.1
Total	S1	6514	3044	55	67	45	1.0	22	6.	6.4	7.0	5.3	0.8	0.7
	S2	7377	3342	59	83	39	1.1	26	7.	6.8	6.7	5.9	1.0	0.8
	S3	6779	3132	58	96	25	1.0	25	8.	6.0	2.2	6.3	0.9	0.8

2.5 Statistical analyses

The normality of the distribution was confirmed using the Shapiro- Wilk test. As our data did not follow a normal distribution, the non-parametrical Kruskal-Wallis test was performed to determine the significance of differences for the element stocks in the humus, the flux of inputs and the different residence times of each element. The comparison was made between elements within the same soil and between soils.

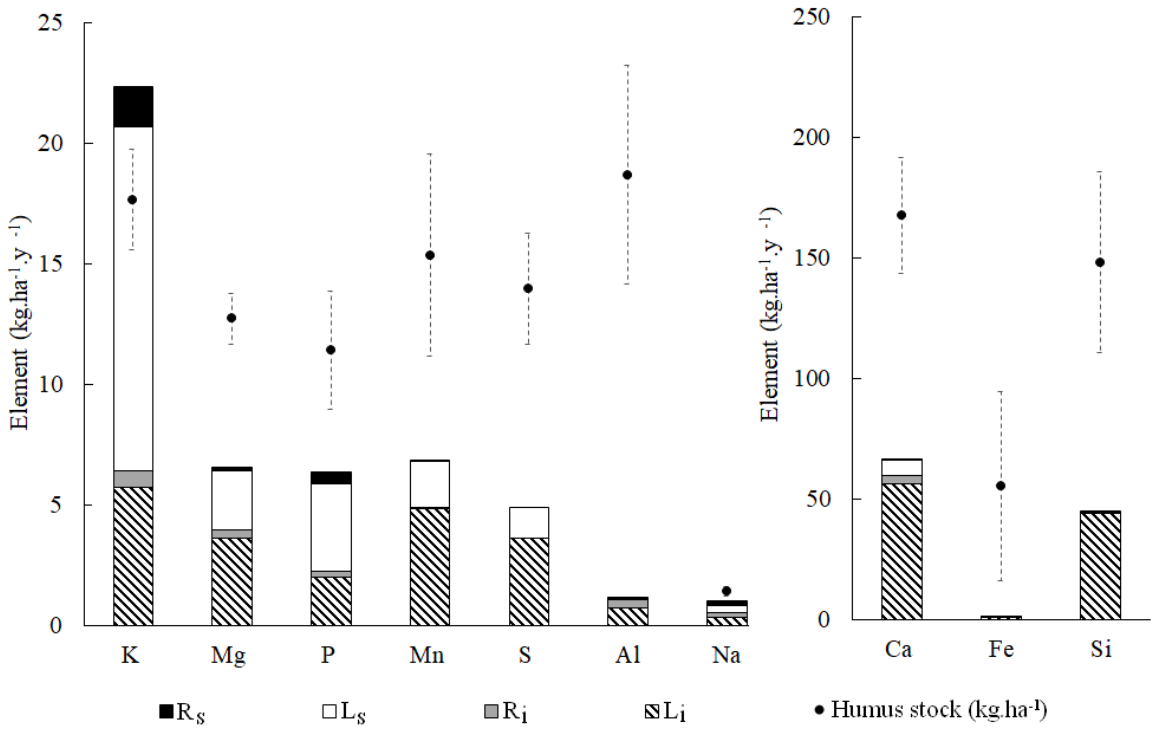

Figure 21 : Annual inputs of element and humus stock in 2010 in kg. ha^{-1}.y^{-1}. Rs: soluble inputs from exploitation residuals. Ls: soluble inputs from litterfall. Ri: insoluble inputs from exploitation residuals. Li: insoluble inputs from litterfall. The humus stock is represented by black dots. SDs of humus stock are represented by dashed black bars.

3 Results

3.1 Observations

3.1.1 Evolution of primary biominerals in fresh leaves and branches

Calcium oxalate (CaOx) crystals were present in the veins of fresh leaves and branch bark (Figure 18 a and b and m). Amorphous Si was found in cell walls of fresh leaves and fresh branch bark (Figure 18 c and d and n), and amorphous Mn was present in the cell walls of fresh leaves (Figure 18 e and o). Aged leaves were degraded, and only veins with weathered crystals remained in some samples (Figure 18 f and g, h, i and m). In the humus, small wood bark was highly degraded with CaOx crystals with a fairly advanced state of deterioration (Figure 18 j and m). In degraded leaves, Si was observed as residual precipitates around cells (Figure 18, k and n) and in intercellular spaces, e.g., stoma cells (Figure 18 l) after partial organic matter degradation.

3.1.2 Secondary precipitation in litter

Large amounts of precipitates of Si on leaf blade were observed. The deposits exhibited two different sizes: one formed large areas (Figure 19 a), and the other small root grains (Figure 19 b). Secondary CaOx formed clusters on the blade (Figure 19 c) and cohabited with a network of hyphae (Figure 19 d). Induced by fungi species, two types of CaOx crystals, i.e., with an elongated shape (Figure 19 e and m) or with an ovoid shape, were present (Figure 19 f and n). Some fungal spores also contained cubic CaOx crystals with K and P (Figure 19 g and o). Amorphous Mn precipitated in the cells of the lower epidermis of the leaf (Figure 19 h) and around some hyphae (Figure 19 i and j, k and p). Other hyphae were surrounded by Mn forming crystalline “shell” (Figure 19 l and q).

Table 7 : The proportion of the insoluble form of an element in litter α_L and the proportion of the insoluble form of an element in the exploitation residual α_R for the different elements. SD are shown in brackets. α = 1 corresponds to an element that is 100% in insoluble form.

		Ca	Si	Fe	K	Mg	P	Mn	S	Al	Na
α_L	S1	0.89 (<0.01)	0.98 (<0.01)	0.95 (<0.01)	0.29 (<0.01)	0.60 (0.01)	0.36 (0.01)	0.72 (<0.01)	0.74 (0.02)	0.98 (<0.01)	0.54 (0.02)
	S2	0.89 (0.01)	0.97 (<0.01)	0.94 (0.02)	0.26 (0.03)	0.57 (0.02)	0.36 (0.02)	0.68 (0.02)	0.74 (0.08)	0.98 (0.01)	0.53 (0.04)
	S3	0.89 (0.03)	0.95 (0.02)	0.95 (0.01)	0.24 (0.03)	0.55 (0.05)	0.38 (0.04)	0.65 (0.05)	0.80 (0.02)	0.99 (<0.01)	0.56 (<0.01)
α_R	S1	0.88	nd	nd	0.29	0.68	0.32	0.73	nd	0.93	0.47
	S2	0.90	nd	nd	0.26	0.65	0.30	0.72	nd	nd	0.35
	S3	0.90	nd	nd	0.28	0.63	0.30	0.71	nd	0.93	0.23

Table 8 : Mean residence time of the total elements (RTt) in the litter of the three soils of Montiers. The standard deviations are shown in brackets. Letters represent significant differences of the residence times between the different elements (p-value 0.05). There is no difference between soils.

	RTt (year)											
	Fe	Al	Si	N	S	Ca	Mn	Mg	Na	P	C	K
S1	58.4 (43.6)^{a}	22.8 (6.6)^{a}	3.3 (1.0)^{bc}	3.2 (0.5)^{b}	2.7 (0.5)^{bc}	2.5 (0.5)^{bc}	2.2 (0.7)^{bc}	2.0 (0.7)^{bc}	2.0 (0.3)^{bc}	1.8 (0.5)^{c}	1.6 (0.3)^{c}	0.8 (>0.1)^{d}
S2	18.1 (8.8)^{a}	14.4 (2.6)^{a}	2.1 (0.3)^{bc}	2.3 (0.3)^{b}	1.7 (0.3)^{bc}	1.9 (0.2)^{bc}	1.7 (0.2)^{bc}	1.7 (0.2)^{c}	1.6 (0.1)^{c}	1.3 (0.1)^{c}	1.3 (0.2)^{c}	0.6 (>0.1)^{d}
S3	13.1 (3.6)^{a}	23.3 (12.5)^{a}	3.3 (0.4)^{b}	2.0 (0.4)^{c}	1.6 (0.5)^{c}	2.0 (0.5)^{c}	2.0 (0.5)^{c}	2.0 (0.5)^{c}	1.2 (0.2)^{d}	1.4 (0.3)^{cd}	1.2 (0.2)^{cd}	0.7 (0.1)^{e}

3.1.3 Testate amoebae and bacteria in litter

Several testate amoebae were found, principally in degraded leaf (Figure 20 a), the large majority of whom had a Si testate similar to *Trinema complanatum* (Figure 20 b and l), *Trinema lineare* and *Euglypha rotunda* (Figure 20 c). In addition, P–Ca (Figure 20 d and e and m) and Mn (Figure 20 f and n) amoebae testate were observed. Sometimes, marks of dissolution were visible on the skeletons of testate amoebae (Figure 20 e). Finally, other species of amoebae had a testate composed of exogenous mineral grain (Figure 20 g). Several groups of bacteria were observed on leaves (Figure 20 h), around testate amoebae (Figure 20 i) and on CaOx (Figure 20 j and k). Dissolution marks under bacteria suggested that the bacteria accelerated the weathering of CaOx crystals.

3.2 Stock and input

3.2.1 Stock 2010, 2018

Between 2010 and 2018, no significant variations of the element stock in the humus were observed regardless of the soil (Table 5). Between the soils, a difference was apparent for Mn (S1 and S2 different from S3) and Na (S2 and S3 different from S1). The stocks were between 3.5 and 5 x 10^3 kg. ha^{-1} for C; 74 and 195 kg. ha^{-1} for Ca, N, and Si; 3 and 55 kg. ha^{-1} for Fe, Mn, K, Mg, S and Al; and 0.8 and 1.4 kg. ha^{-1} for Na.

3.2.2 Inputs in humus: above-ground litter and exploitation residuals

In the Montiers site, no differences were observed among the three soils with respect to the composition of the litterfall, which consisted of 70–75% leaf, 6–9% wood and 19–21% bud, beechnuts and fruit capsules. The main biomass inputs were the litterfall (5.2 $ha^{-1}. y^{-1}$) and the exploitation residue (2.4 $ha^{-1}. y^{-1}$). Table 6 presents the mean element contents of the litter and exploitation residuals for the 3 soils between 2012 and 2015. These inputs in the 3 soils were 0.6–1 kg. $ha^{-1}. y^{-1}$ for Na, Al and Fe, 2.1–7.5 kg. $ha^{-1}. y^{-1}$ for Mn, P and Mg, 25–90 kg. $ha^{-1}. y^{-1}$ for Si, N and Ca, and 2.7 x 10^3 kg. $ha^{-1}. y^{-1}$ for C.

3.2.3 Forms of the inputs

The elements in the inputs could be divided into two forms: soluble and insoluble. By examining the α insoluble fraction of the elements in the litterfall and the exploitation residuals (Table 7), K, P and Na respectively appeared to present α values between 0.24 and 0.56, indicating that these elements were principally found in soluble form. In contrast, the insoluble form was dominant for Si, Fe, Al and Ca with α values between 0.98 and 0.88.

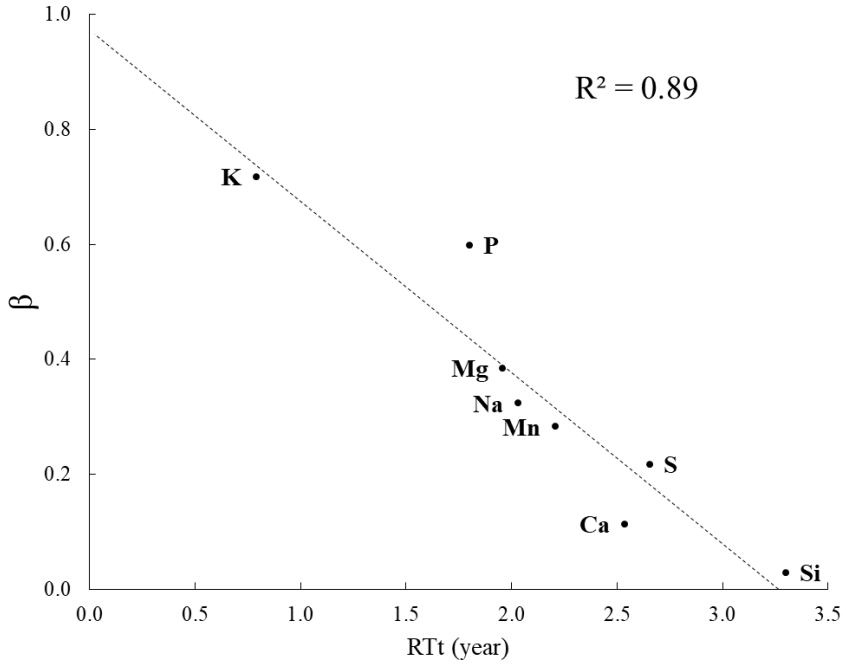

Figure 22 : Comparison between proportion of the soluble form of an element (β) and their mean residence time (RTt) for S1. The dotted line represents the correlation between the two parameters.

Figure 21 shows the elemental contents of incoming litter and exploitation residuals, and their form (soluble and insoluble) in relation to humus stocks. For each element, the input of litter was much larger than the input of exploitation residuals. This difference was derived from the input flux: the biomass quantity and element concentration was higher in the litter. The element stock of the humus was greater than the annual input litter and exploitation residuals for all elements except K. The same variation of elements and stock was observed in the soils.

3.3 Mean residence times

3.3.1 Mean residence time in the humus layer

The previous results showed the steady state of the litter between 2010 and 2018 (stable humus stock and little inputs variability). It was therefore possible to calculate the elements RTt in the humus of the 3 studied soils (Table 8).

The RTt of K was lowest with 0.6–0.8 years. The RTt was between 1.2 and 2.2 years for C, P, Na, Mg and Mn, and between 1.6 and 3.3 years for Ca, S, N and Si. The RTt of Al and Fe was much longer, i.e., between 13 and 58 years, and it varied widely between repetitions (Table 8). Significant uncertainties in Fe and Al RTt are linked to large variations in stocks between replicates. Thus, it is possible to rank elements by their residence times: Fe, Al > Si, N, S, Ca ≥ Mn, Mg, Na, P, C ≥ K.

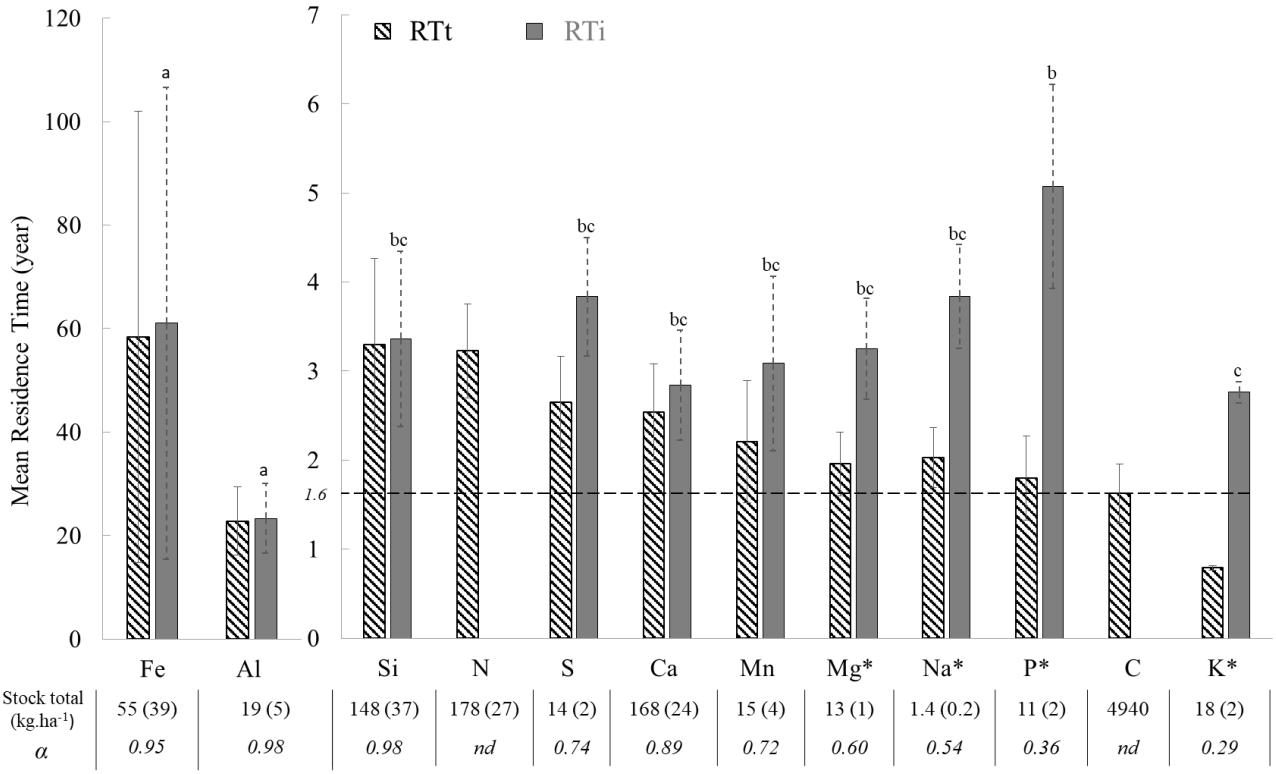

Figure 23 : Comparison between the total residence time of the elements (RTt) and the residence time of the insoluble part of the elements (RTi), as well as the stock total of elements in the humus and the proportion of the insoluble form (α) in soil S1. The standard deviation is indicated for RTt by filled black bars and RTi by black dashed bars. Capital letters correspond to significant differences between RTt, and lowercase letters correspond to significant differences between RTi (p-value < 0.05). The black dashed line corresponds to the value of carbon RTt.

3.3.2 Correlation between form in the inputs and the mean residence time

For each element, there was a strong correlation between the soluble fraction in the inputs and residence time for the 3 soils (data not shown for S2 and S3) (

Figure 22). The larger the soluble fraction, the shorter was the RTt, as observed for K with a soluble fraction exceeding 70% and a RTt of 0.8 years, compared to Si with a soluble form of 5% and RTt of 3.3 years. This correlation was much greater in S1 and S2 (R2 = 0.89 and R2 = 0.90).

3.3.3 Mean residence time of insoluble fraction

Figure 23 presents S1 for the different RTt and RTi of the elements as well as their total stocks in humus and their coefficients of proportion in the insoluble form α (Eq. (5)). For elements other than Fe, Al, C and N, the RTi in S1 was between 2.8 for K and 5.1 for P. The RTi for Si, Ca, Mn, Fe and Al did not differ statistically from their RTt. Similar results were obtained for the other two soils. The classification for RTi for the different soils was as follows: Fe, Al > P S, Na, Si, Mg, Mn, Ca K.

4 Discussion

4.1 Form and localization of elements in inputs and differential tissue degradation

Although, several studies have shown that the solubility of elements directly influences their dynamics in litter (Attiwill, 1968; Gosz et al., 1973; Melin, 1930), few studies have quantified these inputs with studies limited to some species and elements (Aerts and Caluwe, 1997; Chapin and Moilanen, 1991; Ibrahima et al., 1995).

As previously reported, we found that the soluble fraction of inputs was dominant for K and P (Chapin and Moilanen, 1991; Gosz et al., 1973; Schreeg et al., 2013; Sterner and Elser, 2002). For the other elements (Na, N, Ca and C), our results were consistent with the results of Schreeg et al. (2013) in 41 tropical forests in Panama. Our results showed that the insoluble forms of Si.

Our results showed that the insoluble forms of Si and Ca were dominant in the inputs (Figure 21, Table 7). This agrees with SEM observations revealing that these elements were largely immobilized as biominerals, respectively in the form of opal in the walls of cells and CaOx crystals mainly located in leaf veins (Figure 18 a, c, e). It has been noted that Ca is also transferred to humus in the form of organic molecules (e.g pectins) (Jones and Lunt, 1967), also contributing to the humus Ca pool. The same biominerals have previously been highlighted, especially in beech leaves, by Krieger et al. (2017). The abundance of CaOx at this site would come from the richness of the soil in this element. Indeed, on more acidic soils, Ca would be used directly with less stored in the tree's organs.

Similarly, to many authors, we observed a faster alteration of the leaf blade than the veins when the leaves were degraded in litter (Figure 18) (Osono, 2007; Rihani et al., 2001). Leaves are mainly composed of parenchyma and secondary tissues. Parenchymal cells are essentially located in the leaf blade and are composed of a protoplast rich in proteins surrounded by a cellulose "wall" (Sanderman and Amundson, 2003). Leaf veins are composed of more ligneous cells consisting of transport tissues (xylem and phloem) and sometimes support tissues (sclerenchyma) (Campbell et al., 2014), where the proportions of cellulose, hemicellulose and lignin may vary (Berg and McClaugherty, 2014; Sanderman and Amundson, 2003). In general, cells mainly composed of cellulose or hemicellulose are degraded more rapidly than those containing more lignin (Kögel-Knabner, 2002; Leitner et al., 2012). Differences in cell compositional patterns could explain differences in tissue degradation rates (Kleber, 2010). It should be noted that sometimes, veins are preferentially degraded compared to leaf blade. This is the case in the presence of oribatid mites who prefer leaf veins (Ayres et al., 2009).

Consequently, as observed in our study, localized insoluble elements in veins are "protected"/less mobilized, including biominerals, compared to the insoluble element in the blade. In addition, when veins degrade, biominerals appear to be more resistant to dissolution than in the blade. In particular, the organic matter of the veins may be degraded while the opal is still present (Figure 18 k).

The form and localization of the elements in leaves depends on the elements and could influence their release rate when leaves are degraded.

4.2 Residence time of elements in litter

The RTt of C, 1.2 ± 0.2 and 1.6 ± 0.3 years, was slightly lower than the average global value of 2.4-year-old litter on different humus types (Wang et al., 2010). Our average values are close to those obtained for deciduous mulls: 1.72 ± 0.5 years in a mixed deciduous forest in China (Zhang et al., 2010) and 1.9 ± 0.08 years in an oak forest on a mull in Spain (Campo et al., 2014). The latter groups also found a comparable RTt for P (1.8 year ± 0.08) but a lower value for N (1.7 ± 0.11 years) compared with our results (2.0–3.2 years).

Kavvadias et al. (2001), who conducted a similar study in a beech forest (mull humus) developed on shale on the north-eastern slope of the Ossa Mountain in northern Greece, obtained similar RTt values and the same gradient of element RTt as established in our study for N, Mg, C, P, Na, and K but not for Ca. This difference could be due to the acidic substrate (shale) on which the forest grows.

In other types of humus (moder and mor), this classification may differ and the RTt are higher. According to Reiners and Reiners (1970) study on moder or mor humus of three Minnesota forests, the residence times were much higher than those found in our study, e.g in oak forest C had a higher RTt (15 years) than Ca (9 years).

The correlation between the soluble fraction in the inputs and the residence time indicated that the larger the soluble fraction, the shorter was the RTt (Figure 22

Figure 22). This correlation suggests that a fully soluble element (100%) would have a RTt value of 0, corresponding to almost immediate release into that ecosystem. In contrast, the soluble part of an element emerges quickly from the litter, suggesting that RTs were negligible (RTs = 0) and that equation (5) $RTt = \alpha\ RTi + \beta\ RTs$ can be written as $RTi = RTt/\alpha$.

Thus the smaller was α, the larger was RTi, the closer was α to 1, and the closer was RTi to RTt (Figure 23): α could explain the increase or not between RTt and RTi. These increases were more important for K, P, Na, and Mg with increases of 250%, 182%, 89% and 66%, respectively. These elements were also those for which $\beta > \alpha$, i.e., the proportion of the soluble form was greater than that of the insoluble form. Thus, the vision of RTt did not presume what would be observed through RTi. The RTi for Si, Ca, Mn, Fe and Al did

not differ statistically from their RTt, which can be explained by the observation that these elements were mainly in insoluble form and essentially in the form of biominerals.

4.3 Element retention mechanisms in litter

In addition to degradation related to localization in organic molecules (Kleber, 2010), slower degradation of tissues (vein) and dissolution-resistant biominerals, what retention mechanisms in the litter could influence this classification by increasing their residence time?

The SEM observations allowed us to highlight the recycling of 4 elements (Si, Ca, P, Mn) that were strongly recycled and 2 elements (Mg and K) that were less involved in recycling through abiotic and biotic mechanisms. In addition to the deposits of Si, two main types of biotic recycling were observed: i) via testate amoebae and ii) via precipitation around hyphae. Wide amorphous deposits of Si were observed in our study similar to those previous described by Watteau and Villemin (2001) in a litter of beech in the same region (90 km) in a mull-type humus.

Outside the rare testate composed of exogenous mineral grains, numerous testate amoebae with three dominant chemical compositions were highlighted: i) those containing Si, ii), those containing P and Ca and iii) those composed of Mn. These types of amoebae have been previously described by Ogden and Hedley (1980). Sommer et al. (2013) showed that Si testate amoebae can recycle up to 50% of the Si from litter degradation in temperate hardwood ecosystems. It can therefore be assumed that this mechanism immobilizes its elements until the degradation of the testate (Figure 20) and increases the residence times of these elements.

Similar to previous studies, we showed that many crystals of secondary calcium oxalates (pure or accompanied by P, K or Mg) that precipitate around fungal hyphae are linked to oxalic acid production (Cromack et al., 1979; Lapeyrie, 1988). In addition, Mn precipitates surround fungal hyphae (pure or accompanied by Ca) have also been observed on some decaying leaves, as described by Pinzari et al. (2018) in the litter of an oak forest in northern Rome. The location of these precipitates surrounding the hyphae suggests that they contribute to the elements recycling through precipitation in humus.

The recycling of these 6 elements in secondary biominerals and testate amoebae (Si, Ca, P, Mn, Mg, K) observed by SEM corresponded to TRi greater than 2.8 years, significantly exceeding the average residence time of C (1.6 year) in S1 (Figure 23) and other soils. This finding suggests an impact of humus recycling on the RTt of these 6 elements.

However, Fe and Al with high RTt did not seem to be affected by the observable recycling by SEM. This RTt of Fe and Al was derived from very low inputs (0.8–1 kg. ha^{-1}) and proportionally high stocks (4 and 55 kg. ha^{-1}) without the micrometric mineral phase. The inputs of these elements could come from atmospheric dust deposition (Lequy et al.,

2014) or by the rising of soil solutions by capillarity, which would remobilize the elements. In Montiers, the pH values were between 6.4 and 6.7 in humus and 5.6 and 6.8 in soil solution at - 10 cm. Below pH 6, Al is mobile and can migrate. In view of the pH values found in the study site, it is possible that the Al comes from soil solutions.

These findings support an influence of other mechanisms of maintaining Fe and Al in litter. Indeed, it has been shown in soils that relatively stable organic-mineral phases containing Fe and Al can be formed by sorption (Kleber et al., 2015; Rowley et al., 2018). These mechanisms could also occur in litter. These sorption mechanisms could involve other elements such as P, which has the strongest RTi after Fe or Ca, and S, which can bind organic molecules (Likens et al., 2002; Rowley et al., 2018).

Some organic molecules are more difficult to breakdown than others, particularly molecules containing N such as polyphenol protein (Mutabaruka et al., 2007), which represents 25–35% of organic molecules in senescent beech leaves (Berthelin et al., 1994; Zeller et al., 2000). This phenomenon contributes to the increased N relative to C RTt in this type of ecosystem.

5 Conclusion

This study presents for the first time the relationship between the RTt of the elements in mull, the form of the elements in the inputs, especially biominerals, and the recycling mechanisms in the humus.

In the Montiers beech forest ecosystem, the stocks of litter elements and inputs were constant between 2010 and 2018, indicating the ecosystem was in balance which allowed the calculation of the residence times of the major nutrients and beneficial elements (N, P, C, K, Si, S, Ca, Mn, Mg, Fe).

Litter degradation speeds were high regardless of the soil (1.6 years for C) and in accordance with the literature for beech forest with mull. The present findings show, for the first time, the average residence times of all major elements in litter, which were classified as follows: Fe, Al > Si, N, S, Ca Mn, Mg, Na, P, C, and K. There were no differences in RTt among the different soils. This highlights the balance in the biological cycle in the 3 sites, allowing for similar recycling efficiency.

The form and localization of the elements in the inputs determined their release speed. The proportion of the element in soluble form directly influenced its RTt. The soluble forms of the elements seemed to be released almost instantly, while the insoluble forms in biominerals and in the more resistant organic matter tissues such as veins demonstrated higher residence times.

Abiotic and biotic reactions in litter allow the recycling of elements. The SEM observations revealed strong recycling via the skeletons of different amoebae (Si, Ca, P, Mn) through the precipitation of crystals around the fungal hyphae (Ca in CaOx accompanied by K, P or Mg and Mn) and via Si deposits.

The rate of release of an element in litter was therefore positively influenced by the fraction found in the soluble form. In addition to surface reactions (sorption) and organic molecules containing N, this rate was negatively influenced by the presence of the element in the form of biominerals (opal, CaOx, Mn) or in the form of tissues (vein) that were not easily degradable by biotic recycling (testate amoebae - Si, Ca, P, Mn and precipitation by fungal hyphae activity - CaOx, Ca, Mn, P, K, Mg) and by abiotic precipitation (Si).

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IV.

Chapitre IV -

Particles in humus leaching solution influence the input-output budget of the major elements in a beech forest

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Ce chapitre est une étude présentant les différents flux entrants et sortants de l'humus (solutions et solides). Le but est d'identifier l'ensemble des flux d'éléments de l'humus à travers l'étude des bilans des flux en entrées et en sorties de l'humus. Ce chapitre répond à la question n°2 : Tous les flux entrants et sortants de l'humus sont-ils identifiés et qu'elle est l'importance de chacun des flux ?

Résumé

Comme les écosystèmes forestiers se développent généralement sur des sols pauvres chimiquement et non fertilisés, les cycles des nutriments et les bilans des entrées/sorties, notamment dans le compartiment de l'humus, sont essentiels pour la pérennité des écosystèmes forestiers.

Les objectifs de cette étude sont d'établir des bilans entrées/sorties pour C, N, P, K, Na, Ca, Mg, Mn, S, Si Al et Fe en tenant compte notamment des entrées de poussières atmosphériques (>0.45µm) et, pour la première fois, des sorties de particules. Pour atteindre ces objectifs, les entrées (poussières, litière et solutions) et les sorties (solution et particules) des éléments ont été quantifiées dans des mulls de deux sols contrastés sous une même forêt de hêtres suivie pendant 7 ans.

Les concentrations des éléments dans les sorties particulaires sont 3 à 16 fois supérieures aux entrées particulaires par les poussières, indiquant une production de particules dans l'humus. Les bilans sont équilibrés pour K, Na et S dans les deux sols et pour P et Mg dans DC et pour Si dans RL. À l'exception de Si, ces éléments sont principalement présents sous forme soluble dans les végétaux et sont transportés via la solution à la sortie de l'humus. Pour Si, l'intégration des particules en sortie de l'humus a permis d'équilibrer son bilan entrées/sorties. Pour Ca et Mn dans les deux sols, pour P et Mg dans RL et pour Si dans un DC les entrées sont supérieures aux sorties. Pour ces éléments retrouvés dans des biominéraux dans les végétaux et dans des biominéraux secondaires précipités dans l'humus, ce bilan déséquilibré indique que d'autres sorties n'ont pas été identifiées dans ce bilan. Ces sorties pourraient être la sédimentation de biominéraux ou le prélèvement par les hyphes.

Cependant, même si le bilan de certains éléments n'est pas équilibré, l'identification du flux de particules s'est révélée essentielle comme flux de sortie pour des éléments comme Si, Al et Fe.

Particles in humus leaching solution influence the input-output budget of the major elements in a beech forest

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1 Introduction

Forest ecosystems are generally unfertilized and grow in soils that are poor in one or more nutrients. Indeed, forests can be present on acidic soils with a low capacity to provide elements by mineral weathering (Sverdrup and Warfvinge, 1988) or on calcareous soils that may have P and K deficiencies (Sardans and Peñuelas, 2015; Zederer and Talkner, 2018). In response to these unfavorable soil conditions, both perennial plants and trees have developed several strategies. Examples include symbiotic associations with microorganisms (bacteria and fungi) to improve nutrient acquisition (Rambelli, 1973; van Breemen et al., 2000), nutrient conservation in tree with the nutrient resorption from leaves during senescence period (Chapin, 1980) and intensive recycling of elements through litterfall notably (Attiwill and Adams, 1993; Berg and McClaugherty, 2014).

The litter is degraded into humus, and the elements are again available to meet the tree's different nutritional needs, thus showing the importance of the life cycle (Jonard et al., 2009).

Although the identification of all the incoming and outgoing fluxes in the humus layer and their quantification is key to understanding the role of recycling in tree nutrition, these types of studies are generally limited to certain incoming fluxes, such as litterfall and solutions (rainfall and stemflow), or outgoing fluxes, such as gaseous emissions, which only concern certain elements, such as C or N, or solutions from the humus.

In forest ecosystems, plant inputs are most often quantified by considering only litterfall (Kavvadias et al., 2001; Ukonmaanaho et al., 2008). More rarely, inputs in the form of exploitation residuals are included, although their contribution is not negligible (de Dieu Nzila et al., 2002; Laclau et al., 2010). Atmospheric particle inputs (>0.45µm) are also rarely quantified, although these inputs can represent up to 30% of the element inputs into ecosystems (Lequy et al., 2012).

If the element stocks in the humus are stable and in the absence of root uptake within its volume, the humus compartment is in a stationary state; that is, the inputs should be equal to the outputs. Thus, in this condition, and apart from the elements with gaseous cycles, the outputs of elements from the humus layer should correspond to the dissolved and particulate elements found in the outgoing solution from the humus compartment.

While soil solutions are quite frequently studied (Likens et al., 2002; Liu et al., 2003; Manderscheid et al., 1995), chemical data from humus and its fluxes are more rarely mentioned. Indeed, despite the importance of humus in recycling elements for tree nutrition, humus has rarely been specifically studied as regards to fluxes and elemental

balance and most often on C, N and P (Spohn and Sierra, 2018). To specify this recycling it is necessary to identify all the incoming and outgoing fluxes of all the elements and using the input/output balances approach to estimate the possible undetermined fluxes.

Similarly, although particulate fluxes are mentioned, particularly for C and N (Versini et al., 2014), particulate transfers between humus and soil have never been studied. Studies of silica and phytoliths under very specific conditions (special cases in watershed studies) provide the closest examinations of particles (Cary et al., 2005; Cornelis et al., 2011). As a result, a comparison between all these inputs and outputs in solid and dissolved forms has never been undertaken for the main nutrients and beneficial elements.

However, comparisons between the input and output fluxes of an ecosystem that take into account the inputs and outputs of humus, do exist and generally show inputs greater than outputs. In studies by (Berger et al., 2009; Spohn and Sierra, 2018), the input fluxes include all the solution fluxes (stemflow and rainfall) and solid fluxes of dust and litter. For the outputs, only the flux of the outgoing solution is taken into account, and the budget of the elements in the humus layer is not balanced.

In this article, we hypothesize that the particle flux in the humus layer represents an important output of elements, making it possible to balance the inputs/outputs of the elements in the humus layer. Our ambitions are (1) to quantify the plant, dust and dissolved ion inputs and the outputs for the main elements (K, Na, S, Mg, P, Ca, Si, N, Mn, Al, and Fe) in a beech forest developed on two contrasting soil types, (2) to determine, for the first time, the transfer of different elements in particulate form for two soil types and (3) to compare the inputs and outputs and determine whether the particle flux allows the element budget to be balanced.

To avoid the colonization of the humus layer by roots and to limit soil pollution, we chose to conduct this study in mulls. According to (Dincher et al., 2020a), the biomass and element contents of these humus were demonstrated to be stable for 8 years (between 2010 and 2018), and the residence times were short for most elements (for example, 1.6 years for C).

2 Materials and methods

2.1 Experimental site

The study was performed at the ecosystem research site in Montiers, located in northeastern France, in the Meuse department (48° 31′ 54″ N, 5°16′ 08″ E). This experimental site was built in 2011 and co-managed since 2012 by the INRA-BEF (French National Institute for Agricultural Research-Biogeochemical Cycles in Forest Ecosystems research unit) and ANDRA (French National Radioactive Waste Management Agency). The Montiers site is part of the AnaEE research network (https://www.anaee.com/).

The climate is semicontinental, with a mean annual rainfall of 1069 mm and a mean temperature over the last 20 years of 9.8°C (in Météo France). In the winter, snow is possible, and the soil can freeze a few centimeters deep. The geology of the Montiers site is composed of two overlapping soil parent materials: an underlying Tithonian limestone surmounted by acidic Valanginian detrital sediments. In these formations, two types of soil are formed: Rendzic Leptosol (RL) and Eutric Cambisol (EC), which have a eutrophic mull humus, and Dystric Cambisol (DC), which has an acidic mull humus (FAO, 2016) varying from 1 to 3 cm thick and very close to the dense root system colonizing the surface horizon of the soil. The description of these soils and the location of the site are presented in (Kirchen et al., 2017). No roots were found in the humus of the 3 soils. The abundance of earthworms observed in the humus also highlights the high biological activity of the humus in the two soils, especially in the RL.

The Montiers site is covered with a beech forest of the same age (~50 years old in 2010) that has undergone an uniform management regime. The stand is mainly composed of beech (89%) and other species (11%), i.e., sycamore maple (*Acer pseudoplatanus*), ash (*Fraxinus excelsior*), pedunculate oak (*Quercus robur L.*), European hornbeam (*Carpinus betulus L.*) and wild cherry (*Prunus avium*). The Montiers forest has existed since at least 1830, and management carried out by the ONF (French National Forest Office) dates back to the 1960s, with thinning every 7 years.

Three experimental sites were established on the 3 different soils and replicated 3 times each. All the replicates (2500 m^2) were equipped with the same monitoring device for collecting the aboveground and belowground solutions at different depths, soil at different depths, organic horizons (3x8 lysimetric collectors per replicate), throughfall (4 gutters per replicate), tree compartments (stemflow and throughfall, 6 collectors per replicate) and the standing aboveground (6 litter traps of 0.34 m^2 per replicate) and belowground biomass. To collect precipitation and atmospheric deposition, a 45 m high flux tower was installed between the 3 sites with 3 collector replicates. A schematic

representation of the different fluxes collected is presented in the article from Turpault et al., 2019. In this study, we used data collected on DC an RL. For more information, see Calvaruso et al., 2017, Turpault et al., 2018 and Kirchen et al., 2017.

2.2 Sampling

2.2.1 Litterfall sampling

The sampling method for litterfall was described in Dincher et al. (2020a). Briefly, litterfall was collected throughout the year from 2012 to 2018 using 6 litter traps of 0.34 m^2 per replicate. The litterfall was separated according to three fractions: leaves, branches, and buds, beechnuts and fruit capsules.

2.2.2 Exploitation residuals

Exploitation residuals correspond to the branches with a diameter below 7 cm. The other compartments, i.e. branches with a diameter above 7 cm and stem wood and bark are exported during the stand thinning.

Sixteen beech trees were harvested in the two plots of the Montiers experimental site (8 for DC and 8 for RL) in 2009 to collect stem wood and bark and branches according to three diameter classes : >7 cm, between 4 and 7 cm and < 4 cm. As described in Calvaruso et al. (2017), this sampling allowed to define allometric equations linking tree stem diameter and biomasses for the different aboveground compartments, as well as the element content in the different compartments.

2.2.3 Solutions above and below the humus

All the solutions were collected every 28 days from 2012 to 2018 in the two experimental plots. Rainfall was sampled on top of the flux tower in three polyethylene collectors (0.24 m^2) from 2012 to 2018 to obtain bulk deposition (<0.45 µm).

The humus input solutions corresponded to throughfall and stemflow. For each plot, the throughfall was sampled with 12 gutters placed 1.2 m above the forest floor and covering a total of 1.56 m^2. For the stemflow sampling, six trees of different sizes were equipped with flexible polyethylene collars attached horizontally to the stem at 1.50 m height and connected to polyethylene storage containers (120, 150 or 310 L). In the winter, the stemflow was collected from 6 trees in each plot and drained into underground storage containers to avoid freezing. For more information, see Kirchen et al. (2017).

The humus solution was collected by 3x8 zero-tension lysimeter plates, with replicates covering an area of 0.5 m^2 placed under the humus at the boundary with the upper soil horizon. The lysimeter plates were connected to an underground storage

container and collected every 4 weeks between 2012 and 2018. These containers are located in a pit excavated in the soil and were thus protected from light and extreme temperatures. Tests showed the stability of the elements in the solutions over the period between sampling.

2.2.4 Dust and humus particles

All of the atmospheric particles or dust (>0.45 µm) were collected in the solutions beginning in 2012; the humus particles were collected only in 2017 and 2018. The procedure for particle sampling is described in (Lequy et al., 2014). Briefly, the rainfall and humus output solutions were centrifuged for 40 min at 3500 tr.min-1 to separate the solid phase (particles) from the solutions.

In this study, dusts were collected in the rainfall solution to avoid element enrichment after the solution passes though the canopy (Lequy et al., 2014), which could lead to an overestimation of the element inputs into the humus.

2.3 Analytical methods

2.3.1 Microscopic analysis

Samples of the atmospheric and humus particles were selected randomly from each replicate for microscopic examination in 2018. Six of the 26 samples of the humus particles and six of the 91 samples of atmospheric particles were selected for analysis and placed on carbon slides. After carbon metallization, the samples were analyzed by scanning electron microscopy (SEM) with a Hitachi S-48000 equipped with an energy-dispersive system (EDS) at the SCMEM laboratory (University of Lorraine). The SEM analyses were carried out using an acceleration voltage of 3 or 15 kV with a working distance of 15 mm. The obtained images consisted of backscattered electrons.

2.3.2 Elements in litterfall and exploitation residuals

The total Ca, K, Mg, P, Al, Mn, Na, S, Si and Fe contents in the solid samples, such as litterfall and exploitation residuals, were analyzed by X-ray fluorescence sequential spectrometer S8 TIGER 1 kW (Bruker) with uncertainty < 12%. The samples were first dried in a stream air-drier (65°C) and then ground and encapsulated for analysis. For the exploitation residuals, the values in this study are those used in (Dincher et al., 2020a). The total C and N were determined using a CHN analyser (EA/NA 1110, Thermo Quest).

2.3.3 Elements in the solutions

All the solutions were directly filtered at 0.45 µm and analyzed upon return to the laboratory. The throughfall, stemflow and humus solutions were filtered at 0.45 µm,

stored at 4°C and analyzed in the days following sampling. The element contents in the solutions were measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES Agilent Technologies 700 type ICP-OES, Santa Clara, USA). The total C and N contents of the samples were determined by a Thermo Quest NCS 2500 analyser.

2.3.4 Dust and humus particles

The element contents in the particles were oven-dried at 35°C and ground. The atmospheric particles were pooled by year and then analyzed by the CNRS laboratory using inductively coupled plasma optical emission spectrometry (ICP-OES) for the major elements and inductively coupled plasma mass spectrometry (ICP-MS) for the trace elements (Carignan et al., 2001).

2.4 Calculation of the elements flux and input/output budget

2.4.1 Exploitation residuals calculation

A simulation based on National Forestry expertness (selection of trees to cut) and the use of allometric equations revealed that the exploitation residual amounts were about 8.9 x 103 and 7.8 x 103 kg.ha-1 for DC and RL, respectively. As at this stage of stand development, the French National Forestry Office carries out thinning every 7 years, the annual amount of exploitation residuals were about 1.3 x 103 and 1.2 x 103 kg.ha-1 for DC and RL (total exploitation residual amounts equally distributed over 7 years). The annual amount of elements in exploitation residuals were calculated by multiplying the annual amount of residuals by the concentrations of elements in this compartment.

2.4.2 Element flux

In each soil, the monthly element fluxes were obtained by multiplying the monthly flux of solution or solids by the concentration of the elements in a given compartment. Every month, these element fluxes were calculated per hectare and then summed over calendar years to calculate the annual fluxes. Straight mean annual values of elements fluxes or concentrations are also provided in Tables.

A model based on the circumference of stems and developed by Kirchen et al. (2017) was used to obtain the stemflow flux.

The BILJOU© model (Granier et al., 1999) was applied in the two plots at the Montiers site to evaluate the water drainage flux in the humus layer. This model was validated by various measured data, including soil moisture, and allowed the simulation of water fluxes at daily time steps in the different compartments of a forest ecosystem (Granier et al., 1999). The model used a combination of parameters and daily weather

data. All the information on the data and the procedure are available in Kirchen et al. (2017).

2.4.3 Input/output budget

For a selected element, the total input was calculated using Eq. (1):

$$In = L + R + SF + TF \quad (1)$$

where In is the total input in $kg.ha^{-1}.y^{-1}$, L is the litter input in $kg.ha^{-1}.y^{-1}$, R is the exploitation residuals input in $kg.ha^{-1}.y^{-1}$, SF is the stemflow in $kg.ha^{-1}.y^{-1}$ and TF is the throughfall in $kg.ha^{-1}.y^{-1}$.

For a selected element, the total output was calculated using Eq. (2):

$$Out = S_{ol} + P_{art} \quad (2)$$

where Out is the total output in $kg.ha^{-1}.y^{-1}$, S_{ol} is the humus solution in $kg.ha^{-1}.y^{-1}$ and P_{art} is the outgoing humus particles in $kg.ha^{-1}.y^{-1}$.

If In = Out, the budget is balanced.

2.5 Statistical analyses

The normality of the distribution was confirmed using a Shapiro-Wilk test. As our data did not follow a normal distribution, a nonparametric Kruskal-Wallis test was performed to determine the significance of the differences between the elements in the inputs and outputs. The comparisons were made between elements and years within the same soil.

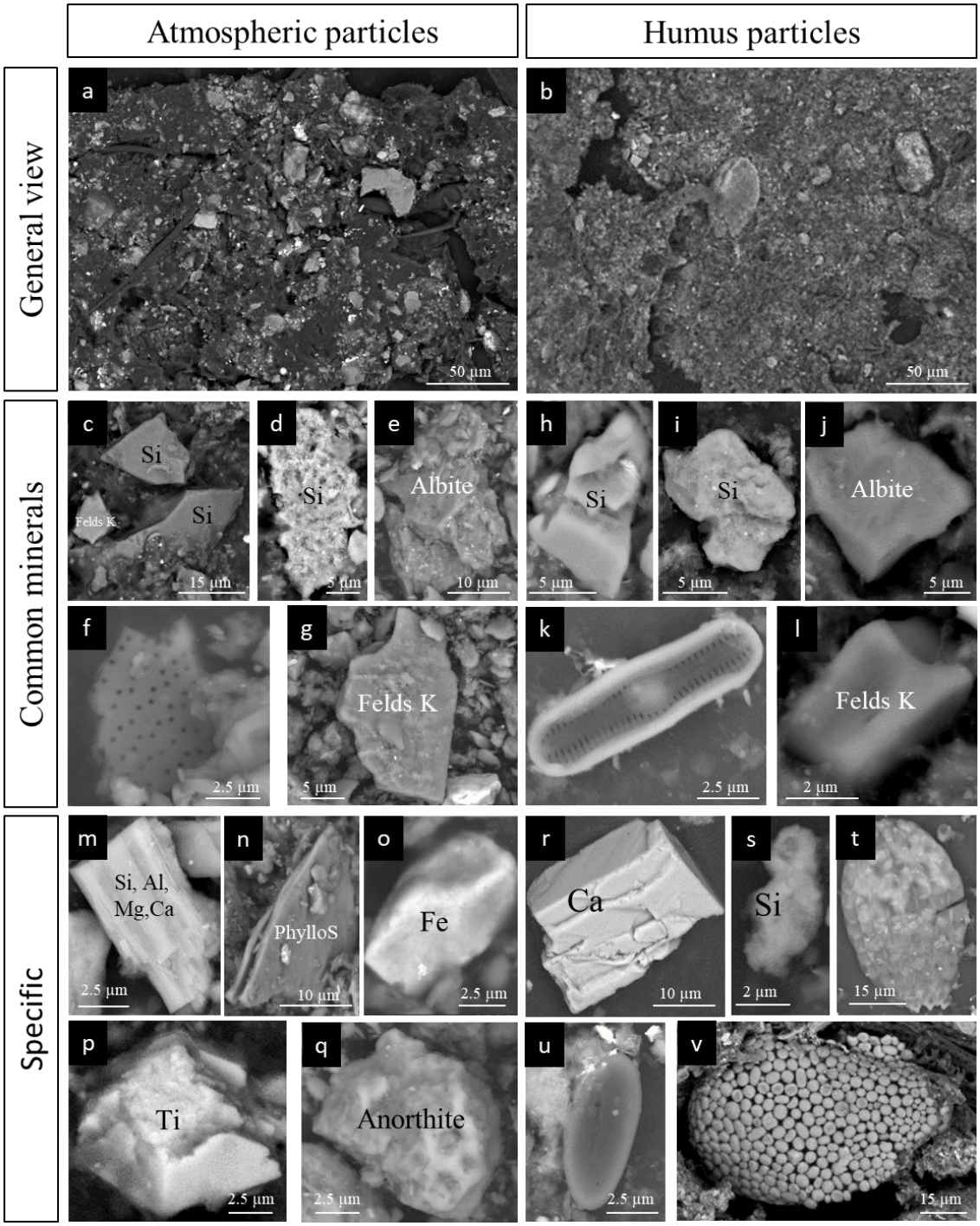

Figure 24 : SEM photographs of samples of the atmospheric and humus particles: a, c-g, and m-q correspond to the atmospheric particles and b, h-l, and r-v correspond to the humus particles. (a) General view of the atmospheric particle sample; (b) General view of the humus particle sample; (c) Two Si minerals and one potassium feldspar with an angular shape; (d) Another Si mineral with dissolution marks; (e) Albite with an angular shape; (f) Piece of a diatom; (g) Potassium feldspar (Felds K) with an angular shape; (h) Si mineral with a rounded shape; (i) Si mineral with a rounded shape and dissolution marks; (j) Albite with a rounded form; (k) Whole diatom skeleton; (l) Potassium feldspar (Felds K) with a rounded shape; (m) Fibrous mineral composed of Si, Al, Mg and Ca; (n) Phyllosilicate (PhylloS) composed of Si, Al, Mg, Fe, and Ti; (o) Iron oxide; (p) Mineral composed only of Ti; (q) Anorthite with dissolution marks; (r) Crystal of calcium oxalate; (s) Si mineral that appears to have an amorphous shape; (t) Very altered Si testate amoeba; (u) Altered organic amoeba; and (v) Altered multielement testate amoeba.

3 Results

3.1 Particles in the inputs and outputs

3.1.1 SEM observations

Observations of the atmospheric and humus particles showed similarities but also specificities (Figure 24). Photos a and b in Figure 24 present general views of these particles. These images revealed a difference in the size of the particle constituents: atmospheric particles are larger than humus particles.

However, in these two particle types, the same minerals were found, i.e., pure Si minerals (c, d, h, and i), albites (e and j), potassium feldspars (g and l) and biological skeletons, such as diatoms (f and k). Although these elements were found in both types of particles, their morphologies were different. While quartz was most often angular in the atmospheric particles (c), it was more rounded in the humus particles (h and i), showing partial dissolution of this mineral. The same observation could be made for albites (e and j) and potassic feldspar (g and l). In the dust, diatoms were more often fragmented (f) than those in the humus particles (k).

In addition to these common elements, each sample had specific mineral grains. The atmospheric particles contained fibrous minerals, including Si, Al, Mg and Ca (m); phyllosilicates (n); iron oxides (o); and minerals composed of Si and Ti (p), and anorthite (q).

In the humus particles, calcium oxalates were found (r) as well as pure Si minerals with an amorphous structure (s). In these particles, several types of testate amoebae (Si, multielement and organic) exhibiting significant traces of alterations (t, u and v) were also observed.

3.1.2 Chemincal composition of the particles

The mean annual concentrations of the elements in the incoming (atmospheric) and outgoing (humus) particles in solution from the humus layer are shown in Table 9. The concentrations of C, Na, Mg, P, Si, N and Al did not significantly differ between the input and output particles. The Ca and Mn concentrations were three times higher in the output particles. These concentration were 4.3 and 0.3 $g.kg^{-1}$ in atmospheric particles, 11.7 and 2.0 $g.kg^{-1}$ in DC and 13.8 and 0.8 $g.kg^{-1}$ in RL humus particles, respectively. The K and Fe were two times more concentrated in the output than in the input. In input, concentrations ranging from 7.5 and 17.1 $g.kg^{-1}$ respectively. In output, concentrations were 14.8 and 35.6 $g.kg^{-1}$ in the DC and 13.9 and 32.2 $g.kg^{-1}$ in the RL, respectively.

Table 9 : Mean annual concentration of the elements in the atmosphere (n=3, 2012-2018) and the output humus particles (n=3, 2017-2018) in g.kg-1. The italic numbers correspond to the standard deviations. The stars correspond to the significantly different concentrations between the humus particles and atmospheric particles (p-value < 0.5) in the two soils.

		g.kg^{-1}																	
		C		K*		Na		Mg		P		Ca*		Si		N		Mn*		Al		Fe*	
Atmospheric particles		255	*65*	7.5	*1.0*	3.5	*1.7*	4.5	*1.2*	3.9	*1.4*	4.3	*1.2*	136	*28*	29.3	*8.8*	0.3	0.1	30.8	*7.5*	17.1	*3.9*
Humus particles	DC	281	*47*	14.8	*5.1*	2.3	*0.5*	3.3	*0.3*	4.5	*0.3*	11.7	*1.9*	116	*6*	22.7	*6.5*	2.0	0.3	29.3	*6.2*	35.6	*8.6*
	RL	262	*50*	13.9	*2.9*	1.4	*0.5*	4.7	*0.4*	4.3	*0.5*	13.8	*2.6*	113	*18*	23.3	*5.8*	0.8	0.2	48.6	*7.3*	32.2	*5.1*

Table 10 : Incoming and outgoing solution fluxes of the humus in kg.ha-1.y-1. The total solution input is the sum of the throughfall (TF) and the stemflow (SF). The solution budget corresponds to outputs minus the inputs. The italic numbers correspond to the standard deviations. The asterisk shows a significant difference (p-value<0.05) between the inputs and outputs.

		kg. ha^{-1}. y^{-1}																					
		C		**K**		**Na**		**S**		**Mg**		**P**		**Ca**		**Si**		**N**		**Mn**		**Al**		**Fe**	
Total solution inputs	DC	53	*13*	17.7	*6.27*	4.87	*0.66*	2.99	*0.48*	1.85	*0.50*	1.92	*0.74*	7.1	*1.10*	1.42	*0.61*	8.3	*1.4*	0.22	*0.07*	0.08	*0.02*	0.34	*0.18*
	RL	56	*10*	20.2	*5.17*	5.24	*0.61*	2.93	*0.61*	1.77	*0.37*	1.67	*0.53*	7.4	*0.95*	1.23	*0.46*	7.9	*1.2*	0.10	*0.03*	0.08	*0.02*	0.35	*0.18*
Solution output	DC	210	*43*	40.3	*8.99*	6.42	*1.04*	4.69	*0.86*	4.87	*0.74*	4.62	*1.15*	28.2	*4.52*	15.1	*3.37*	12.0	*2.3*	0.08	*0.02*	1.12	*0.26*	1.96	*0.68*
	RL	177	*24*	35.2	*6.42*	5.94	*0.87*	9.73	*2.19*	4.71	*0.55*	2.75	*0.73*	48.6	*4.90*	10.6	*1.07*	11.4	*2.0*	0.05	*0.01*	1.77	*0.71*	2.05	*0.93*
Solution budget	DC	157		22.6	*	1.55	*	1.70	*	3.02		2.70	*	21.1	*	13.6	*	3.65	*	-0.14		1.04	*	1.62	*
	RL	121	*	14.9	*	0.70		6.80		2.94	*	1.08	*	41.2	*	9.35	*	3.41		-0.05	*	1.69	*	1.70	*

3.2 Solution flux

3.2.1 Solutions inputs

From 2012 to 2018, the elements in the solutions entering the humus layer, i.e., rainfall (TF) and stemflow (ST), were analyzed (Table 10). In the soils, the inputs were similar and did not vary significantly among years.

In terms of total inputs, the largest mass input was associated with C, with values between 56 and 69 kg $ha^{-1}.y^{-1}$. The fluxes of incoming K were also large, between 17.7 and 22.5 $kg.ha^{-1}.y^{-1}$. The N, Na and Ca fluxes varied between 4.9 and 8.0 $kg.ha^{-1}.y^{-1}$. S, P, Mg and Si had fluxes between 1.2 and 3.1 $kg.ha^{-1}.y^{-1}$. Finally, Fe, Mg and Al had smaller fluxes, between 0.08 and 0.37 $kg.ha^{-1}.y^{-1}$.

3.2.2 Solutions output

The elements contained in the solution leaving the humus layer were analyzed (Table 10). A large amount of C was released by the solution (between 177 and 210 $kg.ha^{-1}.y^{-1}$). For K, Ca, Si and N, the quantities were between 8.8 and 48.6 $kg.ha^{-1}.y^{-1}$. The output fluxes of Mg, Na, P, and S ranged from 2.8 to 9.7 $kg.ha^{-1}.y^{-1}$. The flux of Mn was very low, with values between 0.05 and 0.19 $kg.ha^{-1}.y^{-1}$.

3.2.3 Solution budget

By comparing the input and output fluxes of the elements in solution in the humus, we obtained the budget of the elements in the humus at the solution level (Table 10). The budget was balanced for C, Mg and Mn in the DC and for Na, S and N in the RL. The budget was negative for Mn in the RL with inputs greater than outputs. The budget was positive for the other cases with outputs greater than inputs. For each element, a positive budget indicates that the humus layer is a net source, a negative balance indicates that the humus layer is a net sink.

3.3 Solid flux

3.3.1 Solid inputs

3.3.1.1 Dust

Between 2012 and 2018, the input of elements from dust was relatively large for C and Si, with fluxes exceeding 5 $kg.ha^{-1}.y^{-1}$ (Table 11). This flux was also approximately one kilogram per hectare for Al, N and Fe. For K, Na, P, Ca and Mg, the dust inputs were less than one kilogram per hectare, and those of Mn were very low, at 0.02 $kg.ha^{-1}.y^{-1}$.

Table 11 : Mean annual incoming and outgoing solid flux for the humus layer in kg.ha^{-1}.y^{-1} between 2012 and 2018. The total solid input is the sum of the inputs by dust, litterfall and exploitation residuals. The solid budget corresponds to outputs minus the inputs. The asterisk shows a significant difference (p-value<0.05) between the inputs and outputs.

		kg. ha^{-1}.y^{-1}											
		C	**K**	**Na**	**S**	**Mg**	**P**	**Ca**	**Si**	**N**	**Mn**	**Al**	**Fe**
Input dust		9.7 *0.68*	0.54 *0.08*	0.18 *0.04*		0.25 *0.06*	0.14 *0.02*	0.27 *0.06*	5.6 *1.2*	1.1 *0.1*	0.02 *0.01*	1.5 *0.3*	1.0 *0.2*
Input Litterfall	DC	2867 *7*	24.6 *2.9*	1.3 *0.0*	5.2 *0.1*	7.0 *0.8*	6.3 *0.2*	75 *5*	44.2 *2.2*	54.6 *0.9*	7.3 *0.3*	1.1 *0.08*	1.1 *0.12*
	RL	2924 *209*	23.7 *1.6*	1.3 *0.1*	6.4 *0.8*	8.1 *0.4*	5.8 *0.5*	109 *5*	22.7 *1.4*	58.2 *4.8*	2.1 *0.4*	1.2 *0.02*	1.1 *0.09*
Input residual exploitation	DC	592	2.3	0.1	0.4	0.5	0.7	3.6	0.4	3.3	0.2	0.1	0.1
	RL	515	2.2	0.1	0.3	0.5	0.6	6.4	0.3	3.2	0.1	0.1	0.1
Total solid input	DC	3469 *947*	27.5 *13.9*	1.6 *0.2*	5.5 *1.1*	7.8 *2.3*	7.2 *2.2*	79 *10*	50.2 *5.0*	59.0 *10.4*	7.6 *1.0*	2.7 *0.3*	2.2 *0.2*
	RL	3450 *693*	26.5 *9.5*	1.6 *0.2*	6.7 *1.2*	8.9 *1.7*	6.5 *1.7*	115 *14*	28.6 *4.1*	62.5 *16.2*	2.2 *0.3*	2.7 *0.3*	2.2 *0.2*
Particles output	DC	50 *15*	2.9 *1.8*	0.45 *0.21*	0.58 *0.19*	0.63 *0.30*	0.84 *0.32*	2.2 *0.8*	22 *10*	3.9 *0.8*	0.36 *0.09*	5.8 *3.7*	7.2 *4.7*
	RL	54 *13*	3.2 *1.4*	0.32 *0.12*	0.79 *0.22*	1.06 *0.41*	0.95 *0.35*	3.1 *1.3*	26 *12*	4.7 *1.1*	0.18 *0.05*	11.2 *5.1*	7.4 *3.5*
Solid Budget	DC	-3419 *	-24.6 *	-1.1 *	-4.9 *	-7.2 *	-6.3 *	-76.9 *	-28.0 *	-55 *	-7.2 *	3.2 *	4.9 *
	RL	-3396 *	-23.3 *	-1.3 *	-5.9 *	-7.9 *	-5.6 *	-112.3 *	-2.5	-58 *	-2.0 *	8.5 *	5.2 *

3.3.1.2 Litterfall and exploitation residuals

The contribution of the elements from the solid fraction came mainly from litterfall compared to exploitation residuals and dust. The results of the input fluxes are presented in Table 11. The input of C into the two soils from the litterfall varied between 2867 and 2924 $kg.ha^{-1}.y^{-1}$. The fluxes of Ca, N, Si and K varied between 22.7 and 109 $kg.ha^{-1}.y^{-1}$. Mg, Mn, P and S were applied in quantities ranging from 2.1 to 8.1 $kg.ha^{-1}.y^{-1}$. Finally, Al, Fe and Na were present in smaller quantities: between 1.1 and 1.3 $kg.ha^{-1}.y^{-1}$. The contributions from the exploitation residuals were relatively high for C (between 515 and 592 $kg.ha^{-1}.y^{-1}$) and for N, Ca and K (between 2.2 and 6.4 kg. $ha^{-1}.y^{-1}$). The contributions of the other elements (Al, Fe, Mg, Mn, Na, P, S and Si) ranged from 0.1 to 0.7 $kg.ha^{-1}.y^{-1}$.

3.3.1.3 Total solid input

Five elements (C, Ca, N, Si and K) were introduced in large quantities by solid inputs: C (between 3450 and 3609 $kg.ha^{-1}.y^{-1}$), Ca (between 79 and 115.4 $kg.ha^{-1}.y^{-1}$), N (between 59 and 62.5 $kg.ha^{-1}.y^{-1}$), Si (between 28.6 and 50.2 $kg.ha^{-1}.y^{-1}$) and K (between 26.5 and 27.7 $kg.ha^{-1}.y^{-1}$) (Table 11). Mg, P, Mn and S were introduced in smaller quantities, with fluxes ranging from 2.2 to 8.9 $kg.ha^{-1}.y^{-1}$. Finally, Al, Fe and Na were introduced in the smallest quantities, with fluxes between 1.6 and 2.9 $kg.ha^{-1}.y^{-1}$. The total element inputs were thus strongly influenced by the litterfall inputs, which provided the greatest amount of each element in solid form, although the amount of each input type was not negligible.

3.3.2 Solid output

The outputs of the elements in solid form induced by water leaching through the humus layer are presented in Table 11. C and Si were found to be mobilized via particulate matter, with values between 54.3 and 22.2 $kg.ha^{-1}.y^{-1}$. Particulate Al and Fe had values between 11.2 and 5.9 $kg.ha^{-1}.y^{-1}$. Ca, K and N had moderate outputs associated with particles, with values ranging from 4.7 to 2.2 $kg.ha^{-1}.y^{-1}$. Mg, Mn, Na, P and S had small outputs associated with particles, with values between 1.06 and 0.18 $kg.ha^{-1}.y^{-1}$.

3.3.3 Solid budget

By comparing the inputs and outputs of element fluxes in solid form, we obtain the budget of the elements in the humus layer for the solid components (Table 11). The budget was balanced only for Si in the RL, the humus layer was a net source for Al and Fe in the DC and RL and for the other cases, the budget was a net sink.

4 Discussion

4.1 Comparison of the particles and the contribution of the particles to the outputs

This article presents, for the first time, the fluxes of the major elements leaving humus in particulate form. This flux, as with the input flux of atmospheric particles, is induced by the input and output of solutions from the humus layer. The output particles could therefore be a simple transfer of the input particles.

Indeed, some minerals, such as feldspar (anorthite, albite and potassium feldspar) and quartz, seem to be inherited from dust (Figure 24). However, the mineral grains leaving the humus layer show a rounding of the angular structure and a decrease in size. These morphological modifications reflect a strong dissolution of these minerals in humus (Drever and Stillings, 1997).

In addition, the differences in the chemical concentrations (Table 9) between the input and output particles do not support a simple, direct transfer but instead, a strong evolution of their chemistry. In particular, the concentrations of Ca, Mn, Fe and K in the outgoing particles are two to three times higher than those in the incoming dust. These elements are known to bind to organic materials or strongly cycle through vegetation (Rowley et al., 2018).

For all the elements, a systematic enrichment of the solutions leaving the humus layer, from three to sixteen times, was measured in the two soils (Table 11). A strong evolution of the flux of particles was observed, particularly in the organic phase, with an enrichment (x 5.4) of C. Such enrichment of C was also measured by (Schwesig et al., 2003) in two temperate forests dominated by *Picea abies* in northeast Bavaria, Germany.

The output fluxes of Na and Mg were two to four times higher than the input fluxes of these elements; those of Si, N, Al, C, K, P and Fe were four to seven times higher than the corresponding inputs; and those of Ca and Mn were eight to sixteen times higher than the corresponding inputs. Such enrichment has been observed only for Si in grassland, temperate and equatorial forest soils through the transfer of phytoliths (Cary et al., 2005; Cornelis et al., 2011) .

The chemical elements in the humus from the Montiers forest exist in the form of primary biominerals in plant tissues, such as phytoliths (Si) and calcium oxalates (Ca), or in the form of secondary biominerals, such as testate amoeba (Si, P, Ca, and Mn) or minerals precipitated around fungal hyphae (Ca and Mn) (Dincher et al, 2020a). Observations of the output particles show that they are enriched in these biominerals, which are probably entrained in the solution passing through the humus (Figure 24).

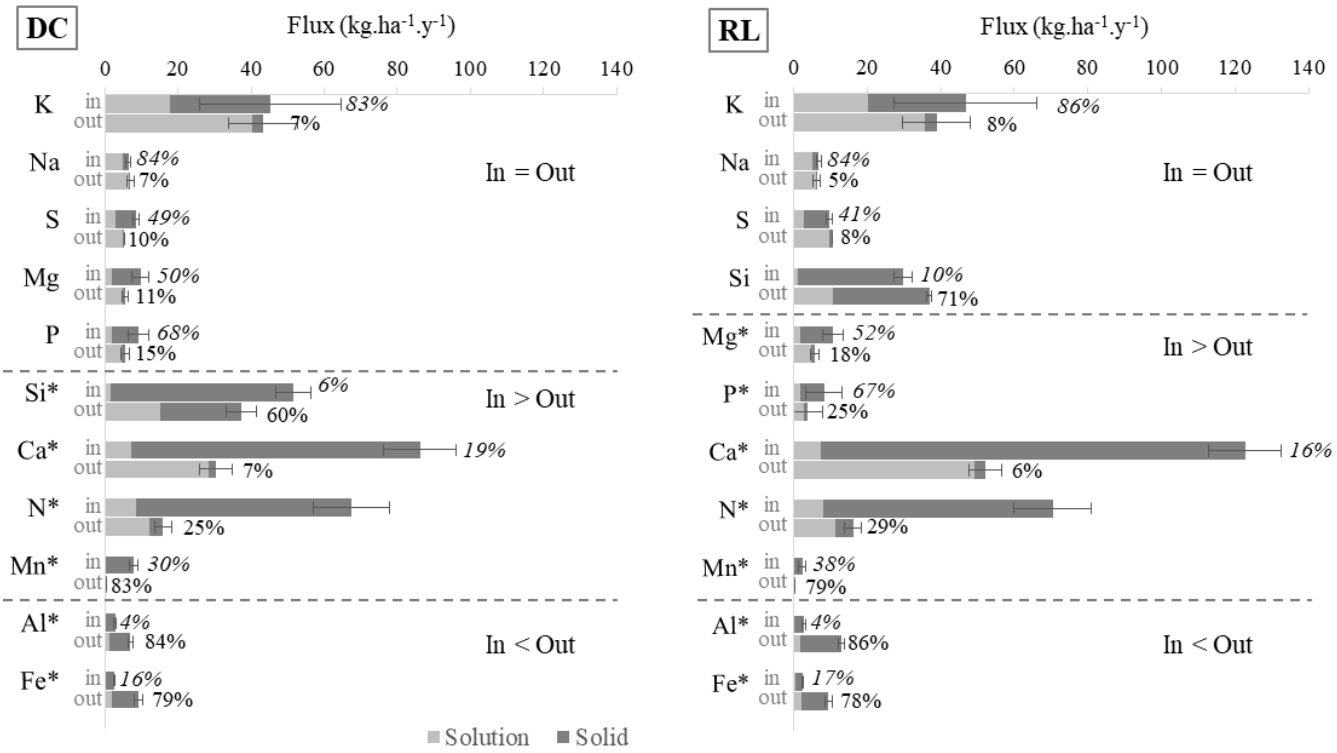

Figure 25 : Mean annual input and output fluxes of the elements in solution or as solids in the humus layer in kg ha^{-1}.y^{-1} between 2012 and 2018. The solid bars represent the total input (in) and output (out) flux with the contribution of the solutions in light gray and the contribution of the solids in dark gray. The error bars correspond to the standard deviations of the total flux. The value represents the percentage of the solid particles in the total output flux of the elements. The value in italics represents the proportion of the solution + soluble matter in the total inputs. The asterisks show the significant differences (p-value <0.05) between the inputs and outputs.

If some of the minerals entering the system via dusts were found in the outgoing humus particles, then the composition and mass of the outgoing humus particles would have been strongly influenced by the release of organic particles, the complexing of elements on the surfaces of the particles and the transfer of primary and secondary biominerals.

While organomineral particles contribute slightly (between 0.2 and 19%) to the inputs of elements other than Fe and Al, they contribute strongly to the outputs, particularly for some elements such as Mn and Si (Figure 25). Their quantification makes it possible to more accurately determine the balance of the elements in the humus layer. The particulate output flux is the main flux (more than 60% of the outputs) for Al, Fe, Mn and Si. This flux accounts for between 10 and 30% of S, Mg, P and N and for less than 10% of K, Na, and Ca. Even if these element fluxes were low, they improved the budget accuracy.

4.2 Element budgets in the humus

The budget of humus inputs and outputs in the two soil types is presented in Figure 25 : Mean annual input and output fluxes of the elements in solution or as solids in the humus layer in kg $ha^{-1}.y^{-1}$ between 2012 and 2018. The solid bars represent the total input (in) and output (out) flux with the contribution of the solutions in light gray and the contribution of the solids in dark gray. The error bars correspond to the standard deviations of the total flux. The value represents the percentage of the solid particles in the total output flux of the elements. The value in italics represents the proportion of the solution + soluble matter in the total inputs. The asterisks show the significant differences (p-value <0.05) between the inputs and outputs. for each element in solution form and in solid form, integrating the particulate flux. As the element stocks do not vary in the humus of the Montiers site during the study period (Dincher et al., 2020a), the element budgets should be balanced when all fluxes have been taken into account. However, on both soils, the budgets follow 3 scenarios: the inputs are effectively equal to the outputs, the inputs are higher than the outputs, and the inputs are lower than the outputs.

4.2.1 Inputs = Outputs

When the inputs are equal to the outputs, the budget is balanced. This is the case for 5 elements in the humus developed in the DC (K, Na, S, Mg and P) and 4 elements (K, Na, S and Si) in the RL humus. Dincher et al. (2020a) showed at the same site that the soluble fraction of K, Na S and Mg in plant tissues is significant. These elements are mainly transferred into the humus in a soluble and solution form (between 41 and 86% of the inputs; Figure 25). In addition, these elements mainly leave the humus in a dissolved form in a solution (between 82 and 95%). For Si in RL, the higher quantity of particles at the humus output had allowed to balance the input/output balance in this soil: this particle flux is therefore particularly important for this element.

This type of input/output budget at the level of the organic layer alone is rare in the literature, because it is generally established at the level of the organomineral layer (Berger et al., 2009; Liu et al., 2003; Whittaker et al., 1979). Spohn and Sierra (2018) also budgeted for humus in a eucalyptus forest in Australia based on solution, particulate and litter inputs and solution outputs. They showed balanced budgets not only for Mg (input = 27 $kg.ha^{-1}.y^{-1}$ and output = 24 $kg.ha^{-1}.y^{-1}$) but also for Ca (input = 70 $kg.ha^{-1}.y^{-1}$ and output = 65 $kg.ha^{-1}.y^{-1}$), while the outputs of K and P were always lower than the corresponding inputs. This difference is interpreted as the result of the uptake of these elements by eucalyptus roots from the humus.

4.2.2 Inputs > Outputs

The inputs are higher than the outputs for Ca, Si, N and Mn in the DC and for Mg, P, Ca, N and Mn in the RL. For these elements, the particle flux in the humus output does not balance that in the input. It therefore seems that some other outputs are not included in this budget.

Indeed, the gaseous outputs have not been quantified. They mainly concern two elements, C and N, which have significant gaseous phases in the form of CO_2 and N_2, respectively (Dixon et al., 1994; Johnson and Turner, 2014).

Although we did not observe roots in the humus, the presence of hyphae in our humus samples could support the removal and export of these elements outside the humus (Dincher et al., 2020a; Orwin et al., 2011). There could also be an export of the elements via earthworms observed in the humus (Sanderman and Amundson, 2003), especially for Ca, Si and Mn which were in biominerals form or bound to organic matter.

In addition, Dincher et al. (2020a) showed the precipitation of secondary biominerals (calcium oxalate and minerals containing Mn, Mg and/or P) in humus around hyphae and in testate amoebae with variable compositions of Si, Ca, P, Mn, and Mg. Primary biominerals, such as opal (Si) and calcium oxalate, are also present in the plant tissues entering the humus at the study site (Turpault et al, 2019; Dincher et al, 2020a).

An unidentified output could be linked to the biomineral properties, because all the elements the biominerals contain show higher inputs than outputs. Because biominerals have higher densities (calcium oxalate density: 2.12, (Molano-Flores, 2001); opal density between 1.9 and 2.5, (Bartoli and Wilding, 1980)) than the organic matter in the forest (humus bulk density: 0.1 to 1.1, (Berg and McClaugherty, 2014), it is possible that they settle on the soil surface.

Mn, which has very low measured outputs (0.2 to 0.4 $kg.ha^{-1}.y^{-1}$), seems to be the element most affected by these mechanisms (i.e., uptake by hyphae and sedimentation).

The fact that Si, Mg and P have a different balance depending on the soil (i.e., they are balanced in the DC but not in the RL) could be due to a richness in biominerals in the RL (Turpault et al., 2019, 2018), which results in a higher percentage of output in particulate form in RL (Figure 25).

4.2.3 Inputs < Outputs

The outputs of Al and Fe are higher than the inputs in the 2 humus types, which suggests that some of the inputs are not quantified.

Half of the inputs of these elements (in the range of 2.2 to 3 $kg.ha^{-1}.y^{-1}$; Figure 25) originated from the dust (46-56%). The outputs (in the range of 7 to 13 $kg.ha^{-1}.y^{-1}$; Figure 25) are mainly in the form of particles (between 78 and 86%). Although these fluxes are not very variable (on the order of 10-12%), the stocks of these elements in these humus

are heterogeneously distributed (stocks varying from 14 to 200%; Dincher et al, 2020a). In addition, these elements remain the longest in humus (mean residence time: 13-58 years; Dincher et al, 2020a).

As a result, unquantified inputs could correspond to an input of soil particles resulting from bioturbation, in particular by earthworms that would bring soil into the humus (Sanderman and Amundson, 2003) or possibly via capillary upwelling of the soil solution at the bottom of the humus. Given the aggressiveness of the medium towards soil particles and the pH of these humus types (4.9 and 5.7 pH solutions; (Kirchen et al., 2017)) (Rowley et al., 2018), organometallic Al or -Fe complexes are probably formed (Kleber et al., 2007; Mikutta et al., 2009) and transferred out of the humus in particulate form via the solution.

In relation to the long residence time and high variability in the stocks, another hypothesis is that a mobilization of Fe and Al from past accumulation is currently occurring in the humus.

5 Conclusion

This study presents a multielement budget of the inputs and outputs of two mull humus types, specifically taking into account the inputs and outputs in particulate form.

Although the particles leaving the humus layer had mineralogical similarities with the incoming particles, their form, chemistry and quantity revealed profound differences mainly due to the formation of organomineral particles in the humus. Element fluxes induced by these particles ranged from less than 1 $kg.ha^{-1}.y^{-1}$ to more than 50 $kg.ha^{-1}.y^{-1}$ and constituted an important factor in the humus output.

By considering particles in the input/output budgets, this study demonstrates balanced budgets for K, Na, S, Mg, and P mainly transferred in soluble form (in solution and by plants) and for Si (in RL) whose main output was in the form of particles. However, for Si (in DC), Ca, Mn, and N, which have positive budgets (i.e., inputs greater than outputs), some additional outputs had to be taken into account, such as the existence of a gaseous output, root uptake through hyphae, and the formation of primary and secondary biominerals (by testate amoebae, calcium oxalates, Mn crystals, and opal), which can settle on the soil surface. The budgets for Al and Fe indicated that not all input sources were taken into account (e.g., capillary upwelling or soil input) and that there was output in particulate form in organomineral complexes.

The elements linked to mineral phases (i.e., dust, primary and secondary biominerals, and secondary organominerals formed in the humus layer) strongly control the input/output budgets in the humus layer. Thus, even if the measurement of the outgoing particles from the humus did not make it possible to balance all the budgets of the elements, it highlighted new unidentified fluxes (in particular, the humus mineral deposits on the soil surface), which future studies will have to focus on. In addition, the contribution of this particulate flux will have to be specified as to its role (i) of new soil components (organo-mineral associations and minerals) with its quantification and proportion in relation to other soil components, (ii) in soil evolution of physical, chemical and biological properties (e.g. aggregate formation, cation exchange capacity, functional diversity of microorganisms) and (ii) in the soil and ecosystem services provided (e.g. carbon transfer and storage in soil). Thus, further studies could refine the understanding of these processes.

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V.

Chapitre V -

Seasonal dynamics of elements produced in the humus leaching solution in a beech forest

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Ce chapitre présente une étude fondée sur la détremination de la production d'éléments dans l'humus via la différence entre les flux en solution entrants et le flux d'éléments en solution sortant de l'humus. Il tend à répondre à la troisième question de recherche : À quelle période sont libérés les éléments dans la solution de l'humus et concorde-t-elle avec la période de prélèvement de l'arbre ?

Résumé

Le recyclage des litières est une clé pour le maintien des écosystèmes forestiers où les entrées de nutriments sont généralement faibles.

La plupart des études ont estimé la dynamique saisonnière de libération des éléments par la méthode des sachets de litières à un pas de temps biannuel et principalement pour C, N et P. Les objectifs de cette étude sont de déterminer les flux mensuels de C, N, P, K, Ca, Mg, Mn, S, Si, Fe et Al sortants de l'humus via le lessivage net (NL, correspondant au flux de solution sortant de l'humus moins les flux de solution entrants dans l'humus), de comprendre les mécanismes de libération des éléments et de déterminer si cette libération satisfait les besoins nutritionnels de la végétation.

L'étude du lessivage net de trois humus de type mull sur trois sols différents (Dystric Cambisol, Eutric Cambisol et Rendzic Leptosol) ayant les mêmes caractéristiques de peuplement et la même gestion forestière a révélé que tous les éléments sont libérés majoritairement entre novembre et février. Cette libération pourrait être liée à l'intensité des précipitations durant cette période. Cependant, différentes tendances entre les NL des éléments au cours des saisons sont apparues, révélant que d'autres facteurs sont responsables de la libération des éléments par le NL.

La forme des éléments dans les entrées pourrait influencer la libération des éléments. En effet, arrivant principalement sous forme soluble dans les végétaux, K et P sont d'abord lessivés dans la canopée, puis rapidement entraînés dans la solution (K est libéré à 97% durant la première anéne dans le dystric cambisol). Al, Fe et Si entrent principalement par les poussières et les biominéraux des végétaux et ont une libération plus tardive que les autres éléments. Ca, Mg et S sont repris dans des mécanismes de recyclage dans l'humus, ainsi la libération en mai pourrait être due à l'action des microorganismes. C et N ont une dynamique très différente due à la minéralisation de la matière organique par les microorganismes. Enfin, Mn n'est pas ou presque pas entraîné dans les NL. Il est donc soit particulièrement recyclé dans l'humus, soit repris par des flux non quantifiés.

Ces libérations d'éléments en hiver pourraient représenter une perte importante pour la nutrition de la végétation (qui prélève les nutriments pendant la période de croissance au printemps). Ainsi, les propriétés du sol ont un rôle-clé dans la rétention des éléments libérés par l'humus.

Seasonal dynamics of elements produced in the humus leaching solution in a beech forest

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1 Introduction

Forest ecosystems generally developed on soils that were nutrient-poor and were not amended. Most often, these soils were acidic, with a low capacity to provide elements through mineral weathering (Sverdrup and Warfvinge, 1988), or calcareous soils with deficiencies in K or/and P (Sardans and Peñuelas, 2015; Zederer and Talkner, 2018). In addition, increased demand for wood energy and short rotation practices reduce the amounts of nutrients in plant material available for plants in the soil (Feller, 2005; Kreutzweiser et al., 2008; Thiffault et al., 2011). Apart from atmospheric and soil mineral weathering inputs, the main source of nutrients for trees came from the intense recycling of elements (Braun et al., 2010; Gallardo et al., 1998; Jonard et al., 2009; Vitousek and Sanford, 1986).

Indeed, in temperate forests, trees can recycle up to 93% of elements (Vergutz et al., 2012). However, these values could differ depending on the type of soil and the type of humus (Kalbitz et al., 2000; van der Heijden et al., 2017).

Tree nutrition in temperate environments occurs during the growing season, e.g., spring and summer (Helcoski et al., 2019; Li et al., 2019; Lovelock et al., 2007; Soper et al., 2018). To be directly usable by the tree, element inputs should be released into the soil during this period. In autumn and winter, if the soil does not have the capacity to retain elements, the elements released are leached and lost for the ecosystem and, therefore, for tree nutrition.

However, the quantity of elements produced through litter decomposition could be different from the quantity that returns to the soil and is directly assimilated by the tree because some of these elements could be trapped in organic particles or in the form of biominerals ((Dincher et al., 2020b)).

Most of the studies investigating these release dynamics used a litterbag method to assess litter decomposition and most often focused on C, N and P (Jacob et al., 2010; Santa Regina et al., 1997). However, some multi-component studies, such as the one of (Vesterdal, 1999)), observed the evolution of 7 elements at a biannual time step. This wide time step, common to most litterbag studies (sampling every 3-6 or 12 months), did not allow the release dynamics over the year for the different nutrients and beneficial elements to be addressed. Only studies presenting a follow-up during the growing season of the element fluxes in the solutions leached below the humus would allow an assessment of these returns to the soil that were directly usable by the tree. Nevertheless, such studies are absent from the literature.

To fill this gap, this study aims to follow the monthly C, N, P, K, Ca, Mg, Mn, S, Si, Fe and Al fluxes in the net leaching (NL, calculated as the outgoing solution flux minus the

incoming solution flux in humus) from January 2012 to December 2018 in 3 different soil types with a mull humus type.

The objectives of this study are: (i) to determine the quantity of elements released via the NL over a monthly step, (ii) to compare the release fluxes during the growing season and the tree needs, and (iii) from the comparisons of the net leaching between the elements, to discuss the main mechanisms responsible for this dynamic.

2 Materials and methods

2.1 Experimental site

This study was conducted in the Montiers-sur-Saulx experimental beech forest site located in North-Eastern France (Meuse department, 48° 31′ 54″ N, 5°16′ 08″ E). This experimental site of 73 ha was managed jointly by the INRAE-BEF (French National Institute for Agricultural, Food and Environmental Research - Biogeochemical Cycles in Forest Ecosystem Research Unit) and by the ANDRA (French National Radioactive Waste Management Agency) since 2012. The Montiers site is part of the national research network AnaEE (Analysis and Experimentations on Ecosystems).

The climate is semi-continental (cold winters and hot summers), with a mean annual rainfall and temperature between 2012 and 2018 of 1081 mm and 9.4°C, respectively (calculated from météo-France data) (Figure 26). In winter, the soil can temporarily freeze over a depth of a few centimetres and is occasionally covered with snow. The site is covered by a homogeneous same-aged stand (approximately 60 years old) with the same management approaches. The stand is mainly composed of beech (89%) and other species (11%), i.e., sycamore maple (*Acer pseudoplatanus*), ash (*Fraxinus excelsior*), pedunculate oak (*Quercus robur L.*), European hornbeam (*Carpinus betulus L.*) and wild cherry (*Prunus avium*). The geology of the Montiers site is constituted of two overlapping soil parent materials: an underlying Tithonian limestone surmounted by detrital acidic Valanginian sediments (Kirchen et al., 2017). The difference in thickness of these sediments induces the development of 3 different soil types: Dystric Cambisol (DC), Eutric Cambisol (EC) and Rendzic Leptosol (RL) (FAO, 2006). A schematic representation of the soil profiles and their location is presented in (Turpault et al., 2019). Humus type is a eutrophic mull for RL and EC and an acidic mull for DC. No roots were found in the humus of the 3 soils.

Three experimental plots were established on the three different soils and replicated three times each. All the replicates (2500 m^2) were equipped with the same monitoring device for collecting the aboveground and belowground solutions at different depths. The soil and organic solutions were collected in lysimetric collectors of 0.12 m^2 and 0.07 m^2 for the organic solutions (3x8 collectors per replicate per depth). Using these thinner lysimetric collectors made it possible to limit the stagnation of the solution and to collect the organic solution as much as possible. The throughfalls were collected in 4 gutters per replicate, stemflows in 6 collectors per replicate and the standing aboveground in 6 litter traps of 0.34 m^2 per replicate. In addition, a 45-m-high flux tower was placed within the site (close to plot DC) to collect rainfall and atmospheric deposits. For more information, see (Calvaruso et al., 2017; Kirchen et al., 2017; Turpault et al., 2019).

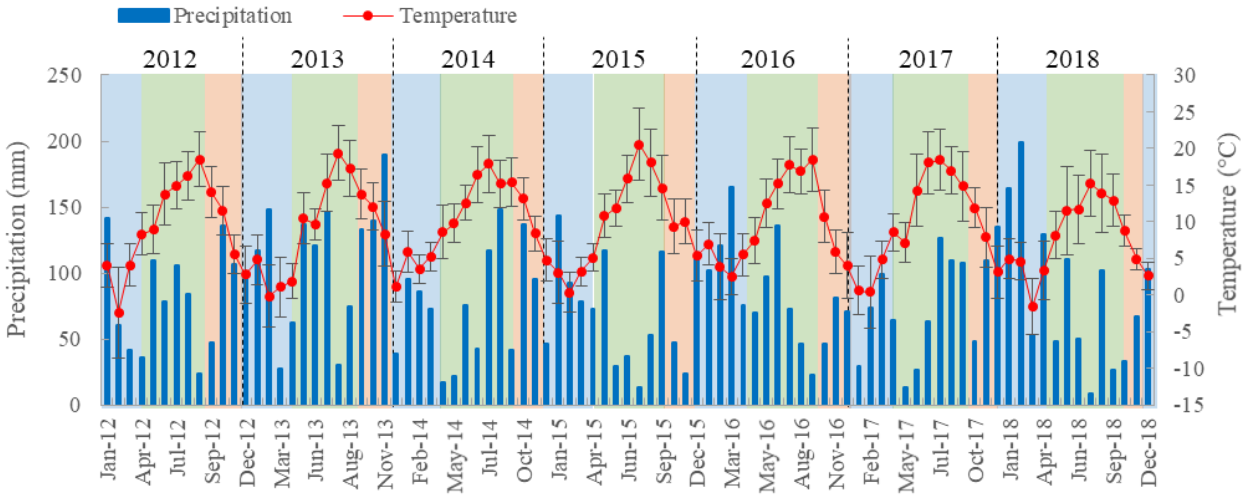

Figure 26 : Mean precipitations and temperatures measured every 28 days from January 2012 to December 2018.

2.2 Sampling and analytical method

2.2.1 Above-ground compartment

In each plot in 2009, eight beech were harvested to collect stem wood, stem bark, and branches. The branches were separated into 3 different classes of diameter : <4, 4-7 and >7 cm. For more information, see (Calvaruso et al., 2017).

All the samples from the above-ground compartments of the trees were dried in a stream air-drier (at 65°C). Then, they were ground and encapsulated for analysis. The total Ca, K, Mg, P, Al, Mn, S, Si and Fe contents were assessed by X fluorescence using an X fluorescence sequential spectrometer S8 TIGER 1 kW (Bruker). The total C and N contents were assessed using a CHN analyser (EA/NA 1110, THERMOQUEST).

2.2.2 Solutions above and below humus

Sampling methods for each solution have already been described in (Turpault et al., 2019, 2018). Briefly, aboveground solutions (throughfall TF and stemflow SF) and humus solution (zero-tension lysimeters) were sampled every 4 weeks between January 2012 and December 2018.

All solutions were filtered at 0.45 µm and analysed just after the sampling. The element contents in the solutions were measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES Agilent Technologies 700 type ICP-OES, Santa Clara, USA). The total C and N contents of the samples were determined by a Thermo Quest Type NCS 2500 analyser.

2.3 Calculation

2.3.1 Element flux in solution

In each soil, the element fluxes (TF, SF and humus solution) were obtained by multiplying the volume of solution by the concentration of the elements in the solution. Every 4 weeks, the element fluxes were calculated per hectare. The Biljou© model (Granier et al., 1999) was used in the three soils of the experimental site to evaluate the water drainage flux in the humus layer. All the information on the data and the procedure are available in Kirchen et al. (2017).

2.3.2 Element net leaching in the humus

Net leaching (NL) in humus is calculated for a period of 28 days as follows:

$NL = S_{ol} - (TF+SF)$

Where NL is the net leaching in one element in the humus layer in $kg.ha^{-1}.period^{-1}$; S_{ol} is the humus solution outgoing in $kg.ha^{-1}.period^{-1}$; TF is the throughfall in $kg.ha^{-1}.period^{-1}$ and SF is the stemflow, in $kg.ha^{-1}.period^{-1}$.

2.3.3 K, P, Mg and S release rate

The studies of (Dincher et al., 2020a) showed that K, P, Mg and S have a short residence time and have a balanced budget in the Montiers site. Thus, it is interesting to know how and when these elements are released in the solution.

For each element, the first release rate was calculated from the total input of the element by the litterfall. From October, the element net leaching in the humus layer was subtracted from the previous month. The first element release can thus be calculated as follows:

$R_E = L - NL_{oct}$

Where R_E is the release rate of one element (K, P, Mg or S) in $kg.ha^{-1}.period^{-1}$, L is the flux of this element in litterfall for one year in $kg.ha^{-1}.y^{-1}$ and NL_{oct} is the net leaching of this element in October in the humus layer, in $kg.ha^{-1}.period^{-1}$.

For the other months, the release rate was calculated as follows:

$R_E = L^{m-1} - NL_m$

Where, L^{m-1} is the flux of on element in the litterfall of the month before in $kg.ha^{-1}.period^{-1}$ and NL_m is the net leaching of this element of the month in the humus layer in $kg.ha^{-1}.period^{-1}$.

2.3.4 Elements in tree perennial part

For the three plots, the biomass of each tree was calculated for each aboveground compartment, i.e., branches <4 cm in diameter, branches between 4 and 7 cm in diameter, branches >7 cm in diameter and wood stem and bark stem for three soils in both 2009 and 2018. To estimate the standing biomass, we used the procedures described in (Calvaruso et al., 2017) based on the allometric equation in (Picard et al., 2012). This allometric equation linked easy-to-measure tree attributes, i.e., height, diameter at breast height, and age of trees.

To simplify site measurements, Calvaruso et al., 2017 developed a model allowing the estimation of the total height of beech trees from their stem diameter at breast level for each soil type.

They obtained the following equation:

$$h = 1.3 + a * (1 - \exp(-b * d))^c$$

Where h is the total tree height in m, d is the stem diameter at breast height in mm; and a, b and c are the parameters to estimate.

To calculate the biomass immobilization, we multiplied the biomass of each compartment by the concentration of an element in this compartment from the element concentrations obtained from the 8 trees cut per soil in 2009.

Annual above-ground immobilization (in the tree perennial part) was calculated as the difference between the amount of element in the biomasses calculated for 2009 and 2018, divided by nine.

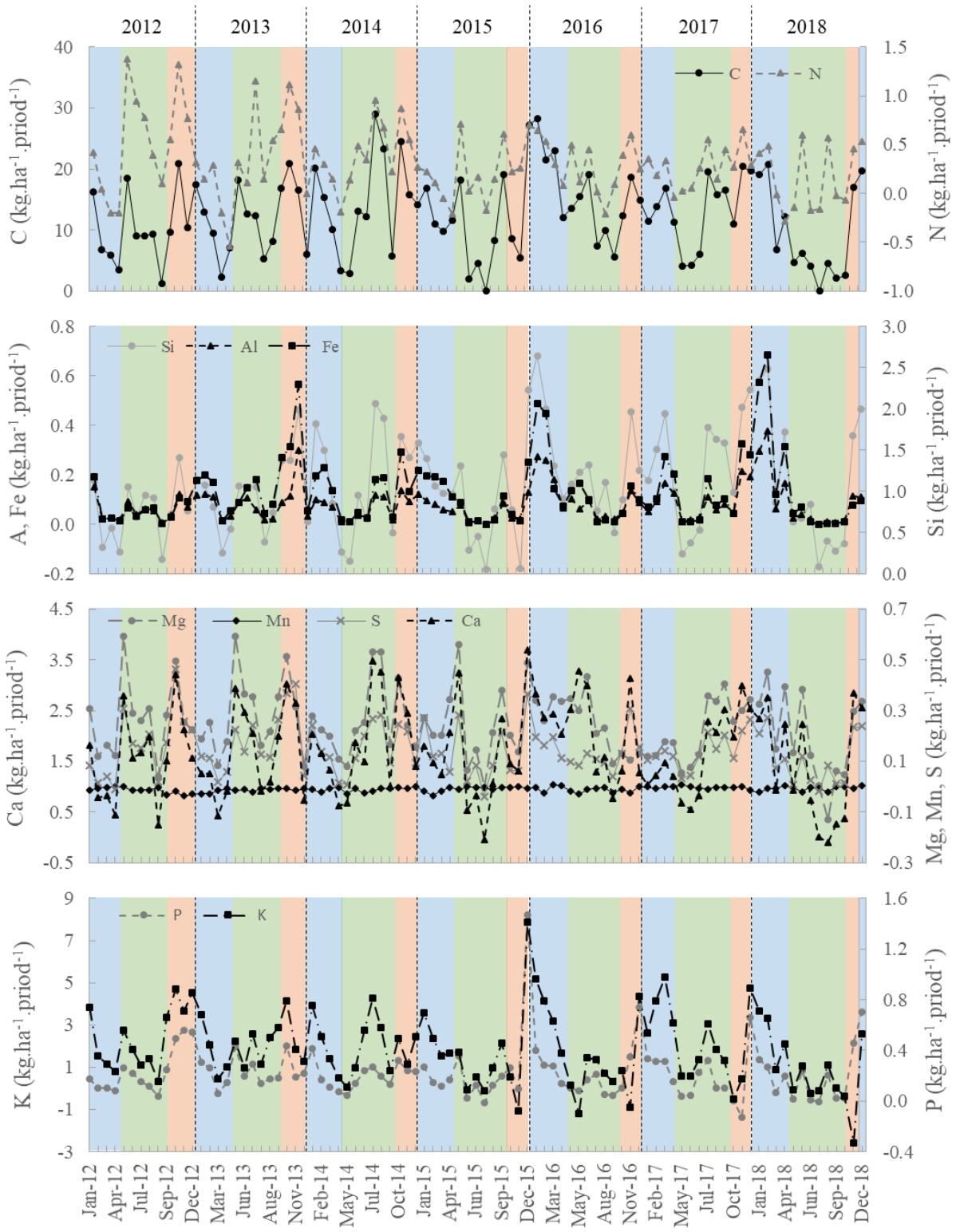

Figure 27 : Net leaching evolution from 2012 to 2018 in kg.ha-1.period-1 for the studied elements in the dystric cambisol. The blue stripes correspond to periods of dormancy (winter), the green stripes to periods of tree growth (spring and summer) and the orange stripes to periods of senescence (fall).

3 Results

3.1 Interannual net leaching evolution

Figure 27 shows the dynamics of net leaching of elements in humus over 7 years (2012-2018) on DC (Figures S1 and S2). For C, the NL was between 0 and 30 kg.ha^{-1}.period^{-1}. K, and Ca and Si had NLs between -2.6 and 8 kg.ha^{-1}.period^{-1}. For N and P, the NLs were between -0.5 and 1.5 kg.ha^{-1}.period^{-1}. The net leaching for Al, Fe, Mg and S ranged from -0.1 to 0.7 kg.ha^{-1}.period^{-1}. Finally, Mn had a very low NL and a very weak variation, with values between -0.04 and 0.01 kg.ha^{-1}.period^{-1}. However, when comparing NL fluxes between soils, it appeared that these fluxes were higher in RL for S (changes between -0.03 and 1.35 versus -0.04 and 0.46 kg.ha^{-1}.period^{-1}) and Ca (changes between -0.1 and 3.7 versus 0.05 and 8.05 kg.ha^{-1}.period^{-1}) (Figure S3). The flux values for the other elements in the different soils showed similar patterns.

According to the seasons, even if a few years showed particularities, a pattern of NL seems to take place: peak of element release in senescence and early winter (October to January) then stability in late winter and during the growing season.

3.2 Mean monthly net leaching evolution

Figure 28 presents the mean monthly evolution of NL of elements in solution in humus of the three soils studied. The curve patterns were similar between the 3 soils. Only the amounts of NL for Ca and S were different between the soils: they were present in greater quantities in RL than in DC and EC.

Several elements showed similar developments from month to month. While Al and Fe showed peaks in NL between November and January, NL then decreased and stabilised until September between 0.8 and 0.5 kg.ha^{-1}.period^{-1} for Si, to approximately 0.05 kg.ha^{-1}.period^{-1} for Al and 0.07 kg.ha^{-1}.period^{-1} for Fe. K and P were highest in December and decreased and then stabilized until October at an NL of approximately 0.8 kg.ha^{-1}.period^{-1} for K and 0.1 kg.ha^{-1}.period^{-1} for P. In November, for these 2 elements, there were brutal decreases in the NL. The NLs of Ca, Mg and S showed peaks in November, January and May and then stabilized until September. C and N showed increases in NL from October to November, and then in January and April, they decreased and then stabilized until September. NLs were stable at approximately 1.4 kg.ha^{-1}.period^{-1} for Ca in EC and RL and 1.5 in DC; 0.2 kg.ha^{-1}.period^{-1} for Mg; 0.4 kg.ha^{-1}.period^{-1} for S in RL and 0.1 in DC and EC. Precipitation had a simple pattern: there was high precipitation during the dormant period (winters) and lower precipitation during the growing season (spring and summer) and senescence (fall).

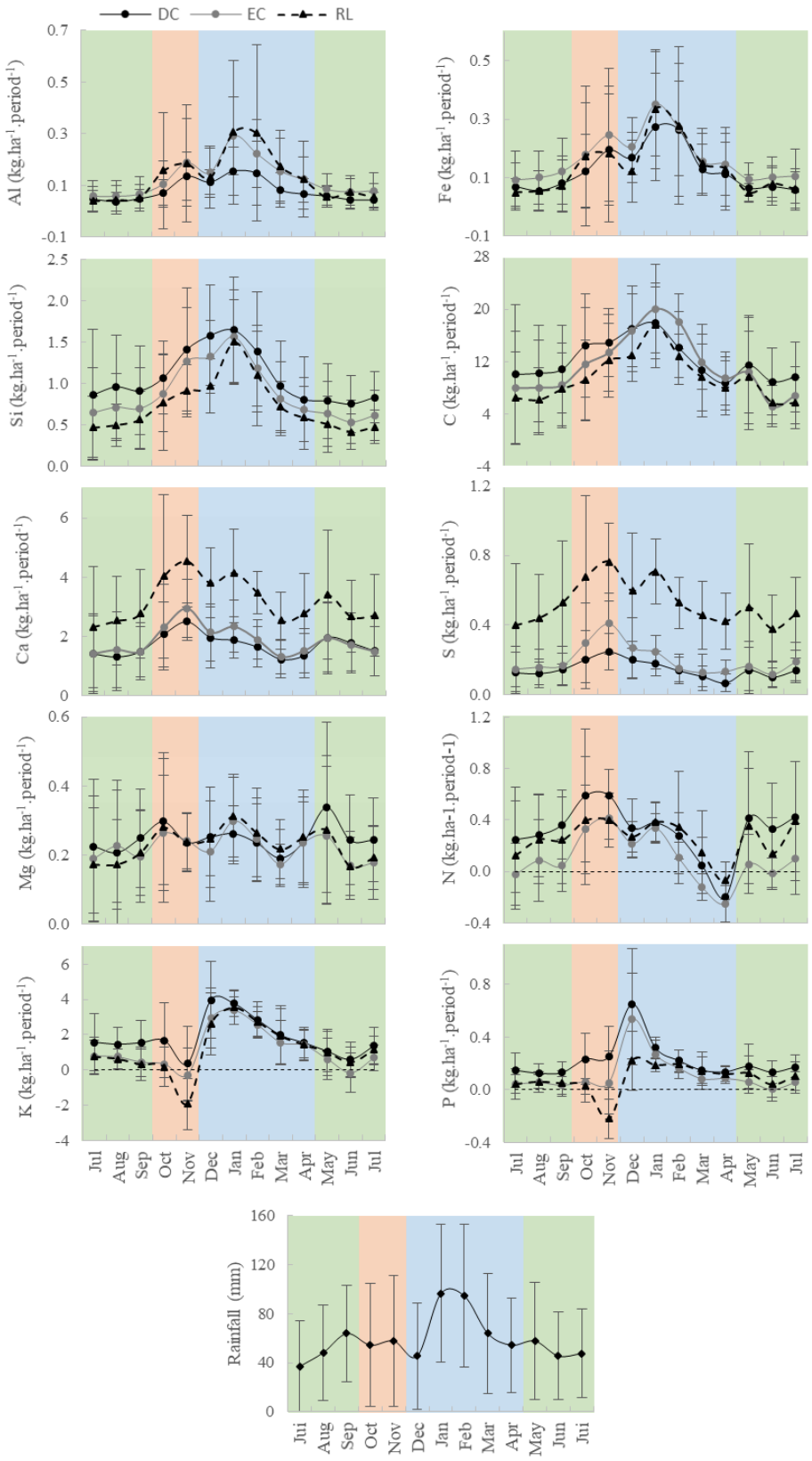

Figure 28 : Mean monthly net leaching evolution in kg.ha^{-1}.period^{-1} in the three soils of the Montiers experimental site (DC, EC et RL) for the elements studied and precipitation every 4 weeks. The blue stripes correspond to periods of dormancy (winter), the green stripes to periods of tree growth (spring and summer) and the orange stripes to periods of senescence (fall). The SDs of net leaching are represented by black bars.

3.3 Release kinetics in net leaching

Figure 29 shows the seasonal release of K, Mg, P and S as a function of the amounts of elements contributing to litterfall (T0) in DC and RL. Figure 28, corresponding to the monthly evolution of the elements in the NL, showed that the NL of K had a peak in December. Indeed, in December, NL represented a departure of 16% of the total K of litter in DC and 11% in RL. In DC, half of the total K of the litter was taken out in NL in March, 4 months after the litterfall. In RL, half of the K was released in April, i.e., after 5 months. From April onwards, the rate of release of K via NL decreased. By November, one year after the fall of litter, almost all of the K had left as NL in DC (97%) and RL (70%). For P and Mg in DC, only half of the litter fall was released after 1 year. In RL, there were less than a third released. For S, only one-third was released after one year in DC. Conversely, in RL, half of S was released in May, 6 months after litter fall. After 1 year, the whole S was released via NL.

3.4 Connexion between elements in net leaching with precipitation

Table 12 presents the relationships between the elements in net leaching as well as the relationships between the elements in NL and in precipitation through a matrix of determination coefficients (R^2). C seemed to be related to most of the elements: Al, Ca, Mg, S and Si ($0.55 < R^2 < 0.82$). Then, several groups of elements can be distinguished: Al, Fe and Si were strongly related ($0.64 < R^2 < 0.89$). Ca, Mg, S and N also appeared to be related in NL ($0.55 < R^2 < 0.79$). Finally, K and P were only related to each other. Although in DC, $R^2=0.5$, in EC and RL, it was greater ($0.60 < R^2 < 0.71$) (Table S1 and S2). Mn in NL was not related to any element or to precipitation in any of the three soils studied. Precipitation was related to the majority of elements in NL (Al, Ca, Fe, Mg, S, Si and C), though with a median coefficient of determination ($0.53 < R^2 < 0.58$).

Table 12 : Matrix of determination coefficients between elements and precipitation in DC. $0.5<R^2<0.7$ are in blue, and $0.7<R^2$ are in green.

	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N	C	Precipitation
Al												
Ca	0.40											
Fe	0.89	0.31										
K	0.28	0.16	0.28									
Mg	0.26	0.79	0.20	0.21								
Mn	0.01	0.02	0.02	0.03	0.00							
P	0.14	0.25	0.10	0.50	0.18	0.02						
S	0.37	0.66	0.30	0.33	0.55	0.05	0.38					
Si	0.72	0.63	0.64	0.34	0.39	0.00	0.28	0.52				
N	0.16	0.43	0.12	0.19	0.37	0.06	0.18	0.63	0.23			
C	0.55	0.79	0.44	0.42	0.60	0.01	0.34	0.65	0.82	0.40		
Precipitation	0.58	0.55	0.53	0.31	0.54	0.06	0.17	0.55	0.54	0.34	0.58	

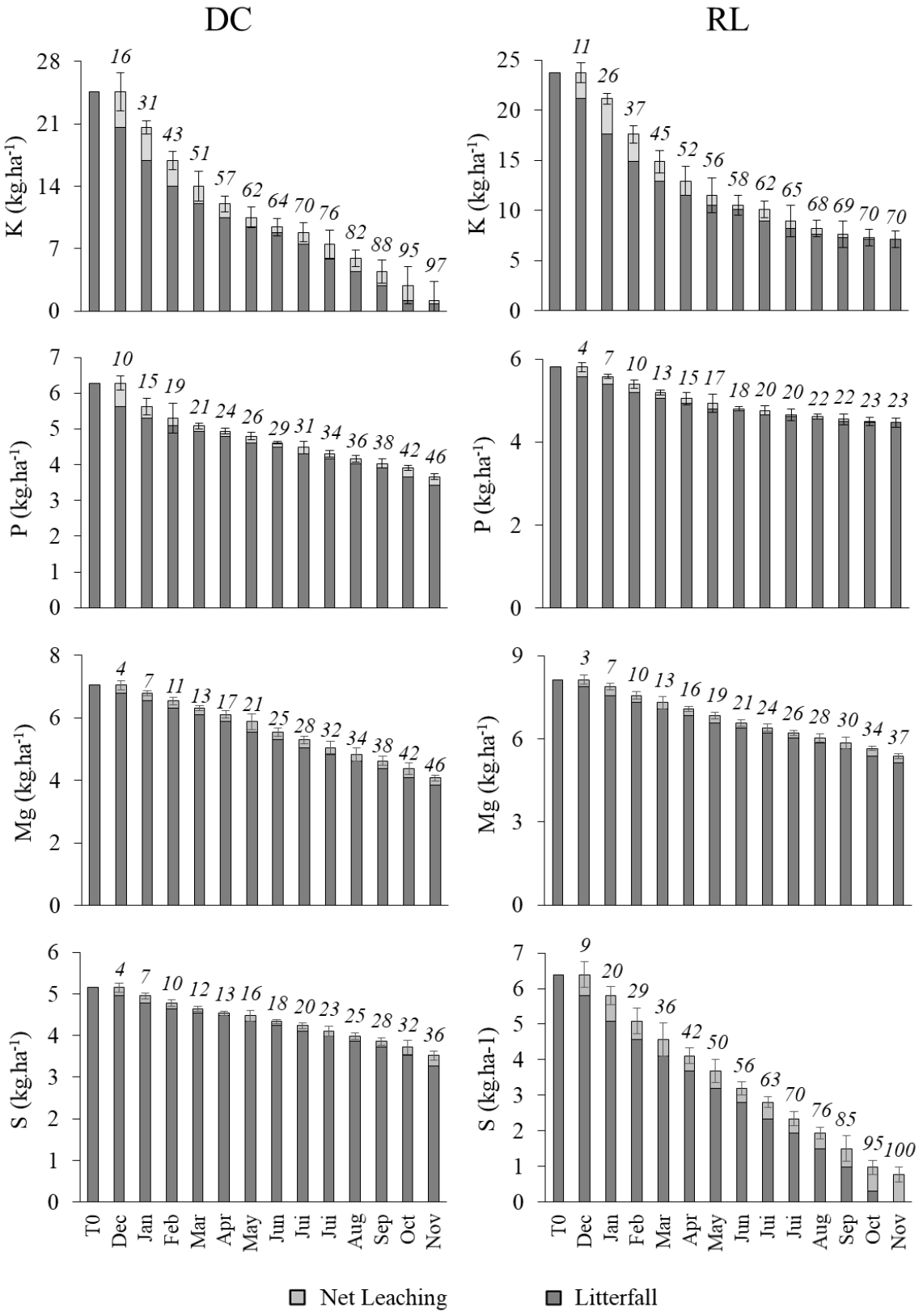

Figure 29 : K, Mg, P and S release per month (4 weeks) in kg.ha^{-1} through NL in relation to the amount brought by litter (T0) in DC and RL. The numbers in italics correspond to the cumulative percentage of elements output as NL. The standard deviation is indicated by black bars.

4 Discussion

4.1 Seasonal dynamics of element release via net leaching and tree needs

4.1.1 Seasonal dynamics of element release via net leaching depending on soils

The seasonal dynamics of the elements released from humus via NL in the three different soils of the Montiers site were globally similar. The soil does not influence the seasonal evolution of the elements in the net leaching. However, some differences in terms of quantity were observed (Figure 28). Indeed, Ca and S were present in higher quantities in RL compared to other soils. Moreover, S released faster in LR: it was completely consumed after one year (Figure 29). These higher quantities in RL were directly related to the composition of the litterfall, which was enriched in Ca and S compared to the other two soils because RL is developed on a limestone bedrock (Calvaruso et al., 2017; Dincher et al., 2020a).

4.1.2 Seasonal dynamics of element release via net leaching depending on tree needs

In general, the elements were released into the NL during the senescence/dormancy period on the three soils studied (Figure 28). In fact, for all elements, whatever the soil, more than 50% of the elements were released during this period (Figure 30). Because tree nutrition takes place during the spring/summer (Helcoski et al., 2019; Soper et al., 2018), this great amount of elements released during the senescence/dormancy period could be lost for the ecosystem. Soil plays a fundamental role here by retaining the released elements, thus limiting leaching loss, and making them usable by trees for the next growing periods.

This release of elements in winter could be a real problem for the recycling of elements. Indeed, it has been shown in the literature that in temperate forest ecosystems, element recycling can be as high as 65, 88, 89, and 93% for Ca, K, P and N, respectively (Vergutz et al., 2012). On the Montiers site, the needs in elements for the perennial parts could largely be covered by the elements released by the NL during the year. For example, for K, the annual demand for the perennial part was 13.0 $kg.ha^{-1}.y^{-1}$, and NL releases 19.4 $kg.ha^{-1}.y^{-1}$ into DC; for Mg, the requirement was 2.7 $kg.ha^{-1}.y^{-1}$ for 3.0 $kg.ha^{-1}.y^{-1}$ of NL (Figure 30). This shows the importance of the element removal by the vegetation via the elements driven into the NL.

To better understand this release, it is important to know the factors that lead to the release of elements during the senescence/dormancy periods.

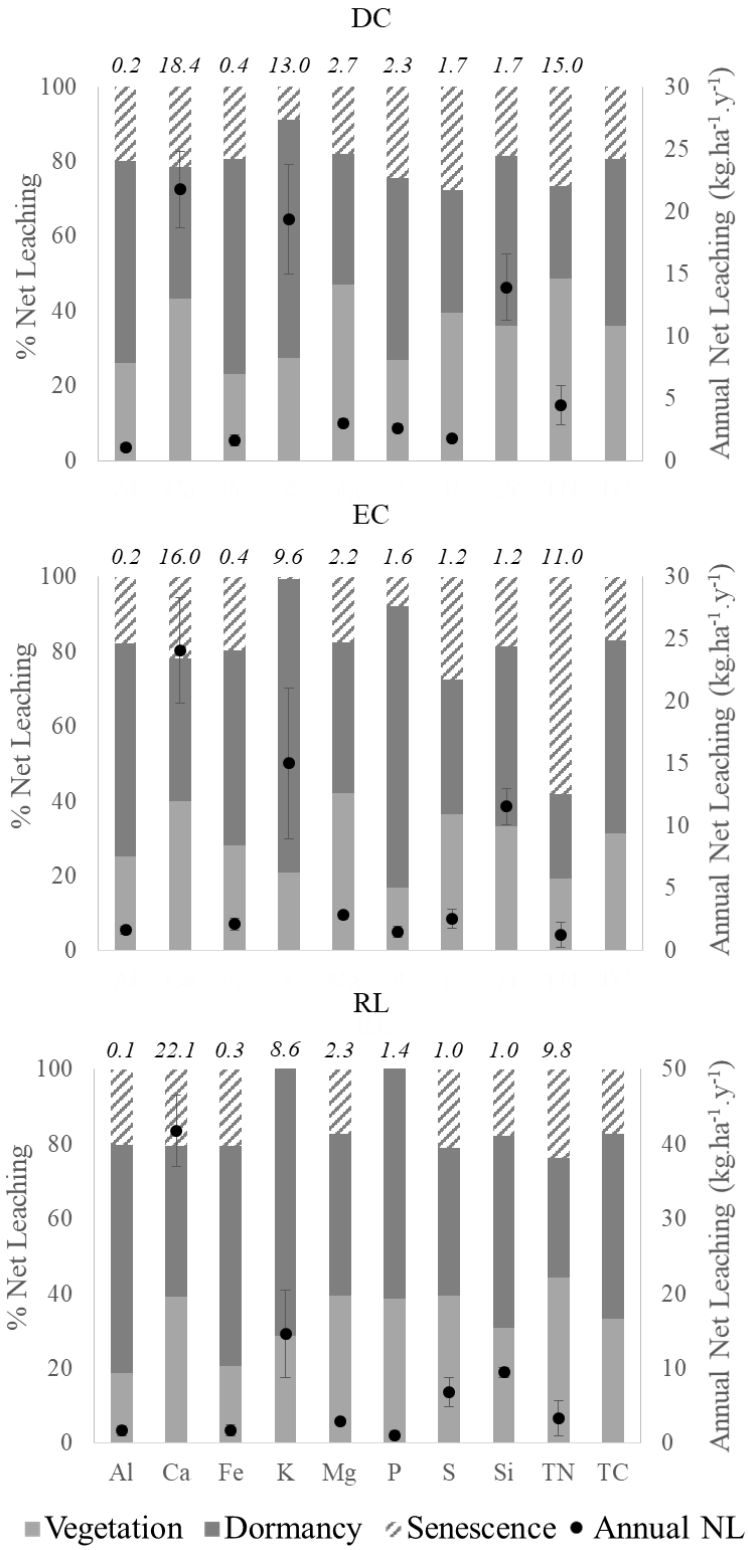

Figure 30 : %Net leaching by season for all elements, amount of annual NL in kg.ha^{-1}.y^{-1}, and in italics, immobilization of elements in the perennial part of the tree in kg.ha^{-1}.y^{-1}.

4.2 Factors contributing to the release of elements via net leaching

4.2.1 Water flux

The main release of elements during senescence/dormancy observed in our study corresponds to the data in the literature, and in particular, the data from the litterbag degradation experiment, which shows losses of mass and elements during this period of the year (Santa Regina et al., 1997; Vesterdal, 1999). This is, for example the case of Jacob et al., 2009, who conducted a study of elemental release via litterbags in a beech forest in Germany. In this study, element loss was measured by determining the remaining elements present in the litterbags. It appears that the greatest losses for C, P, Ca, Mg also occurred during senescence/dormancy.

This element release during this period could be related to the important level of precipitation (Austin and Vitousek, 2000).

In our study, the NLs of Al, Ca, Fe, Mg, S, Si and C were indeed relatively well correlated with precipitation, with r^2 coefficients between 0.53 and 0.58 in DC (Table 12). In addition, the peak of precipitation in winter could increase the leaching of elements into the humus, and the low precipitation in summer could explain the stable state of the NL during the growing season. However, even if all elements showed an overall dynamic similar to the precipitation dynamics (NL high in dormancy and low in vegetation), specific events in the seasonal dynamics suggest that other factors influence the release of elements via the NL.

4.2.2 Influence of element solubility in plants (case of K and P)

K and P, with r^2 values between 0.5 and 0.7 in the NL of the three soils (Table 12 and Annex), have the same seasonal pattern (Figure 27). Inputs were mainly via litterfall and were mainly found in the soluble fraction of plants: 71 and 64%, respectively, in DC, and 76 and 62% in RL (Table 13; (Dincher et al., 2020b)). Due to their soluble nature, K and P were initially leached from the canopy during leaf senescence on the tree (Parker, 1983; Salehi et al., 2016).

The absence or deficit of NLK and NLP could thus be explained by a total loss of labile K and P in the canopy when the leaves fall to the ground. It is also possible that K and P amounts in the throughfall were overestimated because the leaves falling into gutters release elements by leaching. Indeed, soluble P and K could be leached directly into gutters and increase the levels of P and K in the throughfall. As the process of degradation and rupture of new cells progressed, these elements, mainly in solution form, were released during periods of dormancy and senescence (more than 50%). During growing periods, the process was slowed down, with a release between 17 and 47%.

By studying the rate of release of K and P in DC, after one year, K was fully released via NL, and P was fully released after 2.19 years (Figure 29). In RL, the release rates were slower for both elements, with 1.43 years for K and 4.36 years for P, and were similar to those calculated from the ratio between the solid inputs and the stock in humus (Dincher et al., 2020a): for K, 0.8 year in DC and 0.7 year in RL; and for P, 2.2 years in DC and 4.4 years in RL (Table 13). K and P were also elements with a balanced input and output budget. Thus, when P was only 46% released in 1 year in DC, this implies outputs of this element in another form than by net leaching. Table 13 indicates that these elements can also be released as a particle (15% of the output in DC) and that they were taken up by recycling mechanisms such as the use of elements for the creation of amoeba testate or the secondary precipitation of biominerals around the hyphae (Dincher et al., 2020a).

The relationship between K, P, their soluble form and their NL has already been shown by (Attiwill, 1968). This study was conducted in an Eucalyptus obliqua forest in Australia. It presents the relationship between the mobility of the elements in the solid entrances, their short residence time and their leaching in the canopy: the more mobile the element is in the solid entrances, the more it will be leached in the canopy, and the shorter its residence time in the litter will be.

4.2.3 The importance of the element form in inputs and outputs (Case of Al, Fe, Si)

In the NL, Al, Fe and Si appeared to be related elements with similar seasonal variation patterns. Humus inputs were mainly in solid form for Si, via phytoliths issued from plant compartments (97%; (Turpault et al., 2018); (Dincher et al., 2020b); Table 13). The fluxes of Fe and Al were low and variable: a portion entered via dust (22 and 11% of the solid inputs in DC), and the rest, not determined, probably came by fixation in contact with the soil (Dincher et al., 2020b, Table 13). In addition, the humus outputs of these 3 elements were mostly in the form of particles (respectively, 84, 79 and 43% for Al, Fe and Si in DC and 86, 78 and 60% in RL). Al, Fe and Si show peaks of NL in November when leaves fall, which could represent the leaching of the small soluble fraction of these elements from the leaves (Attiwill, 1968). The second peak in January (during the dormant period) could be induced by water flow. Indeed, Al, Fe and Si have an r^2 of 0.5 with precipitation (Table 12). Thus, the action of the water could lead to the solubilisation of part of the insoluble fraction of these elements in the NL (Sanderman and Amundson, 2003). Si was arriving in greater quantities than Al and Fe (Table 13). Thus, the Al and Fe outputs were completely explained by the outputs in particle form and in solution. For Si, 23% of the outputs were not explained; this element could be included in recycling mechanisms taking place in humus. For example, (Sommer et al., 2013) discuss the use of Si in phytoliths by testate amoebae. Si can also be chemically recycled as a precipitation of amorphous silica on the remains of decomposed limbs in humus (Dincher et al., 2020a).

Table 13 : Summary table of total inputs, outputs, NL and if the balance of inputs/outputs is equilibrated in DC and RL. MRT = Mean Residence Time, MRTNL = Net Leaching Mean Residence Time. 1 percentage of total input, 2 percentage of total output, 3 percentage of total NL.

		Al	Ca	Fe	K	Mg	Mn	P	S	Si	TN	TC
							DC					
	MRT	22.8	2.5	58.4	0.8	2.0	2.2	1.8	2.7	3.3	3.2	1.6
	MRT_{NL}				1.0	2.2		2.2	2.7			
INPUT	**Total Input (kg.ha^{-1}.y^{-1})**	**2.8**	**86**	**2.6**	**45**	**9.7**	**7.8**	**9.1**	**8.5**	**52**	**67**	**3525**
	%Solution input [1]	1	8	4	39	19	3	21	35	3	12	2
	%Solid inputs (plants + dust) [1]	39	92	24	61	81	97	79	65	97	88	98
	%Soluble fraction	*2*	*11*	*5*	*71*	*40*	*28*	*64*	*26*	*2*		
	%Insoluble fraction	*76*	*89*	*84*	*27*	*57*	*71*	*34*	*74*	*87*		
	%Dust fraction	*22*	*0*	*11*	*1*	*3*	*0*	*2*		*11*		
	% Undetermined [1]	60	0	72	0	0	0	0	0	0		
	Balanced total budget	No	No	No	Yes	Yes	No	Yes	Yes	No	No	No
OUTPUT	**Total Output (kg.ha^{-1}.y^{-1})**	**7.0**	**30**	**9**	**43**	**6**	**0**	**5**	**5**	**37**	**16**	**260**
	%Solution output [2]	16	33	21	93	89	1	85	89	29	18	6
	%Particles output [2]	84	3	79	7	11	5	15	11	43	6	1
	%Undetermined [2]	0	65	0	0	0	94	0	0	28	76	93
NET LEACHING	**Total NL (kg.ha^{-1}.y^{-1})**	**1.0**	**21.8**	**1.6**	**19.4**	**3.0**	**0.03**	**2.6**	**1.8**	**13.9**	**4.4**	**148**
	%NL Dormancy [3]	54	35	57	64	35	30	49	33	45	25	44
	%NL Vegetation [3]	26	43	23	27	47	65	27	40	36	49	36
	%NL Senescence [3]	20	21	19	9	18	5	24	28	19	27	19
	Recycling	-	++	-	+	+	+	+	-	++		
							RL					
	MRT	23.3	2.0	13.1	0.7	1.3	2.0	1.4	1.6	3.3	2.0	1.2
	MRT_{NL}				2.7	2.7		4.4	1.0			
INPUT	**Total Input (kg.ha-1.y-1)**	**2.8**	**123**	**2.6**	**47**	**10.7**	**2.3**	**8.2**	**9.7**	**30**	**70**	**3506**
	%Solution input [1]	1	6	4	43	17	4	20	30	4	11	2
	%Solid inputs (plants + dust) [1]	21	94	23	57	83	96	80	70	96	89	98
	%Soluble fraction	*1*	*11*	*5*	*76*	*45*	*35*	*62*	*20*	*5*		
	%Insoluble fraction	*87*	*89*	*84*	*23*	*53*	*64*	*36*	*80*	*76*		
	%Dust fraction	*12*	*0*	*11*	*1*	*2*	*1*	*2*		*19*		
	% Undetermined [1]	78	0	73	0	0	0	0	0	0		
	Balanced total budget	No	No	No	Yes	Yes	No	Yes	Yes	No	No	No
OUTPUT	**Total Output (kg.ha-1.y-1)**	**13.0**	**52**	**10**	**39**	**5.8**	**0.2**	**3.7**	**11**	**37**	**16**	**234**
	%Solution output [2]	14	40	22	92	45	2	34	93	24	16	5
	%Particles output [2]	86	3	78	8	10	8	12	7	60	7	2
	%Undetermined [2]	0	57	0	0	45	90	54	0	16	77	93
NET LEACHING	**Total NL (kg.ha-1.y-1)**	**1.7**	**41.8**	**1.7**	**14.7**	**3.0**	**0.0**	**1.1**	**6.8**	**9.5**	**3.4**	**124**
	%NL Dormancy [3]	61	40	59	83	43		78	39	51	32	49
	%NL Vegetation [3]	19	39	21	29	40		39	40	31	44	33
	%NL Senescence [3]	20	21	21	-12	17		-17	21	18	24	17
	Recycling	-	++	-	+	+	+	+	-	++		

4.2.4 *Biotic and abiotic recycling in humus (Ca, Mg and S)*

The release dynamics of Ca, Mg and S in NL showed 3 release peaks: during senescence, at the beginning of dormancy and a third in May. Arriving mainly in solid form (respectively, 92, 81 and 65% in DC and 94, 83 and 70% in RL (Table 13), mainly as biominerals for Ca and Mg) and being correlated with the water flux (r^2=0.5, Table 12), the first two peaks, common to those of Si, Al and Fe, could be explained in the same way: first, a departure of the soluble fraction, and then an action of the precipitation (Attiwill, 1968; Austin and Vitousek, 2000).

The May peak could be related to recycling mechanisms taking place in the humus. Indeed, Dincher et al. (2020a) showed that these elements could precipitate in humus around fungal hyphae or be taken up by teak amoebae. This reuse of elements could explain the decrease of NL in March and its increase around May, when these biominerals would start to be degraded, particularly under the effect of humus microorganisms (Katz and Lieth, 1974).

These microorganisms could also have an effect at the same time for C and N, also showing a peak of NL in May.

4.2.5 Effect of mineralization (C and N)

C and N had release dynamics curves with similar patterns to all other elements. Indeed, the C curve showed a first release peak during senescence, with another one at the beginning of dormancy (like Al, Fe and Si) and a last one in May (like Ca, Mg and S).

The N-curve showed a pattern with a peak during senescence and early dormancy. The pattern also showed a sharp decrease in release in April, just before the dormant period, and then increased again in May. As with the other elements, the peaks during senescence and early dormancy could be due to the departure of the soluble fraction on soil entry and precipitation.

These seasonal dynamics were consistent with those found in the literature, although the time step is more precise in our study (Jacob et al., 2009; Vesterdal, 1999).

C and N being the majority components of organic matter, the May peak could be related to this degradation of organic matter. However, there were differences in the amounts of elements: C was present in greater quantities than N, and N was fully released in April.

These elements can be mineralized by the microorganisms and output from the system in gaseous form. Thus, the consumption of these elements, and particularly N, by microorganisms could explain the April depression and the unrecorded releases in the cycles of these elements in humus (Table 13). This April gap could also be due to the tree's use of N via mycorrhizal colonization of humus for tree removal at the beginning of the growing season.

4.2.6 The special case of Mn

In our study site, the Mn is a special case. Indeed, although it came 7.8 $kg.ha^{-1}.y^{-1}$ in DC and 2.3 $kg.ha^{-1}.y^{-1}$ in RL, it did not come out of the humus (Table 13) (via solution, NL or particles). Mn could be strongly recycled within the humus, in particular by crystallizing around the hyphae of fungi (Dincher et al., 2020a; Pinzari et al., 2018). In addition, Mn showed an average residence time of 2 years in both soils, so it could leave the system in a non-quantified form (e.g., used by microorganisms).

5 Conclusion

This study presents the correlation between the seasonal release dynamics of elements in net leaching under humus at a monthly time step and the nutrient requirements of trees as a function of the seasons. This study showed that the majority of the elements were released during the senescence and dormancy periods, which represents a risk for the ecosystem of element loss, which can be leached more or less below the rhizosphere according to the retention capacity of the soil. In fact, all of the elements studied (C, N, P, K, Ca, Mg, Mn, S, Si, Fe, and Al) showed a release of more than 50% between November and February. For example, in DC, the release was more than 70% for Al, Fe, K and P and between 50 and 65% for Ca, Mg, S, Si and N. However, this seasonal dynamic and speed of release in solution was driven by several factors that induced differences between the release curves of the elements.

The first factor identified was the climate through the level of precipitation. It had a rather important impact on the dynamics of Al, Ca, Fe, Mg, S, Si and C, with r^2=0.5. Thus, the release peak during senescence/dormancy could come from the leaching of the most soluble fraction of these elements in the litter during intense fall/winter precipitation.

The second factor was the element form in litterfall and branch residues as soluble form (principally K and P), bound to organic matter (C and N) or in biominerals (Si and Ca), or as dust (Al and Fe). K and P were very soluble elements in solid inputs. This high solubility could explain the deficit in these elements during leaf fall: the labile fraction of K and P would be leached out at the canopy level when the leaves fall to the ground. The release peak following this deficit would correspond to the entrainment of the soluble fraction via winter precipitation. C and N were elements strongly bound to organic matter, so the peak in May could also be due to the high activity of microorganisms. The difference in the quantity of each element in the litter (C in greater quantity than N) could also explain the N deficit at the beginning of the growing season: it would be entirely used for the tree needs at that period. Inputs in solid form (e.g., dust or biominerals) also influence the release dynamics. Al, Fe arriving with the dust and Si and Ca with the biominerals, could explain the later release in winter in NL.

Recycling in the humus could also be a factor in the dynamics of release of the elements. Recycling mechanisms within the humus could take place for Si, Ca, Mg and S, delaying the release of the elements during the year, especially during May, at the beginning of the growing season by the microorganisms present in the humus.

A final factor could be the form of the release. C and N were highly mineralized and escaped from the recycling cycle in the form of gas. There were also elemental departures in the form of solid particles entrained in solution, which were not taken into account in the NL. This was particularly the case for Al, Fe and Si.

Acknowledgements

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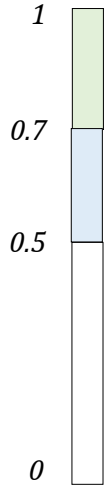
Supplementary Data

Table S1 : Matrix of determination coefficients between elements and precipitation in EC. $0.5<R^2<0.7$ are in blue, and $0.7<R^2$ are in green

	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N	C	Precipitation
Al												
Ca	0.40											
Fe	0.86	0.56										
K	0.27	0.13	0.27									
Mg	0.34	0.77	0.43	0.23								
Mn	0.07	0.07	0.10	0.04	0.06							
P	0.07	0.12	0.11	0.60	0.13	0.02						
S	0.16	0.66	0.28	0.09	0.39	0.12	0.19					
Si	0.76	0.66	0.81	0.37	0.50	0.08	0.23	0.42				
TN	0.10	0.29	0.14	0.08	0.17	0.02	0.14	0.48	0.23			
TC	0.54	0.65	0.63	0.49	0.60	0.10	0.29	0.43	0.78	0.23		
Precipitation	0.55	0.64	0.58	0.22	0.67	0.03	0.10	0.31	0.60	0.17	0.58	

Table S2 : Matrix of determination coefficients between elements and precipitation in RL. $0.5<R^2<0.7$ are in blue, and $0.7<R^2$ are in green.



	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N	C	Precipitation
Al												
Ca	0.31											
Fe	0.91	0.33										
K	0.12	0.11	0.13									
Mg	0.35	0.83	0.37	0.23								
Mn	0.01	0.00	0.01	0.03	0.00							
P	0.01	0.02	0.01	0.71	0.10	0.02						
S	0.09	0.70	0.13	0.08	0.54	0.01	0.02					
Si	0.71	0.61	0.75	0.28	0.62	0.00	0.08	0.38				
TN	0.01	0.30	0.02	0.07	0.25	0.04	0.06	0.36	0.14			
TC	0.44	0.74	0.43	0.29	0.69	0.00	0.11	0.45	0.79	0.18		
Precipitation	0.47	0.65	0.50	0.20	0.65	0.02	0.07	0.32	0.62	0.14	0.64	

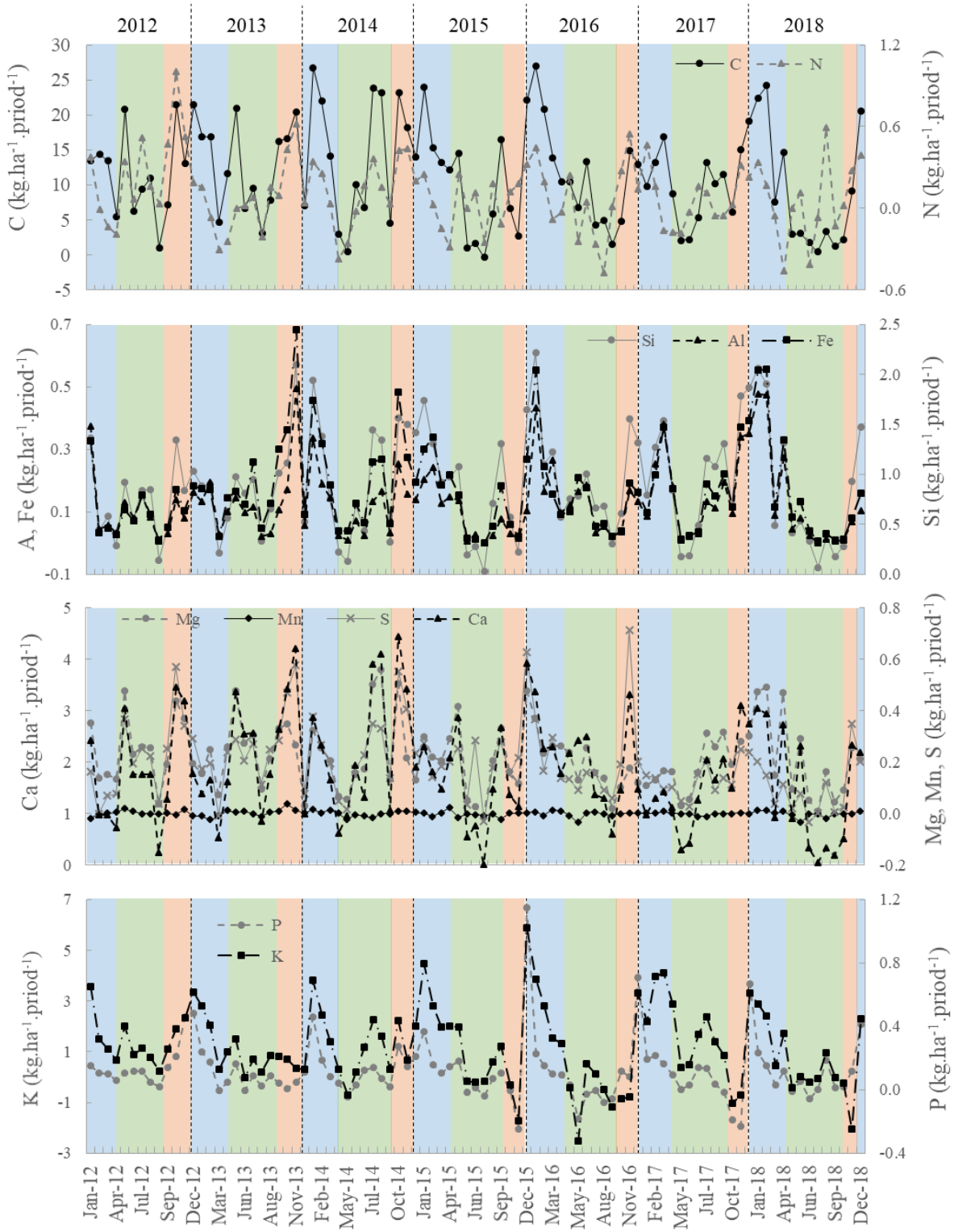


Figure S1 : Net leaching evolution from 2012 to 2018 in kg.ha-1.period-1 for the studied elements in the eutric cambisol. The blue stripes correspond to periods of dormancy (winter), the green stripes to periods of tree growth (spring and summer) and the orange stripes to periods of senescence (fall).

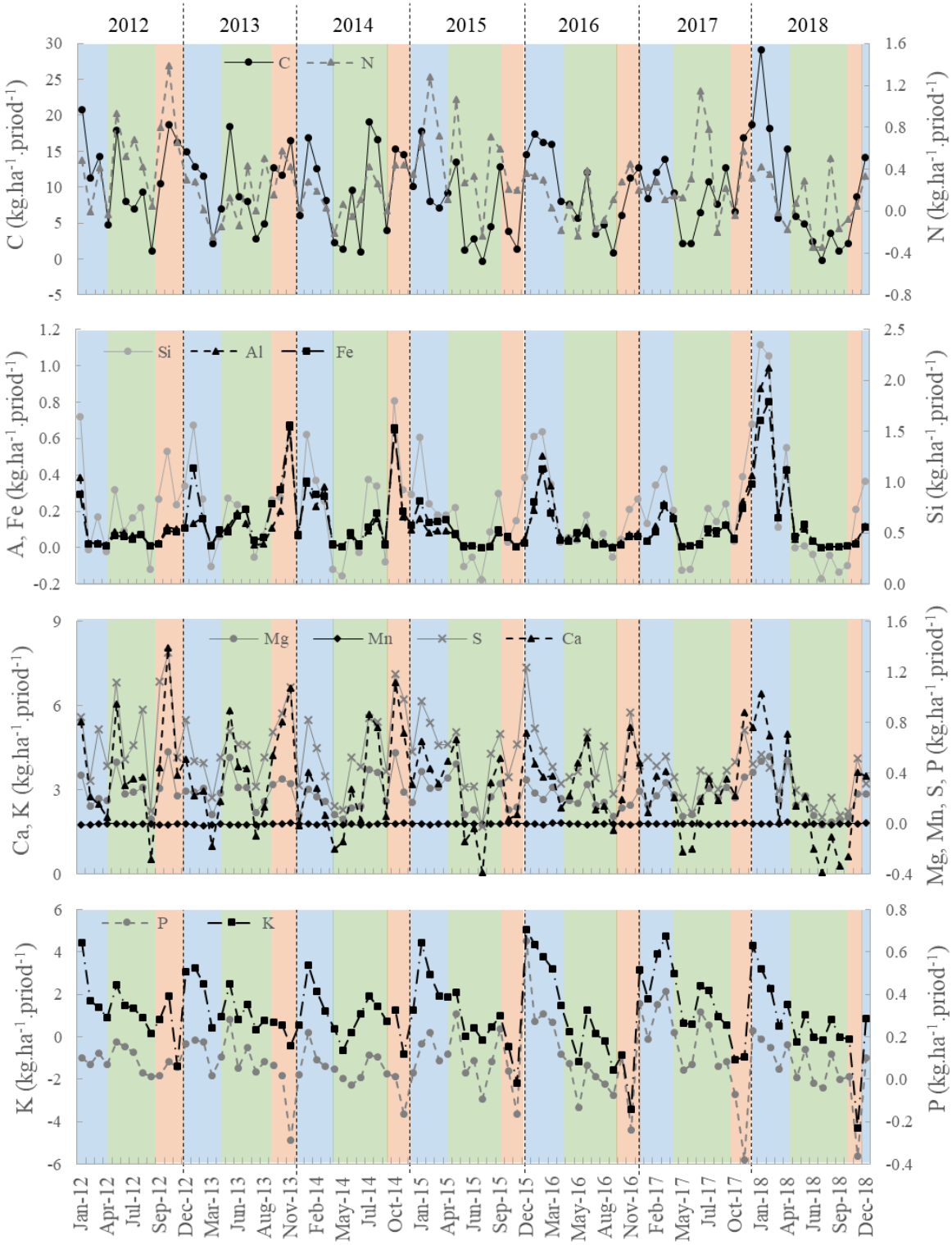


Figure S2 : Net leaching evolution from 2012 to 2018 in kg.ha-1.period-1 for the studied elements in the rendzic leptosol. The blue stripes correspond to periods of dormancy (winter), the green stripes to periods of tree growth (spring and summer) and the orange stripes to periods of senescence (fall).

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VI

Chapitre VI -

Litter decomposition and dynamics of elements in a beech forest: comparison between experimental approaches and humus results

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Ce chapitre présente une étude expérimentale de dégradation de litière par la méthode des sachets de litières. Il tend à répondre à la quatrième question de recherche : Une étude expérimentale à l'aide de sachets de litière peut-elle refléter la réalité de la dégradation de la litière en forêt ?

Résumé

Dans les écosystèmes forestiers où les entrées sont généralement faibles, la décomposition de la litière est une source importante de nutriments pour le sol. Cette décomposition a été largement étudiée pour des éléments comme C, N ou P mais beaucoup moins pour d'autres éléments comme K, Ca, Mg, Mn, Si, S, Al ou Fe. L'utilisation de la méthode par sachets de litière est la méthode la plus courante pour suivre cette dégradation au cours du temps.

Les objectifs de cette étude sont de (1) suivre sur plusieurs années le taux de dégradation de la litière sur différents sols, (2) observer l'influence de paramètres expérimentaux (variation pluriannuelle, taille de maille, séchage préliminaire des feuilles) sur la dynamique de la perte de masse et de la libération des éléments de la litière foliaire, (3) déterminer l'influence de différents types de sols sur ce taux de dégradation et (4) comparer les données obtenues avec cette méthode aux temps moyens de résidence obtenus sur le même site d'étude.

Pour répondre à ces objectifs, nous avons suivi la dégradation de la litière à l'aide de cinq expérimentations différentes utilisant la méthode des sachets de litières, de décembre 2013 à décembre 2020, en faisant varier la taille de maille des sachets (5mm vs. 1 cm) ainsi que le séchage préalable des feuilles de litière (feuilles séchées à 60°C vs. feuilles fraiches). Ces différents sachets ont été répartis sur 3 sols différents (Dystric Cambisol, Eutric Cambisol et Rendzic Leptosol) dans une même forêt de hêtres du Nord-Est de la France.

Nous observons une perte de masse totale lors de la dégradation des litières similaire sur les 3 sols. Cependant, le type de sol peut avoir une influence sur la perte d'éléments : selon la manipulation, la perte d'éléments peut être plus rapide sur DC que sur RL. Les paramètres expérimentaux testés n'influencent pas les pertes d'éléments lors de la dégradation et la dégradation est similaire entre les années. Enfin, la dynamique de libération des éléments est principalement régulée par leur forme (et leur labilité), ainsi que par leur localisation dans les feuilles.

En comparant les temps de demi-vie des éléments par la méthode des sachets de litières et les demi-temps de résidence des éléments dans l'humus, on en conclut que la méthode des sachets permet d'évaluer le taux de dégradation de la litière et la perte d'éléments. Cependant, les temps de résidences totaux des éléments ne sont pas similaires à ceux trouvés dans l'étude : cette méthode n'est donc pas assez précise pour accéder aux temps de résidence réels des éléments dans l'humus.

Litter decomposition and dynamics of elements in a beech forest: comparison between experimental approaches and humus results

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1 Introduction

Degradation of plant matter (litter) is the process of physical and chemical degradation of plant tissues (Tardif, 2013). The transformation of this dead organic matter results in the release of water, carbon dioxide and mineral elements into the soil in a plant available form (Begon et al., 2005; Chapin et al., 2002). This decomposition is a key process for ecosystems (Cadisch and Giller, 1997; Swift et al., 1979) as it is a major factor influencing the carbon cycle as well as the availability of nutrients and beneficial elements.

The rate of litter degradation as well as the biotic and abiotic factors controlling this process have been widely studied (Aerts, 2006). Abiotic factors correspond to climate, geography and soil properties. Biotic factors include the quality of the litter and the activity of the macro-, meso-, micro-fauna and microorganisms (Berg and McClaugherty, 2014). Macro-decomposers are involved in the first stage of litter degradation, which were then mainly decomposed by microorganisms. (Huhta, 2007; Persson et al., 1980). Indeed Schaefer, (1990) showed that 90% of the litter is mineralized by bacteria and fungi. This rate of degradation is also influenced by the recycling mechanisms that take place during the degradation of organic matter (biotic or abiotic secondary biomineral precipitation) (Dincher et al., 2020a). The second stage of litter degradation correspond to the mineralization of the litter, i.e. the chemical transformation of the fragments into basic inorganic molecules (ammonium, phosphate, carbon dioxide, water, etc.) (Cadisch and Giller, 1997; Swift et al., 1979).

The decomposition litter process results in a loss of litter mass due to the associated action of leaching of soluble cellular compounds, litter fragmentation and mineralization and reduction of reduction of litter mass consisting essentially of compounds that are difficult to degrade such as lignin or microbial cell walls (Chapin et al., 2002).

For beech litter, the losses vary from 50 to 90% depending on the study site and the duration of the experiment. For example for beech litter in Germany, Albers et al. (2004) observed a mass loss of 60% of the litter after 1 year of experimentation, whereas after 3 years of follow-up in Austria, Berger and Berger (2012) estimated a 53% loss of litter mass and that Hristovski et al. (2014), in a Macedonian forest, recorded a mass loss of 90% after 6.5 years of experimentation.

In experiments of litter degradation, the main elements studied are C, N or P (Albers et al., 2004; Bachega et al., 2016; Zeller et al., 2000), more rarely Ca, K and Mg (Jacob et al., 2009; Purahong et al., 2014; Santa Regina, 2000) or Al and Fe (Joergensen et al., 2009), but for other elements such as Si, S or Mn, there is a severe lack of data.

The majority of experiments to assess litter degradation have been carried out using the litterbag method for different species in different climates (Bachega et al., 2016; Gautam et al., 2019; Jacob et al., 2009; Santa Regina, 2000).

This method consists of putting a known mass of fresh or previously dried leaves, into porous bags with possible different mesh sizes. It allows to recreate as closely as possible in situ conditions involved in litter degradation. However, the interannual variability of the in situ conditions as well as experiment parameters, i.e., mesh size (Huhta, 2007) and preliminary drying of leaves, influence the rate of litter decomposition. Most often, studies conducted with the litterbag method compare the litter degradation of different plant species and focus on degradation dynamics and comparison between litter types. Few studies take into account the differences according to soil properties and the comparison with the rate of decomposition of the humus present on the site. In this study, litterbags of different mesh sizes (0.5 cm and 1 cm) and different leaf states (dry or fresh), a multi-annual monitoring (2 to 6 years) of mass and element losses (C, N, P, K, Mg, Ca, Mn, Al, Fe, Si and S) was carried out on three soil types under the same stand and climate conditions in order to limit the factors influencing litter degradation.

The aim of this study is: (1) to monitor over several years the degradation rate of litter on different soils, (2) to monitor the influence of experimental parameters (multiannual temporal variation, mesh size, preliminary drying of leaves) on the dynamics of mass loss and leaf litter elements release (3) to determine the influence of different soil types on this degradation rate and (4) to compare the data obtained with this method to the residence times obtained at the same study site.

2 Materials and methods

2.1 Experimental site

The study was carried out in the Montiers beech forest experimental site, built in 2011 and co-managed since 2012 by INRAE-BEF French National Institute for Agricultural, Food and Environmental Research – Biogeochemical Cycles in Forest Ecosystems research unit) and ANDRA (French National Radioactive Waste Management Agency). This site is located in north eastern France, in the Meuse department (48° 31′ 54″ N, 5°16′ 08″ E). The Montiers site is part of the research network AnaEE (Analysis and Experimentations on Ecosystems).

The climate is semi-continental (cold winters and hot summers) with a mean annual rainfall and temperature between 2012 and 2018 of 1081 mm and 9.4°C, respectively (from météo-France data). In winter, soil can temporarily freeze over a depth of a few centimetres and occasionally be covered with snow. During these study years, the years 2015 and 2018 were dry years and were marked by very low rainfall in summer with respectively 253 and 257 mm between May and September compared to the average of 7 years of study for the same period, i.e., 320 mm.

The Montiers site geology is composed of an underlying Tithonian limestone surmounted by acidic Valanginian detrital sediments. The site with a low slope has allowed the development of 3 types of soils on these different formations: Dystric Cambisol (DC) at the top, Eutric Cambisol (EC) downhill and Rendzic Leptosol (RL) downslope, directly on the limestone (FAO, 2006). RL and EC have a eutrophic mull humus and DC has an acidic mull humus. No roots were found in the humus of the soils. A schematic representation of the soil profiles and their location is presented in (Turpault et al., 2019). The abundance of earthworms observed in the humus also highlights the high biological activity of the humus in the three soils, especially in the RL.

A beech forest covered the Montiers site. The tree of this forest have the same age (~50 years old in 2010) and the same management. The stand is mainly composed of beech (89%) and other species (11%), i.e., sycamore maple (*Acer pseudoplatanus*), ash (*Fraxinus excelsior*), pedunculate oak (*Quercus robur L.*), European hornbeam (*Carpinus betulus L.)* and wild cherry (*Prunus avium*). Since at least 1830, the site of Montiers has been occupied by a beech forest. The management was carried out by the ONF (National Forest Office) since the 1960s with a cut every 7 years.

On each soil, three experimental plots were established and replicated three times each. Each replicate (2500 m^2) was equipped to collect above- and below-ground solutions at different depths, soil at different depths, organic horizons (8 groups of 3 lysimetric collectors per replicate), litterfall (4 gutters per replicate), tree compartments

(stem flow and throughfall, 6 collectors per replicate) and for the standing above-ground (6 litter traps per replicate) and belowground biomasses.

In addition, a 45 m high flux tower was placed within the site (close to plot DC) to collect rainfall and atmospheric deposits. For more information see Calvaruso et al. (2017), Kirchen et al. (2017) and Turpault et al. (2019).

2.2 Litterbag

Beech leaves were sampled by applying a net on the forest floor in autumn 2013 and 2018 in the non-instrumented place in the three plots.

All experimental conditions are presented in Table 14. The litter material sampled in 2013 was dried at 60°C and litter components other than beech leaves were separated by hands. To test period effect, leaves sampled in 2013 were used for 3 manipulations: M1, M2 and M3 whose start has been delayed by one year to test the multi-year effect and potential annual climate effect. For M1, 2.0 g of leaves were placed in 5 mm mesh litterbag (20 x 20 cm) and the litterbags were collected between December 2013 and December 2016. For M2 and M3, 6.0g of leaves were placed in the same litterbag and collected between December 2014 and June 2018 for M2 and between December 2015 and January 2018 for M3. A total of 351 litterbags were placed in the various plots: 99 for M1, 144 for M2 and 108 for M3.

To test the effect of dry and mesh bag size, leaves sampled in 2018 were separated for two manipulations: M4a and M4b. For M4b, the litter was handled as in 2013. In the 1 cm mesh litterbag (22 x 22 cm), 10 g of dried leaves were placed. A total of 72 litterbags (24 per plot) were placed in the various plot and they were collected between November 2018 and December 2020. For M4a, 40 g of fresh leaves were placed in 126 litterbags (42 per plots, 1cm mesh and 22 x 22 cm). Litterbags were collected between November 2018 and December 2020.

Table 14 : Experimental conditions of 5 manipulations studies

	Leaf	leaf weights in litterbag	Litterbag dimension	Mesh size	Start	End	Total litterbag
M1	Dried at 60°C	2 g	20 x20 cm	5 mm	December 2013	December 2016	99
M2	Dried at 60°C	6 g	20 x20 cm	5 mm	December 2014	Janvier 2018	144
M3	Dried at 60°C	6 g	20 x20 cm	5 mm	December 2015	Janvier 2018	108
M4a	Fresh	40 g	22 x 22 cm	1 cm	Novembre 2018	Décembre 2020	126
M4b	Dried at 60°C	10 g	22 x 22 cm	1 cm	Novembre 2018	Décembre 2020	72

2.3 Analyses

Samples were taken after 6, 12, 18, 24, 30, 32 and 36 months for M1; after 6, 12, 18, 24, 30, 36, 42, and 48 months for M2; after 6, 12, 18, 24, 30 and 36 months for M3; after 3, 5, 6, 9, and 12 months for M4a and after 3, 6, et 12 months for M4b. At each sampling, six litterbags were taken from each plot. The leaves were oven-dried at 60°C and were hand-cleaned leaf by leaf very carefully to remove most of the soil present. Then, the leaves were ground and encapsulated for analysis. The total Ca, K, Mg, P, Al, Mn, S, Si and Fe contents were assessed by X fluorescence using an X fluorescence sequential spectrometer S8 TIGER 1 kW (Bruker). The total C and N was assessed using a CHN analyser (EA/NA 1110, THERMOQUEST).

2.4 Calculation

2.4.1 Titanium model

Despite the careful cleaning of collected leaves, some soil particles could contaminate the organic horizon. The percentage of soil remaining in the organic horizon can be calculated using the equation described by Turpault et al. (2018), taking into account the titanium content:

Soil % = [(TiHb - TiHp) / (Tis - TiHp)]

Where TiHb is the concentration of Ti in the organic horizons, TiHp is the concentration of Ti in the pure organic horizons, and TiS is the mean concentration of Ti in the 0–5 cm horizon of soil for each soil. To obtain the actual content of each element in the organic horizons, the fraction introduced by soil contamination was deducted from the total.

In this studies we observed that the soil contamination of the organic layer was very low, i.e., < 2 %.

2.4.2 Litter degradation rate

The decomposition rate of the litter was calculated using the model proposed by Bärlocher, 2005:

$Mt = M0.e^{-kt}$

With Mt is the remaining mass or remaining mass of element at t, M0 the initial mass of the litter or initial element mass in the litter (here 100), k the decomposition constant and t the duration of exposure of the litterbags in the field in years.

2.4.3 Half-time of litter mass and litter element amount

For the mass or the element amount, the half-life (time for the loss of 50% of the litter mass or litter element amount) was calculated from the decomposition rate equation:

$t = -(\log (Mt / M0) / k)$

Thus, for $t = 0.5$

$Mt = 0.5 \times M0$ and $T(50\%) = -\log (0.5) / k$

With T(50%), the half-life of losses

2.4.4 Half mean residence time

To be able to compare T(50%) with the residence times of the elements calculated in Dincher et al (2020a), these residence times were divided by two :

½ MRT = MRT / 2

with MRT = Mean residence time

2.4.5 Statistical analyses

The normality of the distribution was confirmed using the Shapiro-Wilk test. As our data did not follow a normal distribution, the non-parametrical Kruskal-Wallis and Wilcoxon tests were performed to determine: (i) the significance of differences for accumulated mass loss and remaining element content and (ii) the significance of differences for ½ MRT and the half-life of elements. The comparison was made between soils within the same manipulation for (i) and between variable for the same element for (ii).

3 Results

3.1 Mass loss

3.1.1 Comparison of mass loss between manipulations

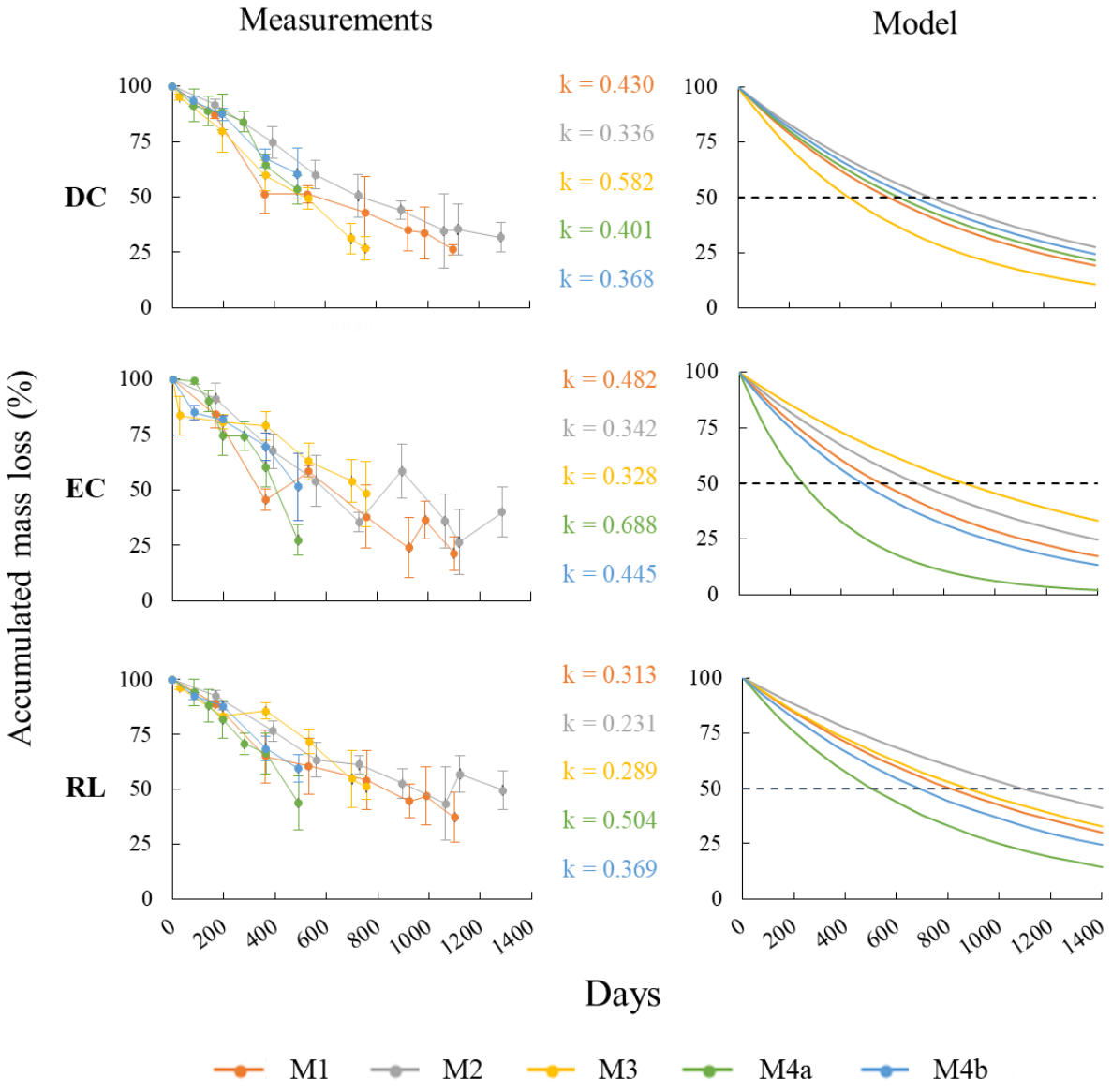

Figure 31 : Evolution of the mass in percentage of loss in comparison with the initial mass as a function of the duration in days of each manipulation and degradation constant k for the 5 experiments. On the left the measurements taken in the field and on the right the curves according to the model Mt = M0.e^{-kt}. M1 in orange, M2 in grey, M3 in yellow, M4a in green and M4b in blue. The standard deviation is given by the coloured bars corresponding to the targeted manipulation. The term k is a factor related to the rate of litter degradation. The higher it is, the faster the degradation. The colour of k corresponds to the associated manipulations.

The follow-up of litter mass loss showed a general trend of decreasing for all manipulations and for all soils (Figure 31).

In DC and RL, the mass loss appeared similar for the first 200 days between all manipulations (Figure 31). For EC, litter degradation was faster in M4b and M3 than in M4a and M2 in the same time period.

In DC, the fastest litter degradation rate is observed in M3 with k=0.582 and a mass loss of 73% in 756 days. Then litter degradation rate decreases as follows, M1, M4a, M4b and M2 with k ranging from 0.430 to 0.336. In EC, litter degradation was faster in M4a than the other manipulations with k=0.688 and a loss of mass of 73% in 490 days. Litters in M1, M4b, M2 and M3 were degraded more slowly with k ranging from 0.482 to 0.342. As in EC, litter in M4a was degraded faster than other manipulations with k= 0.504 and a mass loss of 56% in 490 days. The litters with the lowest degradation rate were in M4b, M1, M3 and M2 with a k between 0.369 and 0.231.

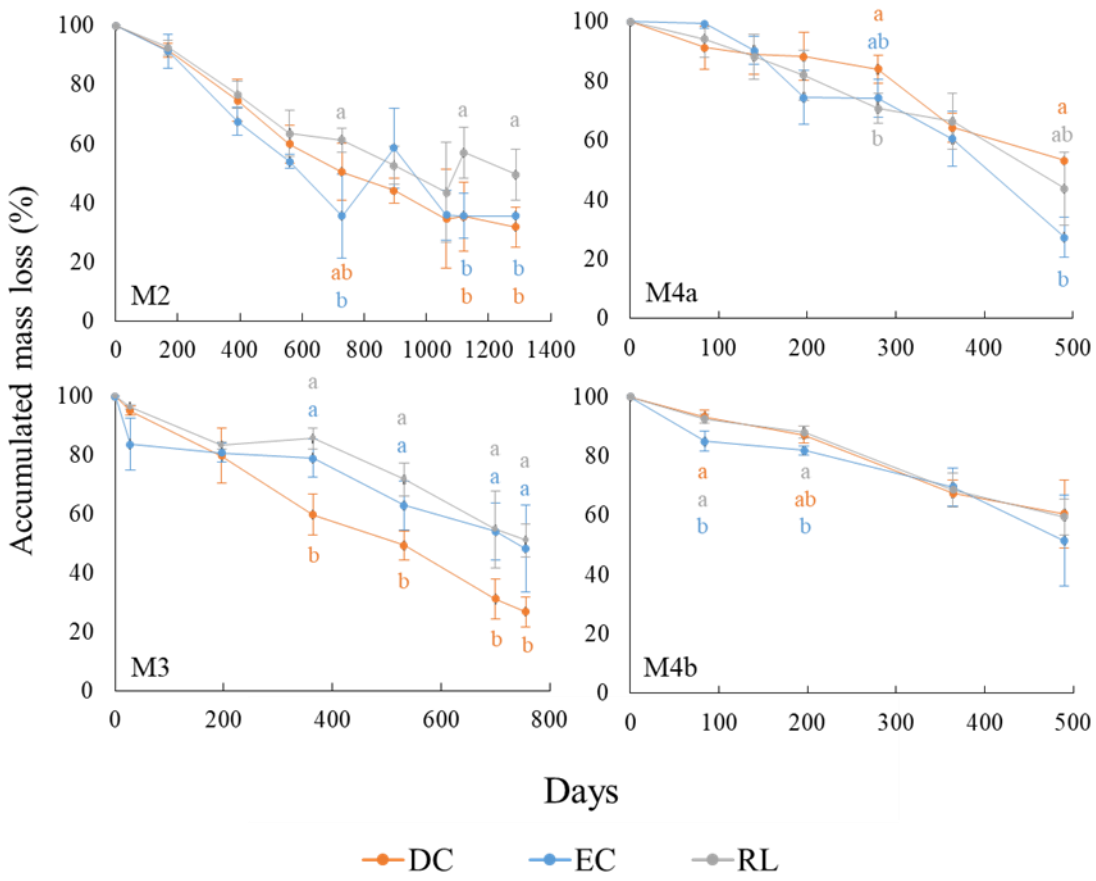

Figure 32 : Evolution of the mass in percentage of loss in comparison with the initial mass according to the duration of the manipulation for the different soils in function of the different manipulations. DC: orange, EC: blue, RL: grey. The standard deviations are given by the coloured bars. Values with different letters are significantly different between soils according to the Kruskal-Wallis test at the threshold P value of 0.05.

3.1.2 Comparison of mass loss between soils

By comparing each soil by experiment, significant differences (p-value<0.05) between these soils in terms of mass loss were observed (Figure 32). For M2, the differences in mass loss appeared after 728 days of incubation where litter in RL would tend to be degraded slower than in DC and EC. In M4a, this difference was small throughout the study, where DC generally contrasted with the other soils. Litter degradation was faster in the early stages of M4b in EC compared to those in DC and RL. Finally, in the M3 manipulation, there was a clear difference in litter degradation rate between DC and EC/RL.

3.2 Element released during litter degradation

3.2.1 Comparison of element loss between manipulations

Figures 33 and 34 show the evolution of the elements through the different manipulations in DC and RL. Depending on the elements, the trends range from a mass decrease of elements to a stabilization or even increase.

For all manipulations, K and P showed an abrupt drop in the first 200 days of degradation and then declined more gradually over time. In RL, P tended to stabilize at about 60% of the initial mass. Mg also showed a greater slope at the beginning of the degradation and then stabilized at about 20% and 40% of the initial mass in DC and in RL, respectively. C and Ca had a more linear slope with a tendency to a plateau at the beginning of the degradation (C in DC and Ca in RL). The release of Mn in DC was lower than for the preceding elements, however, this loss was much more variable and seemed, for M2, to stabilize around 45% of the lost mass. In RL, the slope of degradation at the beginning appeared to be greater in the first 100 days and then stabilized. For M2, all of Mn was released in 1288 days.

Al and Fe showed different behaviours depending on the manipulations. The percentage of these elements increased in M4a and M4b and decreased in the other manipulations. The Fe was totallylost in approximately 363 days in DC and about 756 days in RL for M1, 2 and 3. Al showed a more irregular evolution for these manipulations. N also showed some differences between M4a and M4b and the other manipulations at the beginning of the degradation: M4a and M4b tended to increase while the others tended to decrease slightly (RL) or remain stable (DC).If in DC tended to remain stable with a slight decrease in DC and an increase towards the last days of handling M1 and M2. S remained stable regardless of manipulation and regardless of soil.

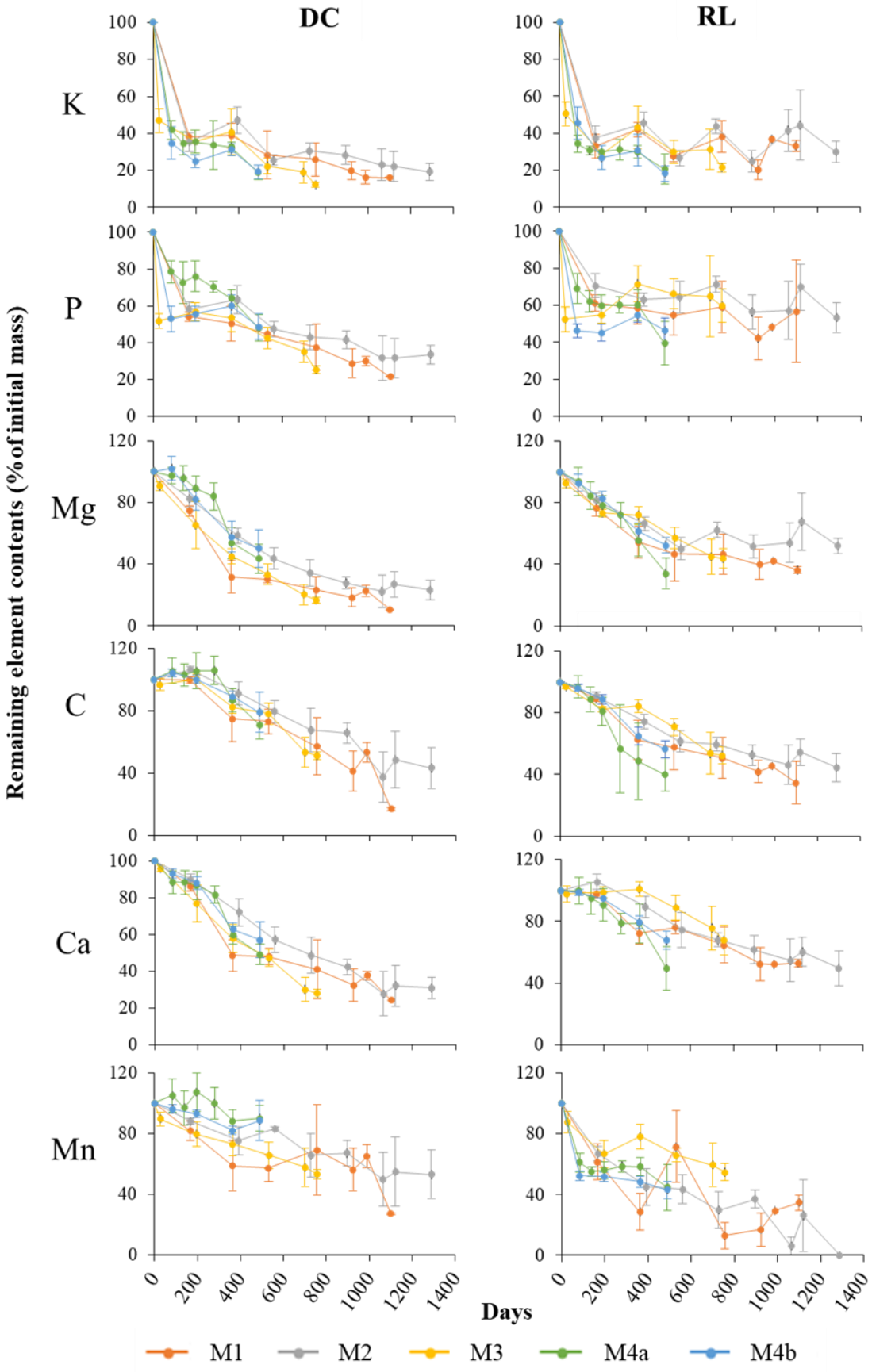

Figure 33 : Evolution of the release of K, P, Mg, C, Ca and Mn as a percentage according to the duration in days of each manipulation. M1 in orange, M2 in grey, M3 in yellow, M4a in green and M4b in blue. The standard deviation is given by the coloured bars.

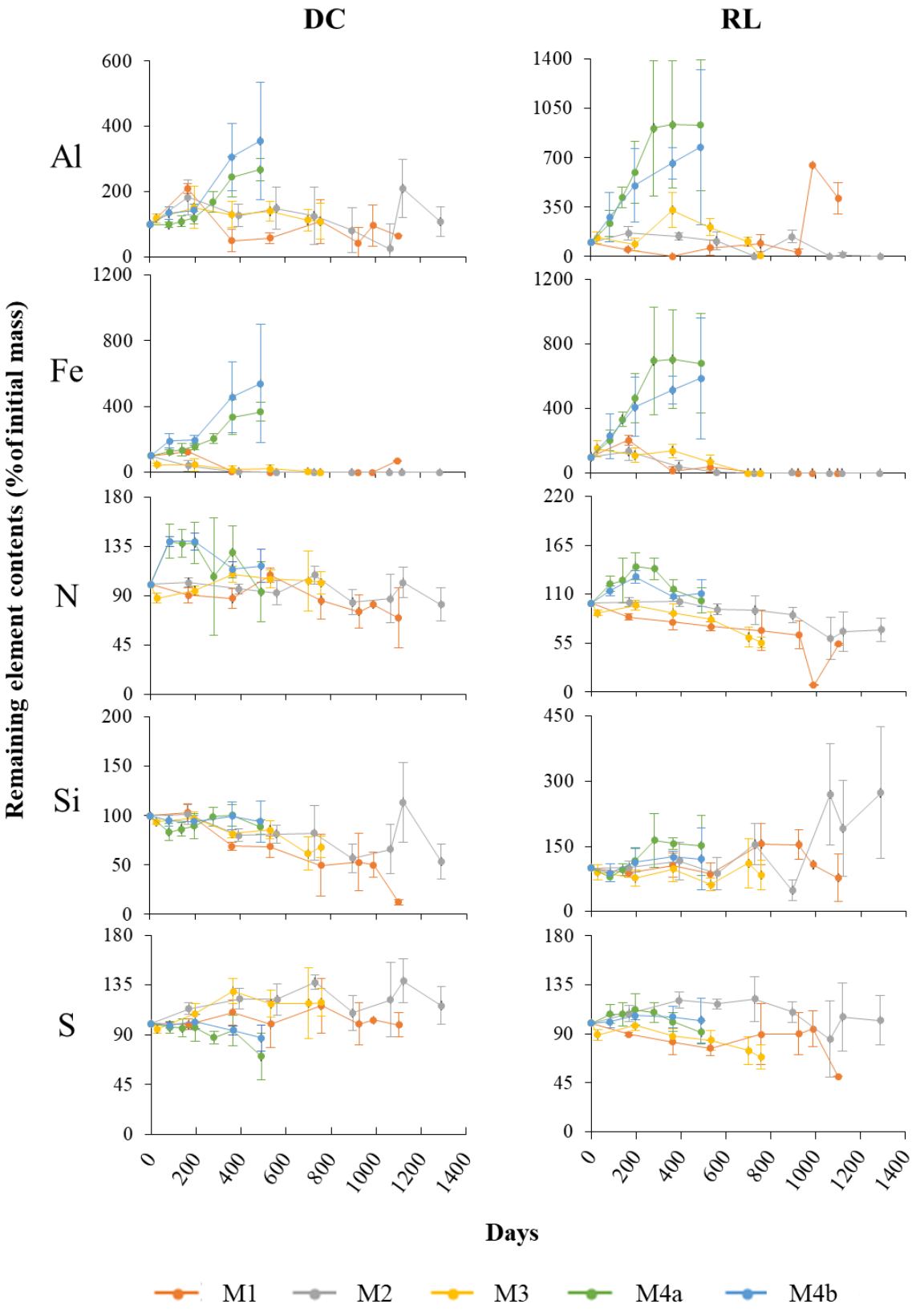

Figure 34 : Evolution of the release of Al, Fe, N, Si and S as a percentage according to the duration in days of each manipulation. M1 in orange, M2 in grey, M3 in yellow, M4a in green and M4b in blue. The standard deviation is given by the coloured bars.

3.2.2 Comparison of element release between soils

By comparing the evolution of litter element content between soils according to manipulations, it appeared that K, C, Ca, Fe, N and S had a similar degradation rate (p-value<0.05) between the 3 soils studied. Al, Mg, Mn, Si and P showed differences between the soils according to the manipulations (Figure 35).

The degradation of Al differently evolved between soils in M1, M2, M3 and M4a. DC was lower EC in all manipulations and RL in M3 and M4b. In M2 and M3 the degradation of Mg was different between soils, especially at the end of degradation and as with Al, DC was in opposition to EC and RL. For Mn, the difference in degradation between soils was more pronounced, especially in M4a and M4b where the three soils had very different percentages of remaining mass. In M1 and M2, DC and EC were in opposition to RL. The Si showed different degradation rates between soils in M1, M2, M4a and M4b, in each manipulation EC differed from DC and RL. Finally, P showed differences between soils in all manipulations. In M1, this difference appeared at the end of the manipulation where EC and RL were different from DC. In M2, RL was different from EC and DC; in M3, DC was different from RL and EC; and in M4a and M4b, EC was different from DC and RL.

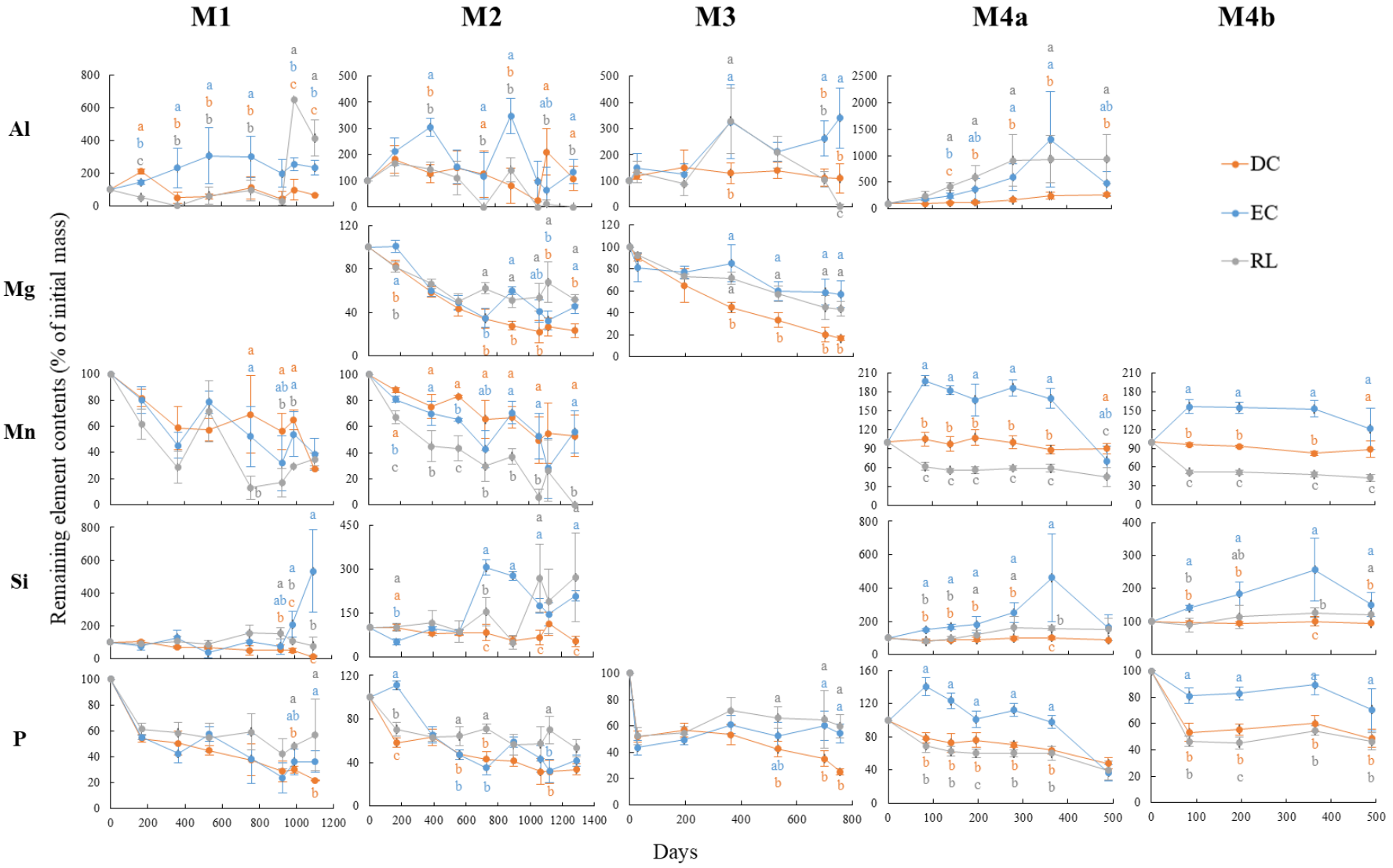

Figure 35 : Evolution over time of the remaining element contents in % of initial mass in the litter according to the manipulations. DC: orange, EC: blue, RL: grey. The standard deviations are given by the coloured bars. Values with different letters are significantly different between soils according to the Kruskal-Wallis test at the threshold P value of 0.05.

4 Discussion

4.1 Litter degradation rate

The litter degradation rate, obtained in the different manipulations though the follow-up of litter mass, can be compared using the decomposition constant k. In our study, the mean value of k varied between soils: for DC k= 0.423, for EC k = 0.457 and for RL k = 0341 resulting for the Montiers site to a mean value of k of 0.407. This value is close to that found by Jacob et al. (2009) in a central German beech forest (Hainich National Park) on Luvisols formed on limestone with mull humus type (k= 0.400) but is higher than that measured by Joergensen (1991) in a beech forest (Göttingen Forest), Germany) on a rendzic leptosol with moder humus type (k=0.209). This difference is in agreement with the observation of (Ponge, 2003) according to which mulls have a faster turnover than moders

In the experiments with larger-mesh litterbags (M4a and M4b), for all soils, k was larger than in the other experiments ($0.368 < k < 0.688$). Two hypotheses can explain this result: (i) an easier access to organic matter of a great diversity of decomposers would accelerate litter degradation and (ii) due to the larger mesh size, the amount of matter transported outside the bag increased (Huhta, 2007).

Although M2 and M3 had suffered dry years during the incubation period (in 2015 for M2 and in 2015 and 2018 for M3), no change in litter degradation rate was observed in the three soils. Moreover, the difference in mass loss between the soils was minimal even if on DC, this loss seems faster than on RL. This could be related to the hydric stress always present in RL and only present in DC during dry years (2015 and 2018) (Kirchen et al., 2017).

4.2 Element release

4.2.1 Evolution of element release during litter degradation

Some elements may be bound to organic components of litter such as proteins, carbohydrates, lipids and nucleic acids (e.g. N) or they may be present in organic tissues as inorganic components in soluble or biominerals (K and Ca (Berg and Staaf, 1980; Krieger et al., 2017) ; Si (Puppe et al., 2016; Turpault et al., 2018)). As a result, elements are released in different ways during the decomposition of litter through degradation of the organic components by soil decomposers, by physical leaching of the elements in soluble form (Staaf and Berg, 1982) or by dissolution of primary biominerals (biominerals arriving through plant entrances) and secondary biominerals (biominerals precipitated during leaf degradation) (Dincher et al., 2020a).

4.2.1.1 Case of K, P and Mg

In our study, K, P and Mg showed a strong release at the beginning of the degradation (in particular for K and P) then there was a decrease or even a stabilization. This trend has already been observed in several studies on *Fagus sylvatica* (Berger and Berger, 2012; Jacob et al., 2009; Purahong et al., 2014) but also in studies on other species such as *Pinus sylvestris* (Jasińska et al., 2019) and *Quercus pyrenaica* (Santa Regina et al., 1997).

K and Mg are only partially integrated into the structure of plant tissues (e.g. Mg is a constituent of chlorophyll). Moreover, in leaves, these elements are mainly in soluble form (71% for K and 40% for Mg for all soils) (Dincher et al., 2020a). They are thus more sensitive to foliar leaching losses as confirmed by a fast release during the first phase of the decomposition process, followed by a more irregularly and more slowly release (Gosz et al., 1973; Staaf and Berg, 1982).

In most studies, P, and N, have a rather stable dynamic in the early stages of litter decomposition and decline thereafter (Hristovski et al., 2014; Li et al., 2007; Purahong et al., 2014). However, in our study, P followed the same dynamics as K: the release of P observed later in literature appears earlier in our study. This behaviour could be due to the high solubility of P in the leaves of the Montiers site. This is corroborated by Dincher et al. (2020a) who estimated that P is 64% under soluble form in the leave of this site.

4.2.1.2 Case of C and Ca

In the experiments, C and Ca showed a steady decrease. Many authors including Albers et al. (2004), Jacob et al. (2009) and Vesterdal (1999), also observed this pattern for C, regardless of the study site parameters. Indeed, as a major component of organic matter, the C degradation is strongly linked to that of organic matter and will be strongly influenced by both soil organisms and physical degradation factors.

Interestingly, the release of Ca from litter in our study is similar to those found in other studies with different tree species, i.e., litter from *Fagus sylvatica* (Jacob et al., 2009; Joergensen, 1991) or from other species such as *Acer rubrum, Cornus florida* or *Quercus prinus* (Blair, 1988) or *Quercus pyrenaica* (Santa Regina et al., 1997). Ca is a structural component of plant tissues (such as cell walls) and is present as calcium oxalate crystals in leaf veins. In the Montiers this Ca form represent 37, 39 and 33% of the total calcium in the leaves of the respective litter of DC, EC and RL (Turpault et al., 2019). Ca will tend to be released at the same time from the degradation of leaf cell tissues and will therefore follow the same dynamics of organic matter and C degradation.

4.2.1.3 Case of Mn

Most of the time in the literature, the degradation curve of Mn is similar to that observed in our DC soil (Jasińska et al., 2019). However, few studies show these curves directly and simply discuss the degradation of the measured Mn (Vesterdal, 1999). The

stability or even the accumulation of Mn enduring the last phase of the experimentation could be explained by the production and activity of manganese peroxidase by hyphae, which is an enzyme necessary for the degradation of lignin. (Hatakka, 2001; Trum et al., 2015).

On RL, Mn has the same dynamics as K, P and Mg. This decrease was already observed in the study of Jasińska et al. (2019), but more generally, Mn shows a more steady decline (as in DC). This strong decrease at the beginning of litter degradation could be a site effect: in leaves, Mn is more soluble in RL than in DC (24% vs 35% respectively).

4.2.1.4 Case of Al and Fe

In our study Al and Fe showed two different behaviours according to the manipulations: in M1, M2 and M3, Al and Fe seemed rather stable and in M4a and M4b their contents increased strongly. The stability of Al and Fe in the litter of M1, M2 and M3 is consistent with the observations of Berger et al. (2015) and Schlesinger (2005). This stability of Al and Fe could result from the absorption of Al and Fe by the plant litter during the formation of organo-mineral complexes. The increase of Al and Fe was observed by several authors including Joergensen et al. (2009) and Palviainen et al. (2004) on different species including *Fagus sylvatica*. Three hypotheses could explain this increase: (i) the colonization of plant debris by microorganisms capable of accumulating large amounts of Fe in their cells (Joergensen et al., 2009), (ii) as the decomposition progresses, a large quantity of organo-mineral complexes would be formed (Donisa et al., 2003) and (iii) incorporation of soil via biological (e.g. earthworms) or through sampling (Joergensen et al., 2009), even if we had used a mathematical model based on Ti to eliminate the fraction of soil removed.

4.2.1.5 Case of N, Si and S

During the various manipulations, N, Si and S showed a stable to a slightly decreasing trend. The release of N is well documented while the monitoring of Si and S is still poorly documented. Whatever the site or the species studied, the trend of N always shows a stable to slightly decreasing trend (Berger et al., 2015; Blair, 1988; Purahong et al., 2014; Santa Regina et al., 1997; Zeller et al., 2000) which can be divided into two stages: an accumulation/immobilisation phase followed by a release phase. One explanation for the phenomenon of temporary accumulation of N is an increase in the bags of hyphae carrying N and its microbial immobilization (Berg and Staaf, 1987; Gosz et al., 1973). In addition to the absorption of atmospheric ammonia, atmospheric precipitation and dust, several factors influence the dynamics of N during litter degradation, such as pollen, insect, vertebrate and invertebrate droppings or other green wastes (Gosz et al., 1973; Melillo et al., 1982; Zeller et al., 2000).

Flux of Si in the litter is poor documented. The study of Joergensen et al. (2009) presents similar results to those found for Si in our study. Si in leaves is mainly in insoluble

form (98% (Dincher et al., 2020a)) in solid or amorphous biominerals (Dincher et al., 2020a; Turpault et al., 2018). It can also be present in secondary biominerals precipitated around the hyphae that colonize the leaves of the litter (Hees et al., 2004). These primary and secondary biominerals, which are more complex to degrade, could strongly influence the release dynamics of Si and give this stable aspect to its decomposition curve.

As for Si, the release dynamics of S was very little studied in the literature (Berger et al., 2015; Berger and Berger, 2012). We can make the following assumptions: (i) S is as much taken up by microorganisms as it is brought by other inputs than litter (e.g. atmospheric particles, (Lequy et al., 2014)) and (ii) is immobilized by the hyphae colonizing the leaves.

4.2.2 Manipulation and soil effect on the degradation

In our study, the manipulation parameters (mesh size and preliminary drying) was no significant over the long term except for Al and Fe in M4a and M4b. Globally, at the beginning of the manipulation, the loss of mass and element release were more important in M4a and M4b. This difference could be induced by the mesh size, either because they allow the penetration of larger decomposers than in M1, M2 and M3 (Edwards and Heath, 1963) or by the loss of fragments of material through the meshes (Huhta, 2007) or by a higher proportion of soil in the litter..

The effect of the soil varies according to the elements. Most often, DC contrasts with RL and EC varied widely. By their nature, DC is a richer soil in elements than RL. (Calvaruso et al., 2017) and therefore more biologically active, so degradation is faster on this type of soil.

4.3 Half-life of the elements and mean residence time

Figure 36 presents the half-life times in M1, M2 and M3 and the average half-lives (½ MRT) calculated in Dincher et al. (2020a). This is the first time that litterbag degradation times have been compared to residence times on the same site for a set of elements. Although the data are not fully comparable, as we are comparing litterbag leaves to humus (supplied by leaves, small branches, fruit and slash), the half-life times and ½ MRT are statistically similar in all cases. This similarity can be explained by the the fact that leave compartment represent between 70 and 75% in humus and this compartment is the more degradable. However, the mean total residence times are not similar to those obtained in the experiments. The study of litter alone doesn't allow the calculation on a specific residence time of the elements in the humus.

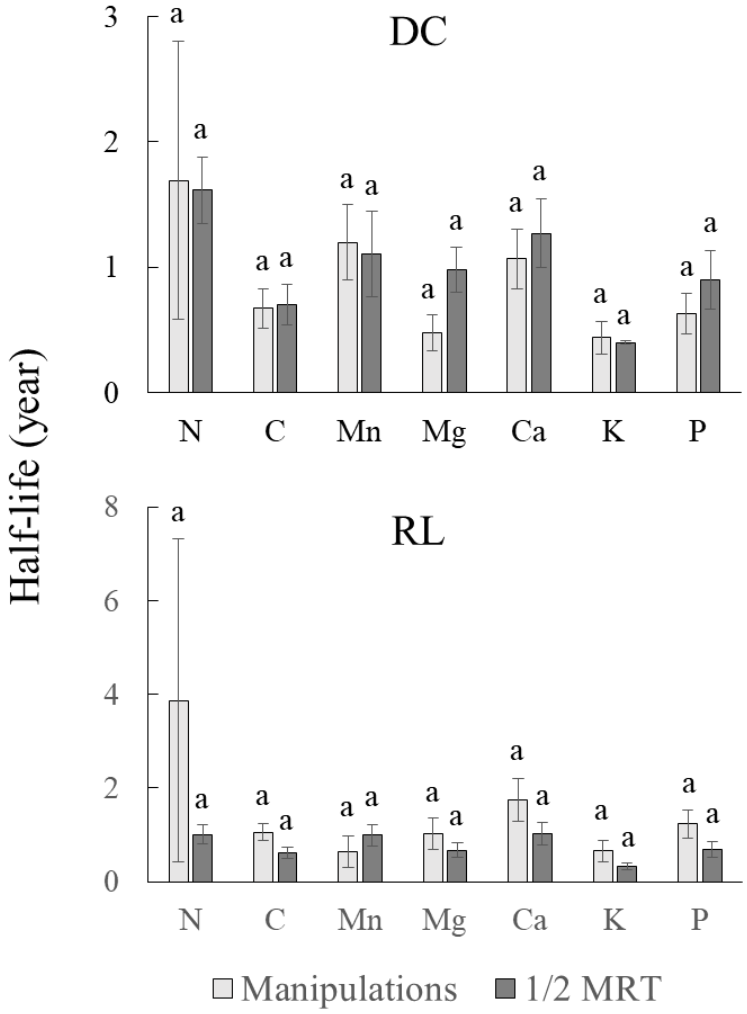

Figure 36 : Comparison of half-life times of elements in M1, M2 and M3 and ½ mean residence time (MRT) of elements of the Montiers site for DC and RL. In light grey are the experimental values and in dark grey are the MRT values. The standard deviation is given by the vertical black bars. The letters represent the significant differences for the same element between the ½ MRT values and the half-life time in the manipulations (p-value < 0.05).

5 Conclusion

This study determined for three soils with contrasted properties variations of the degradation rate of litter leaves (i.e. mass and element content) through litterbag experiments, as well as the influence of different experimental parameters (multiannual temporal variation, mesh sizes and leaf drying) on the dynamics of litter degradation. These measurements allowed us to compare the half-life of elements in humus obtained experimentally and by in situ measurement of mean residence times. Overall, the loss of litter mass is similar between soils in all experiments (i.e. for M1, k between 0.313 and 0.430).

Except for the effect of mesh size on the dynamics of Al and Fe, the experimental parameters tested (i.e. date, pre-drying, mesh size) do influence the element losses by different ways, i.e., access to organic matter, moisture… The influence of the soil was little. However, on a few manipulations for mass and given elements, the degradation of litter leaves on DC was faster than on RL. This effect could result from the difference in element content of the two soils (DC > RL except for Ca) or from the water stress state present, with RL being under water stress in all years and DC only in dry years. However, although 2015 and 2018 were dry years, the dynamics of litter degradation do not seem to have been influenced. The dynamics of the elements is mainly regulated by their forms and their location in the leaf. Four groups of elements can thus be distinguished according to their lability: (i) K, P and Mg, which are elements with high solubility in leaves. They are released rapidly, due to their leaf-soluble form, and show a strong release at the beginning of the experiment and then a stabilization over time; (ii) C and Ca are elements bounded to organic matter because C is the main component and, Ca is a constituent of plant tissues and is in the form of calcium oxalate trapped in vein cells. Their release rates are similar to that of organic matter and quite rapid; (iii) Mn, which is more soluble in RL than in DC, has a dynamics similar to K in DC and similar to C in RL; (iv) N, S and Si show a stable to slightly decreasing trend. Although widely used by microorganisms, this trend could be explained for N by its transport and immobilisation by hyphae or by the absorption of atmospheric ammonia, atmospheric precipitation and dust. Si, mainly in insoluble form in solid biominerals or amorphous form in leaves, is less easily degradable. It is taken up in secondary biotic and abiotic precipitation in humus, which may explain its stationary state.

These element dynamics are similar to those in the humus of the soils studied. Indeed the half-life times of the elements are similar in experiments with small-mesh litterbags and the mean residence times in humus. In our study site, for mull type humus, the litterbag method provides an unbiased view of the rate of litter degradation (i.e. loss of mass and elements) for half-life times.

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VII-

Chapitre VII -

Synthèse et conclusions générales

Ce chapitre présente une synthèse générale des données et résultats obtenus dans cette thèse. Il vise à faire le lien entre les résultats des chapitres 3, 4, 5 et 6 afin de répondre aux questions scientifiques posées et aux hypothèses de travail.

1 Dynamique et coévolution des éléments dans des mulls

Cette section présente dans une première partie les différents résultats acquis au cours de cette thèse ainsi que de nouveaux résultats obtenus par une approche fondée sur la corrélation entre les éléments dans les flux d'entrées (poussières, litière et solutions) et de sorties de l'humus. Ces données seront ensuite synthétisées dans une seconde partie qui permettra de statuer sur la dynamique des éléments et leurs relations dans l'humus (ou dans l'ensemble des couches holorganiques). Il est à préciser que dans cette partie, nous ne parlerons que des deux sols les plus contrastés (DC et RL). Nous ne commenterons pas les corrélations existantes avec Al et Fe qui présentent de grandes variations dans les flux étudiés.

1.1 Synthèse des résultats

Les Figures 37, 38, 39 et 40 présentent les différentes entrées et sorties de chaque élément, leurs stocks et leurs temps de résidence dans les humus de DC et RL. Les Tables 15 et 16 répertorient les flux totaux d'entrées et de sorties des éléments dans l'humus, leur distribution dans les entrées et les sorties (en pourcentage) ainsi que les différentes observations réalisées au MEB, les corrélations entre éléments dans les flux et les périodes de production d'éléments dans la solution de sortie.

Dans DC, les bilans entrées/sorties de K, P, S et Mg sont équilibrés avec des temps de résidence allant de 0.7 an pour K à 1.7 an pour Mg (Figure 37, Table 15). Pour K et P, le flux d'entrée principal correspond à la fraction soluble des végétaux et pour S et Mg à la fraction insoluble des végétaux. Pour tous ces éléments, la sortie principale est sous forme de solution. Dans RL, les bilans entrées/sorties sont équilibrés pour K, S et Si. K et S ont les mêmes flux principaux en entrées et en sortie que dans DC. Si arrive en majorité via la fraction insoluble des végétaux et via les poussières (Figure 38, Table 16). Leurs temps de résidences sont de 0.6 an pour K, 2.5 ans pour S et 3.7 ans pour Si.

Le flux d'entrée est supérieur au flux de sortie pour Mn, C, N, Ca et Si dans DC, ainsi que pour P, Mg, Mn, C, N, et Ca dans RL (Figures 37, 38, 39 et 40). A l'exception de P (49% des entrées sous forme soluble), les entrées principales de ces éléments pour les deux humus sont la fraction insoluble des litières (> 45%) (Tables 15 et 16).

Les stocks de ces éléments dans l'humus sont tous plus importants que les entrées. Les sorties se font majoritairement par la solution sauf pour Si dans DC où la principale sortie s'effectue à travers les particules.

Le flux d'entrée est inférieur à celui de sortie pour Al et Fe dans les deux humus (Figures 38 et 40). Ces éléments arrivent principalement par les poussières et sortent très majoritairement par les particules (84 à 86% des sorties pour Al et 78 à 19% pour Fe) (Tables 15 et 16). Les temps de résidence de ces éléments sont très variables. Par exemple

dans DC les temps de résidence sont de 15.9 ± 4.3 ans pour Al et 45.5 ± 31.1 ans. Puisque ces temps présentent de grand écarts-types dus à la forte variation des concentrations dans les stocks d'humus, un temps de résidence corrigé a été calculé en émettant l'hypothèse d'une entrée d'éléments similaire aux sorties. Ainsi, les temps corrigés obtenus dans DC sont de 3.1 ± 0.9 ans pour Al et de 5.7 ± 1.8 ans pour Fe (Figures 38 et 40).

1.2 Relation entre les éléments dans l'humus

Pour une meilleure compréhension des mécanismes et de la dynamique des éléments dans l'humus, il est indispensable d'identifier les relations existantes entre les différents éléments. Ces relations peuvent être observées à travers l'approche des bilans entrées-sorties et à travers une approche plus classique, fondée sur des corrélations entre deux éléments dans un même flux. Les annexes 1 et 2 présentent ces corrélations dans les flux entrants (solutions, poussières, litière totale) et sortants (solution et particules). Ces données sont reprises dans les Tables 15 et 16 et commentées dans les paragraphes suivants. A partir de la combinaison des deux approches, il est possible de différencier trois groupes d'éléments ayant une dynamique comparable (Tables 15 et 16).

1.2.1 K et P

Le premier groupe est composé de **K et P**. Leurs bilans entrées/sorties sont équilibrés dans DC et dans RL pour K. Cet équilibre peut s'expliquer par leur arrivée majoritaire sous **forme soluble** dans les végétaux (43 et 42% des entrées de K, et 50 et 49% des entrées de P dans DC et RL respectivement) et leur sortie principale en **solution** (93 et 92% pour K et 85 et 75% pour P dans DC et RL respectivement).

Ces éléments présentent également des temps moyens de résidence courts : entre 0.6 et 0.7 an pour K et 1.6 et 1.3 ans pour P dans DC et RL respectivement. Cette différence pourrait provenir du fait que K est presque directement entraîné par la solution alors que P est repris dans plusieurs mécanismes de recyclage (amibes à thèques et précipités autour des hyphes). Enfin, ces deux éléments sont principalement libérés dans la solution lors de la période de dormance de la végétation : ce sont donc des éléments qui pourraient être plus facilement perdus pour l'arbre si le sol n'a pas la capacité de les retenir. Ces deux éléments sont corrélés ($r^2 \geq 0.5$) dans les flux de solutions entrants et sortants et dans les retombées de litière (Tables 15 et 16). Ils sont également corrélés ponctuellement avec les éléments composant le second groupe.

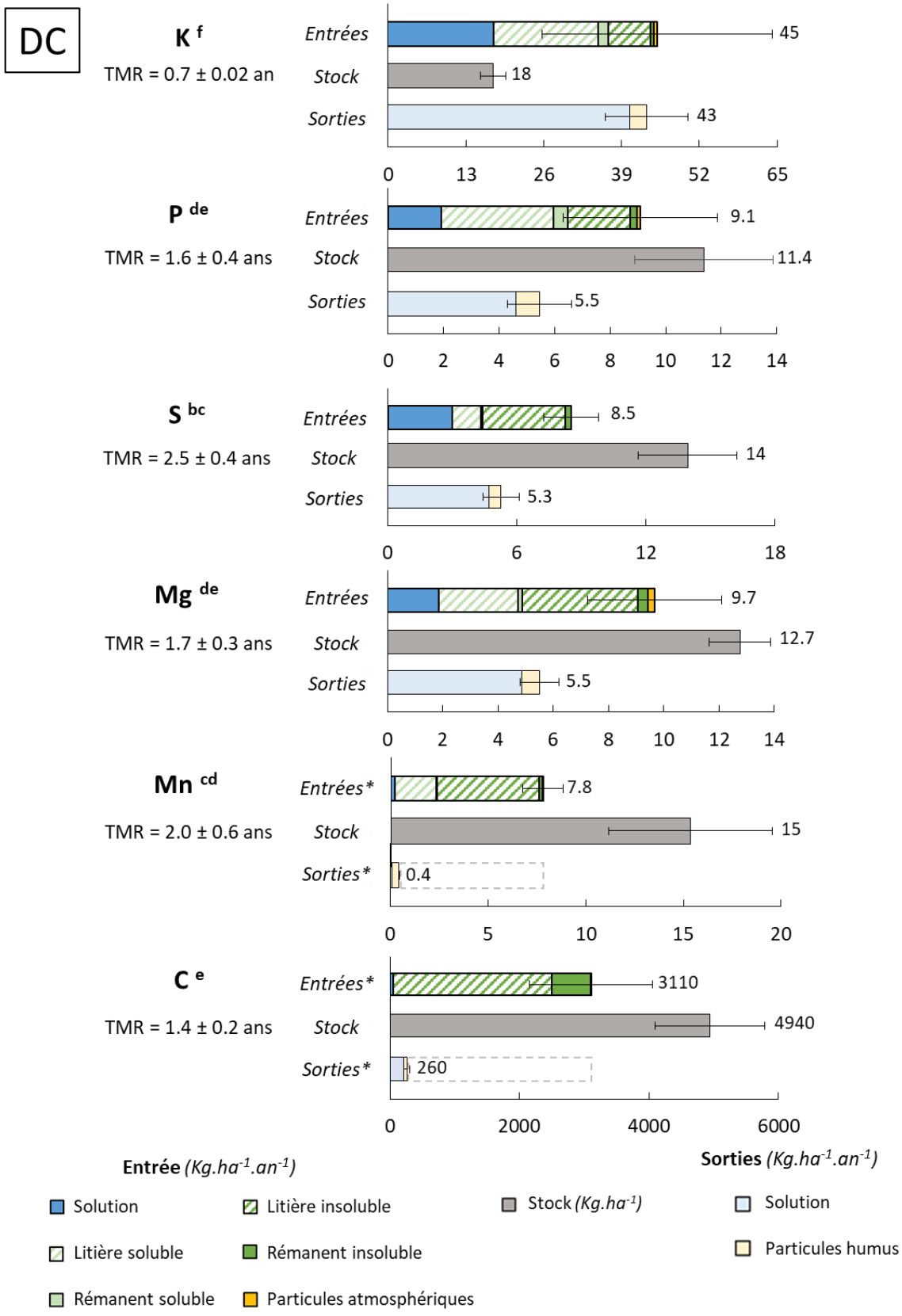

*Figure 37 : Figure de synthèse (1/4). Temps moyen de résidence (TMR), flux d'entrées, de sorties et stocks mesurés pour K, P, S, Mg, Mn et C dans DC. Les lettres représentent les différences significatives entre les différents TMR. Les * représentent les différences significatives entre le flux d'entrée total et le flux de sortie total. Les nombres en face des flux et des stocks représentent la quantité totale mesurée. Les carrés gris en pointillés montrent les données non mesurées dans les flux étudiés.*

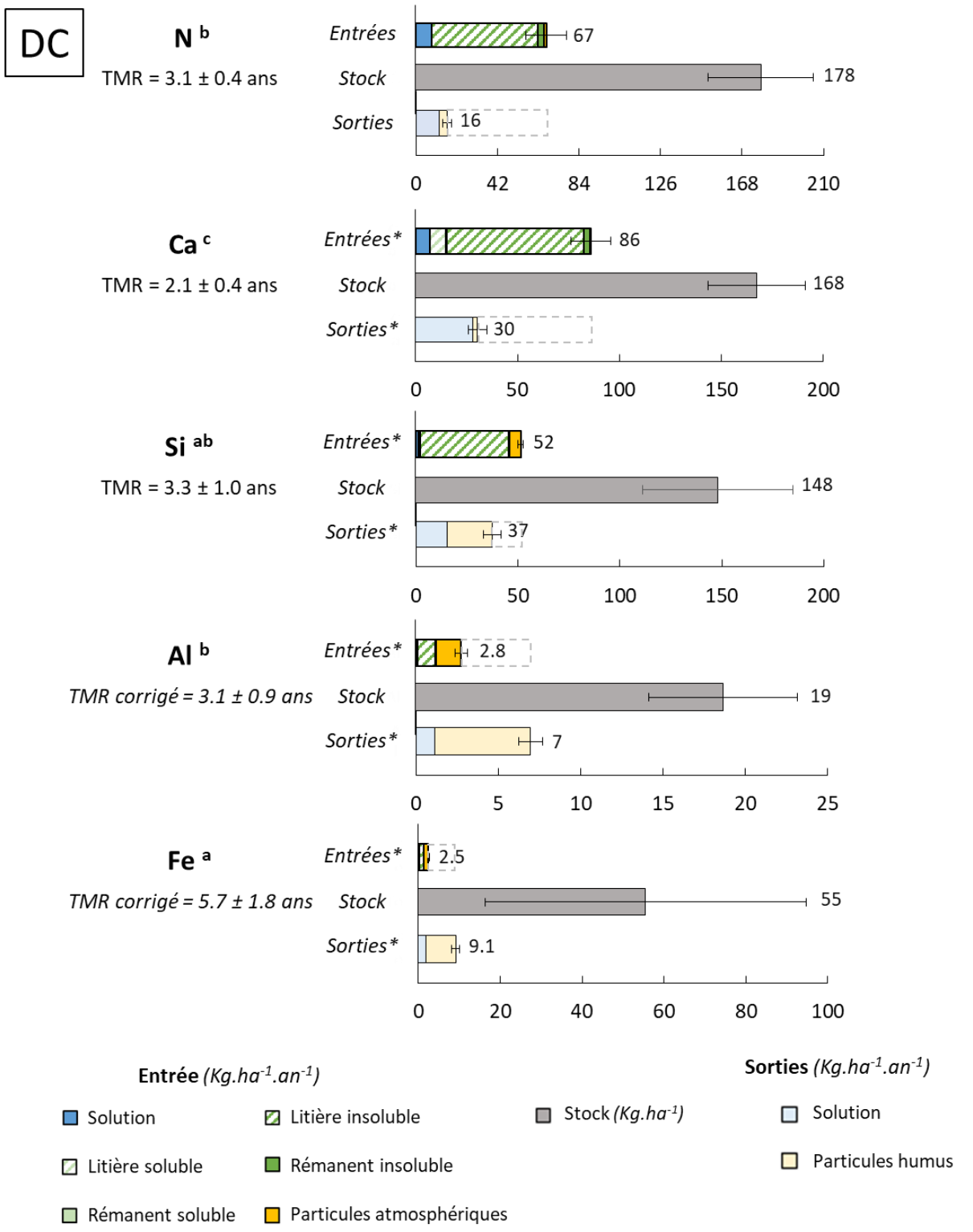

*Figure 38 : Figure de synthèse (2/4). Temps moyen de résidence (TMR), flux d'entrées, de sorties et stocks mesurés pour N, Ca, Si, Al et Fe dans DC. Les lettres représentent les différences significatives entre les différents TMR pour N, Ca et Si et les TMR corrigés pour Al et Fe. Les * représentent les différences significatives entre le flux d'entrée total et le flux de sortie total. Les nombres en face des flux et des stocks représentent la quantité totale mesurée. Les carrés gris en pointillés montrent les données non mesurées dans les flux étudiés.*

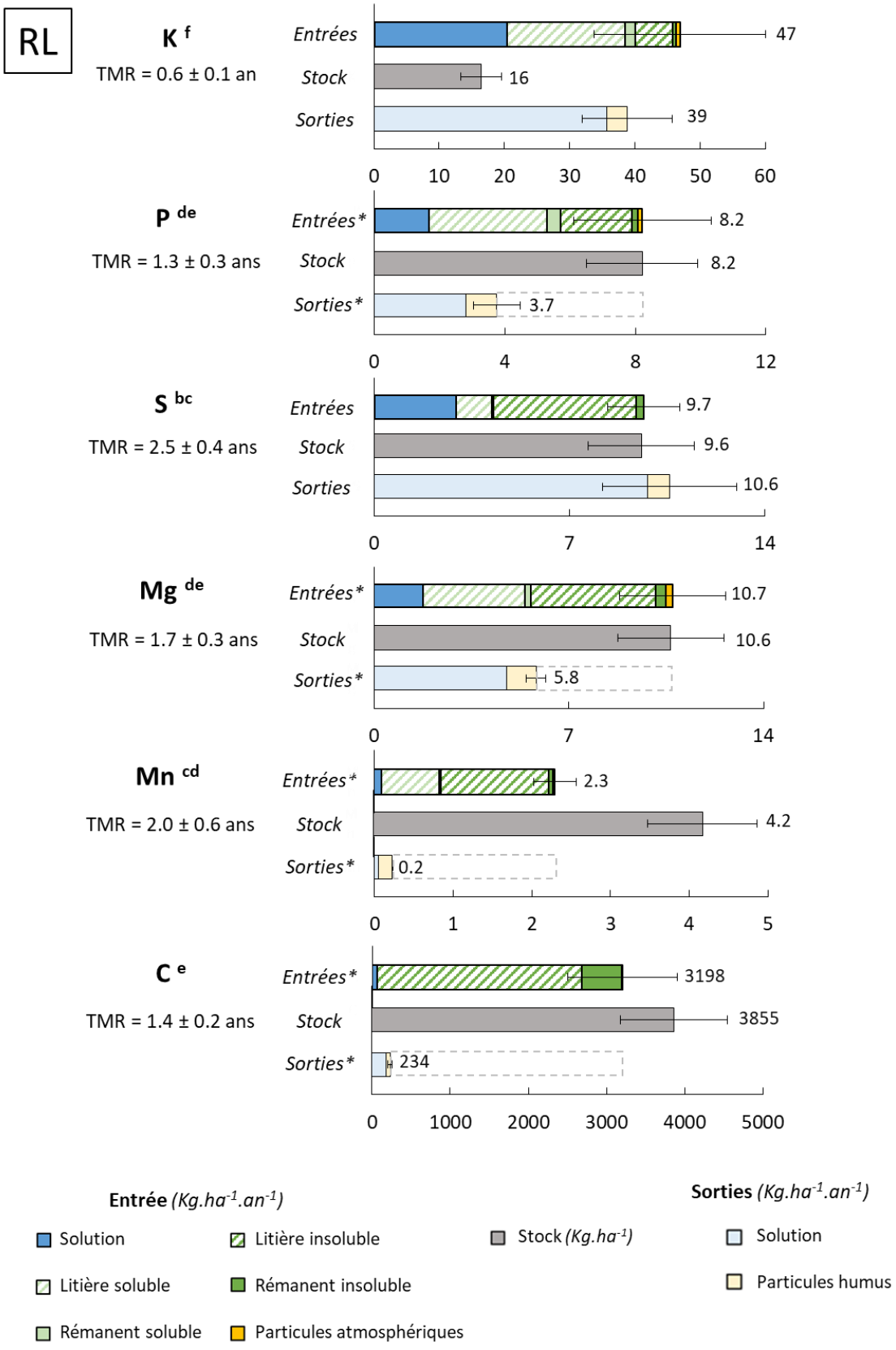

*Figure 39 : Figure de synthèse (3/4). Temps moyen de résidence (TMR), flux d'entrées, de sorties et stocks mesurés pour K, P, S, Mg, Mn et C dans RL. Les lettres représentent les différences significatives entre les différents TMR. Les * représentent les différences significatives entre le flux d'entrée total et le flux de sortie total. Les nombres en face des flux et des stocks représentent la quantité totale mesurée. Les carrés gris en pointillés montrent les données non mesurées dans les flux étudiés.*

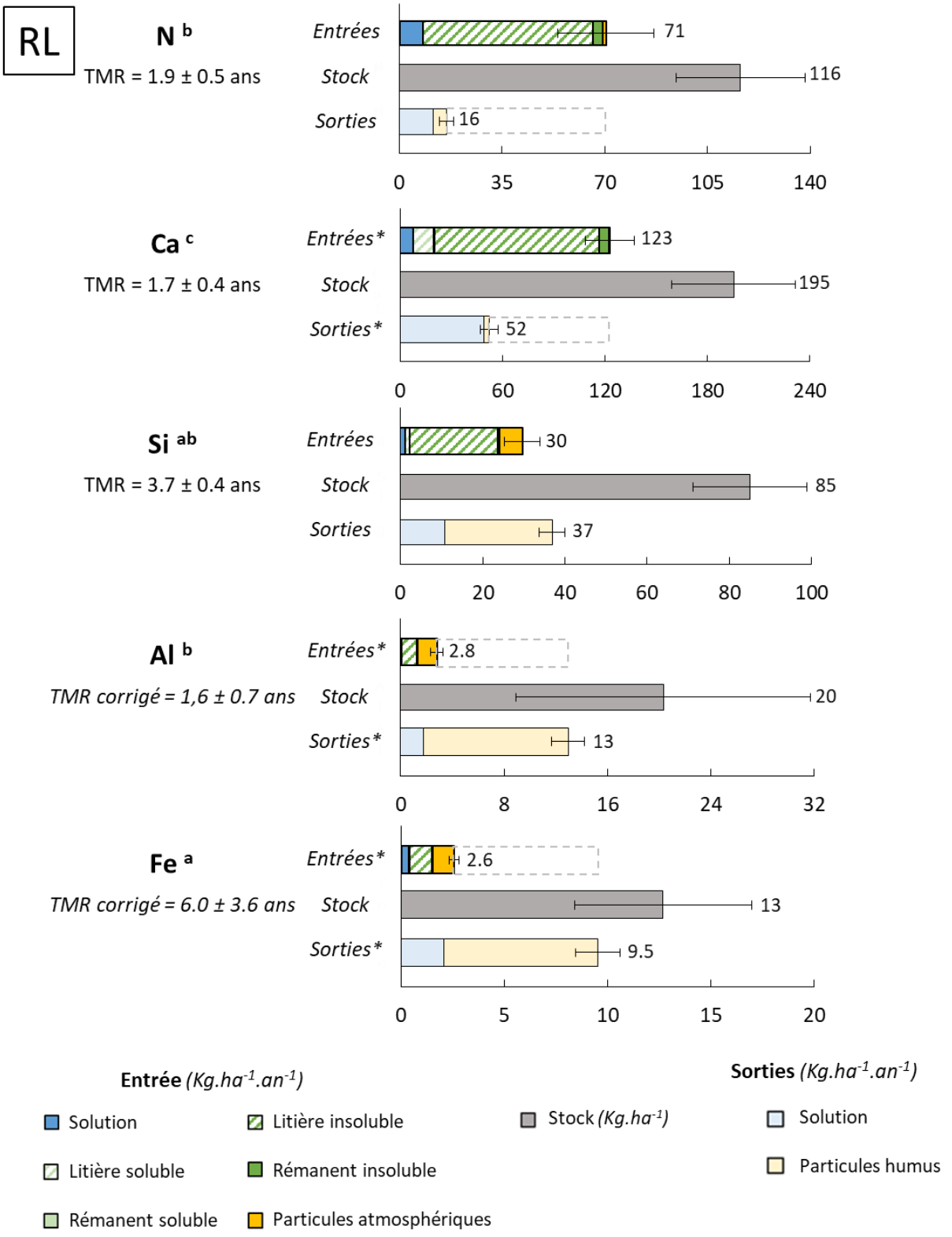

*Figure 40 : Figure de synthèse (4/4). Temps moyen de résidence (TMR), flux d'entrées, de sorties et stocks mesurés pour N, Ca, Si, Al et Fe dans RL. Les lettres représentent les différences significatives entre les différents TMR pour N, Ca et Si et les TMR corrigés pour Al et Fe. Les * représentent les différences significatives entre le flux d'entrée total et le flux de sortie total. Les nombres en face des flux et des stocks représentent la quantité totale mesurée. Les carrés gris en pointillés montrent les données non mesurées dans les flux étudiés.*

1.2.2 S, Mg, C, N, Ca, Si et Mn

Le second groupe est composé de **S, Mg, C, N, Ca, Si et Mn**. Il s'agit des éléments dont les entrées sont principalement représentées par leur **forme insoluble** dans les végétaux (entre 45 et 98% des entrées dans les deux sols). Ces éléments présentent généralement des bilans dont les entrées sont supérieures aux sorties, indiquant soit une forte immobilisation dans l'humus, soit des flux non mesurés en sortie. Pour ce groupe, les sorties sont majoritairement sous forme de solution (de 71 à 94% des sorties pour DC et RL) exceptés pour Mn et Si dont les sorties sont principalement sous forme de particules (60 à 83% des sorties pour DC et RL).

La libération de ces sept éléments dans la solution est répartie relativement équitablement tout au long de l'année : la production dans la solution varie de 25 à 45% en période de dormance et de 19 à 42% en période de végétation. Enfin ces éléments présentent des temps moyens de résidence allant de 1.4 an pour C à 3.3 ans pour Si. Ils sont globalement corrélés entre eux dans les flux d'entrées et de sorties. Ces corrélations pourraient être liées à leur source, à savoir la matière organique.

Dans ce groupe, un sous-groupe composé de **Ca et Si** peut-être différencié en fonction du **rapport sorties/entrées > 30%**. Ce rapport correspond à un indicateur de l'immobilisation des éléments dans l'humus, indiquant la présence d'un fort recyclage. En effet ces éléments ont été fréquemment observés dans des mécanismes de recyclage biotique (amibes à thèques et précipités autour des hyphes) et abiotique (précipités amorphes). Ces éléments sont également localisés dans les nervures des feuilles de litières (sous formes d'oxalates de calcium et de silice amorphe et comme composant des parois cellulaires pour Ca). Cependant, malgré leurs similitudes, ces éléments ne sont corrélés que dans le flux de solution en sortie. Ces corrélations nous informent que ces éléments proviennent de différentes sources mais ont une évolution similaire dans l'humus.

Dans le second groupe, **Mn** est un élément à part car le flux de sortie mesuré (0.4 $kg.ha^{-1}.an^{-1}$ dans DC et 0.2 $kg.ha^{-1}.an^{-1}$ dans RL) représente une faible proportion du flux entrant (5% dans DC et 8% dans RL). Avec un stock de 15 $kg.ha^{-1}$ dans DC et 4.2 $kg.ha^{-1}$ dans RL (Figures 37 et 39), soit cet élément est fortement immobilisé sur le long terme (précipité autour des hyphes et dans des thèques d'amibes), soit un flux de sortie n'a pas été quantifié comme le prélèvement par des hyphes. En raison du temps de résidence de 2 ans, la seconde hypothèse semble la plus probable.

Table 15 : Tableau de synthèse des résultats dans DC obtenus lors de la thèse. Les écarts-types des temps moyens de résidence sont entre parenthèses. nd : non déterminé, - : faible présence, + : présence, ++ : forte présence. Les temps moyens de résidence présentés ont été calculés pour la période 2012-2018 et ne présentent pas de différences significatives avec ceux calculés pour 2012-2015.

DC	Temps moyen de résidence (année)		Flux totaux (kg.ha.an^{-1})		Bilans annuels des entrées et sorties totales mesurées				Distribution des entrées (%)				Distribution des sorties (%)		Observations MEB et localisation des éléments	Corrélation (r^2) entre les éléments dans les flux entrants et sortants				Période de production d'éléments dans la solution en sortie (%)	
Eléments	Total	Corrigé	Flux des entrées totales	Flux sortie total	Entrées = Sorties	Entrées > Sorties	Entrées < Sorties	Sorties / Entrées pour entrées > sorties (%)	Solution	Forme soluble végétaux	Forme insoluble végétaux	Poussières (>0.45 µm)	Solution	Particules humus		Solution en entére	Litière	Poussières (>0.45µm)	Solution en sortie	Dormance	Végétation
K	0.7 (0.02)		45	43	oui				39	**43**	17	1	93	7	Précipités autour des hyphes -	P; C; Mg; S	Ca; Mg;Mn; S; N; P; C	N ; Al; Fe ; Mg; Mn; Si	Ca; P; S; Si; C	64	21
P	1.6 (0.4)		9.1	5.5	oui				21	**50**	27	2	85	15	Précipités autour des hyphes + Amibes à tecques ++	Mg; C; K; Si	Al; K; Mg; Mn; S; N; C		K	49	17
S	2.5 (0.4)		8.5	5.3	oui				35	17	**48**	0	89	11		Mg; K	Al; K; Ca; Mg; Mn; N; C	*nd*	K; Si; N; Ca; Mg; C	33	36
Mg	1.7 (0.3)		9.7	5.5	oui				19	31	**47**	3	89	11	Précipités autour des hyphes - Amibes à tecques -	P; S; Si; K; C	Al; Fe; K; Mn; N; Ca; P; S; C	Si; N; Al; Fe; K; Mn	N; S; C; Ca	35	42
Mn	2.0 (0.6)		7.8	0.4		oui		6	3	28	**69**	0	17	83	Précipités autour des hyphes + Amibes à tecques -		Al; K; Mg; N; C; Ca; P; S	Ca; Al; Fe; K; Mg; Si		*nd*	*nd*
C	1.4 (0.2)		3110	260		oui		*nd*	2	*nd*	**98**	0	81	19		Ca; P; S; Si; K; Mg	Ca; Fe; Mn ; K; Mg ;P ; S; N		Al; Ca; K; Mg; S; Si	44	32
N	3.1 (0.4)		67	16		oui		*nd*	12	*nd*	**86**	2	75	25			K; Mg ;Mn; P; S; C	K; Si; Mg; Al; Fe	Mg; S	25	19
Ca	2.1 (0.4)		86	30		oui		**35**	8	10	**82**	0	93	7	Précipités autour des hyphes ++ Amibes à tecques +	C	K; P; C; Mg; Mn; S	Mn	K; Si; N; Mg; C	35	40
Si	3.3 (1.0)		52	37		oui		**72**	3	2	**85**	11	40	60	Précipités amorphes + amibes à tecques ++	Mg; C; P	Al	Mg; N;Al; Fe; K; Mn	Al; Fe; K; S; C; Ca	45	33
Al	15.9 (4.3)	3.1 (0.9)	2.8	7.0			oui		3	1	42	**54**	16	84			Mg; Mn; P; S; Si	Fe; K; Mg; Mn; Si; N	Si; C; Fe	54	25
Fe	45.5 (31.1)	5.7 (1.8)	2.6	9.1			oui		13	2	41	**44**	21	79			Mg; C	Al; K; Mg; Mn; Si; N	Al; Si	57	28

Table 16 : Tableau de synthèse des résultats dans RL obtenus lors de la thèse. Les écarts-types des temps moyens de résidence sont entre parenthèses. nd : non déterminé, - : faible présence, + : présence, ++ : forte présence. Les temps moyens de résidence présentés ont été calculés pour la période 2012-2018 et ne présentent pas de différences significatives avec ceux calculés pour 2012-2015.

RL	Temps moyen de résidence (année)		Flux totaux (kg.ha.an^{-1})		Bilans annuels des entrées et sorties totales mesurées				Distribution des entrées (%)				Distribution des sorties (%)		Observations MEB et localisation des éléments	Corrélation (r^2) entre les éléments dans les flux entrants et sortants				Période de production d'éléments dans la solution en sortie (%)	
Eléments	Total	Corrigé	Flux des entrées totales	Flux sortie total	Entrées = Sorties	Entrées > Sorties	Entrées < Sorties	Sorties / Entrées pour entrées > sorties (%)	Solution	Forme soluble végétaux	Forme insoluble végétaux	Particules atmosphérique (>0.45 µm)	Solution	Particules humus		Solution en entére	Litière	Particules atmosphériques (>0.45µm)	Solution en sortie	Dormance	Végétation
K	0.6 (0.1)		26	39	oui				43	**42**	14	1	92	8	Précipités autour des hyphes -	Mg; C; P; Si	Ca; S; C; Mg; P	N ; Al; Fe ; Mg; Mn; Si	Ca; Mg; S; Si; C; P	64	21
P	1.3 (0.3)		8.2	3.7		oui		46	20	**49**	29	2	75	25	Précipités autour des hyphes + Amibes à tecques ++	Mg; K; Si	Ca; C; K; Mg; S; N		K	49	17
S	1.5 (0.4)		9.7	10.6	oui				30	14	**56**	0	93	7		Ca; Mg	K; N; C; Ca; Mg; P	*nd*	K; Mg; Ca; C	33	36
Mg	1.2 (0.3)		10.7	5.8		oui		55	17	35	**45**	3	82	18	Précipités autour des hyphes - Amibes à tecques -	Ca; K; P; S; C	N; C; Ca; K; P; S	Si; N; Al; Fe; K; Mn	K; S; Si; Ca; C	35	42
Mn	2.0 (0.5)		2.3	0.2		oui		6	4	34	**62**	0	21	79	Précipités autour des hyphes + Amibes à tecques -			Ca; Al; Fe; K; Mg; Si		*nd*	*nd*
C	1.1 (0.2)		3198	234		oui		*nd*	2	*nd*	**98**	0	77	23		Ca; K; Mg	K; Mg; P; S; N		K; S; Ca; Mg; Si	44	32
N	1.9 (0.5)		71	16		oui		*nd*	11	*nd*	**87**	2	71	29			Ca; Mg; S; P; C	K; Si; Mg; Al; Fe		25	19
Ca	1.7 (0.4)		123	52		oui		**35**	6	11	**83**	0	94	6	Précipités autour des hyphes ++ Amibes à tecques +	Mg; S; C	K; P; N; Mg; S	Mn	K; Mg; S; C	35	40
Si	3.7 (0.4)		30	37		oui		**72**	4	4	**81**	11	29	71	Précipités amorphes + amibes à tecques ++	K; P	Al	Mg; N;Al; Fe; K; Mn	K; Al; Mg; Fe; C	45	33
Al	16.4 (9.0)	1.6 (0.7)	2.8	13.0			oui		3	1	42	**54**	14	86			Si	Fe; K; Mg; Mn; Si; N	Si; Fe	54	25
Fe	10.6 (3.1)	6.0 (3.6)	2.6	9.5			oui		14	2	41	**43**	22	78				K; Mg; Mn; Si; N	Al; Si	57	28

1.2.3 Al et Fe

Le troisième groupe différencié se compose d'**Al et Fe**. Les entrées de ces éléments sont principalement sous forme de **minéraux** dans les poussières (54% pour Al et 43 à 44% pour Fe dans DC et RL) et leurs sorties sous forme de particules (84 à 86 % pour Al et 78 à 79% pour Fe dans DC et RL). Comme les sorties sont supérieures aux entrées, il pourrait y avoir une entrée non mesurée. Cette entrée pourrait être liée à la présence de sol dans l'humus avec Al et Fe des particules de sol qui se complexeraient avec les fractions de matière organique des végétaux dégradés présents dans l'humus. Les stocks d'Al et Fe sont extrêmement variables dans les humus des deux sols. De ce fait, leurs temps moyens de résidence calculés sans correction sont très élevés et très variables (16 ± 4 ans pour Al et 45 ± 31 ans pour Fe dans DC) (Figures 38 et 40). Considérant que les bilans de ces éléments sont équilibrés (car le stock dans l'humus ne varie pas) et que les manques proviennent d'entrées du sol, un temps moyen de résidence corrigé est calculé. Il est pour Al 3.1 ± 0.9 ans dans DC et 1.6 ± 0.7 ans dans RL. Pour Fe, il est de 5.7 ± 1.8 ans dans DC et 6.0 ± 3.6 ans dans RL.

1.3 Limites de l'approche

L'approche utilisée pour estimer la dynamique des éléments dans l'humus peut présenter certaines limites :

- Malgré la durée de l'étude (7 ans) et les nombreux échantillonnages, la variabilité interannuelle, notamment liée au climat, est visible à travers des écart-types plus ou moins importants dans les différents flux.
- Bien qu'en 2018, 27 échantillons d'humus par sol sur une surface de 0.1 m^2 aient été prélevés, on remarque une grande variabilité dans les stocks des éléments dans l'humus, due à une variabilité spatiale du stock d'éléments sur le site d'étude.
- Malgré toutes les précautions prises et l'utilisation d'un modèle utilisant le Ti comme indicateur de sol, le mélange du sol avec l'humus pourrait induire des biais dans l'estimation des éléments dans l'humus.
- Le positionnement des fluteaux pourrait ne pas être exactement à la limite humus/sol.
- Le non dosage des flux gazeux ne permet pas d'équilibrer les bilans de C et N

Cependant, il convient de préciser que toutes les conclusions et les discussions réalisées dans cette thèse ont tenu compte de ces différentes limites et ne remettent pas en cause les résultats obtenus tout au long de ce travail.

1.4 Contrôle de la dynamique des éléments dans l'humus

Dans la littérature, la dynamique des éléments dans l'humus (ou dans l'ensemble des couches holorganiques) est très peu rapportée en dehors de C et N. Le plus souvent, la dynamique des éléments est étudiée à travers la méthode des sachets de litières. La fraction soluble est directement lessivée à l'arrivée au sol et les matières organiques sont décomposées plus ou moins rapidementselon leurs teneurs en cellulose et lignine mais aussi en fonction des communautés d'organismes présents. Par ailleurs, les études par bilans tiennent compte de l'écosystème dans son ensemble et ne sont donc pas centrées sur l'humus. De plus, dans la majorité des cas, les seules entrées prises en compte dans l'humus sont les litières et les pluviolessivats. Les sorties des éléments correspondent le plus souvent à la solution drainée hors du sol et les émissions gazeuses (notamment pour C et N). L'immobilisation des éléments dans l'humus a été évoquée, spécialement pour Ca et Si, à travers les amibes à thèques et/ou des précipités autour des hyphes. Il n'existe cependant à ce jour aucune étude présentant dans son ensemble la dynamique des éléments dans l'humus.

La Figure 41 synthétise le travail effectué dans cette thèse autour de la dynamique des éléments (K, P, S, Mg, Mn, C, N, Ca, Si, Al et Fe). L'ensemble des flux entrant a été mesuré soit : les solutions (écoulement de tronc + pluviolessivats), les débris végétaux (litière et rémanents) et les poussières (> 0.45 µm). Dans les entrées solides, les éléments ont été identifiés sous deux formes différentes : soluble et insoluble (minéraux dans les poussières, biominéraux dans les végétaux et matière organique). Ces différentes entrées sont transférées avec des dynamiques différentes : les éléments dans la solution et le soluble sortent directement de l'humus, viennent ensuite les matières organiques, puis les minéraux et biominéraux (parfois localisés dans les parties des végétaux plus concentrées en lignine) sont dégradés. Dans l'humus, les éléments provenant de ces différentes sources peuvent être impliqués dans différents processus :

- **Le transfert direct via la solution.** Les éléments sont directement emportés par la solution.
- **La dissolution**. Les éléments contenus dans les poussières ou les végétaux sont dissous sous l'effet des conditions bio-physicochimiques présentes.
- **Le recyclage biotique et abiotique.** Certains éléments peuvent être immobilisés dans l'humus par des processus de recyclage (qui peuvent ensuite être dissous).
- **La complexation entre des éléments du sol et les éléments de l'humus.** Les particules de sol incorporées dans l'humus pourraient interagir avec les éléments présents par des réactions de complexation (notamment entre Al, Fe et les matières organiques présentent).

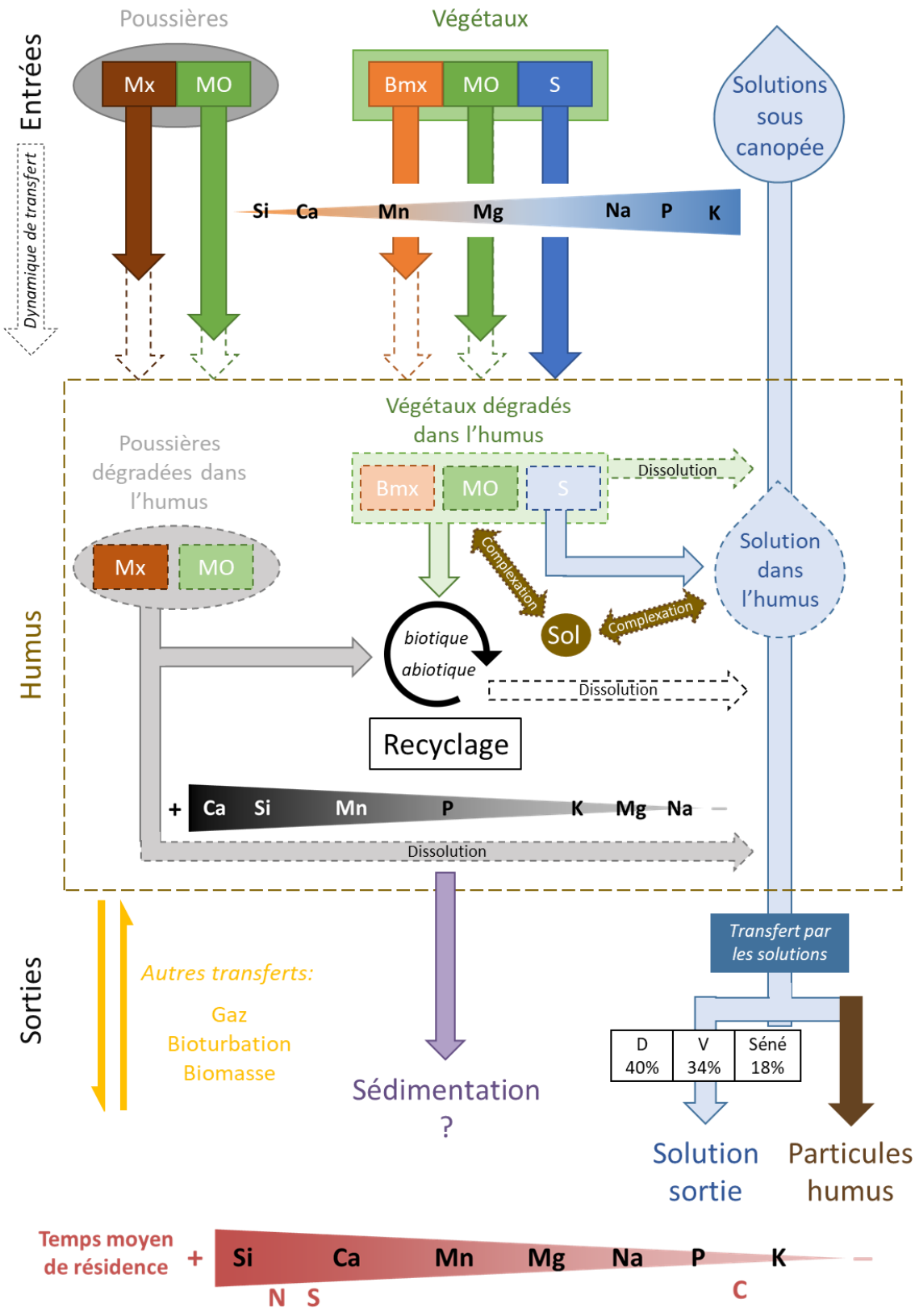

Figure 41 : Schéma de synthèse présentant les différentes entrées et sorties et les mécanismes dans l'humus ainsi que la relation entre les temps moyens de résidence, leur solubilité dans les entrées et leur recyclage dans l'humus. Mx : minéraux, MO : matière organique, Bmx : biominéraux, S : soluble, D : dormance, V : végétation, Séné : Sénéscence

Dans cette étude, nous avons identifié et de quantifié trois flux non pris en compte dans les précédentes études, à savoir les particules en sortie de l'humus, les rémanents et les poussières. Ces nouveaux flux ont permis de compléter les bilans entrées/sorties des éléments. Cependant, la plupart des éléments étudiés ne présentent pas un bilan entrées/sorties équilibré : des flux n'ont donc pas été quantifiés dans cette thèse. Pour C et N, une sortie non quantifiée correspond aux gaz qui n'ont pas été mesurés pendant cette thèse. Pour les éléments sous forme de biominéraux ou complexés avec la matière organique, une hypothèse pourrait être leur dépôt sur le sol, c'est-à-dire une sédimentation à la surface du sol. D'autres transferts pourraient provenir de la bioturbation, par exemple par l'apport de sol par les vers de terre ou la sortie non quantifiée d'éléments par ces mêmes organismes. La biomasse microbienne n'est également pas prise en compte dans cette thèse, qu'il s'agisse des microorganismes vivants ou morts. Enfin, bien qu'aucune racine n'ait été observée dans les humus étudiés, les hyphes pourraient prélever une partie des nutriments directement dans l'humus.

2 Conclusions et perspectives

2.1 Conclusions

Cette thèse a répondu à l'objectif de comprendre la dynamique (en dehors de C et N) et la relation des éléments dans l'humus (ou dans les différentes couches holorganiques) en se fondant sur un modèle conceptuel identifiant et quantifiant les entrées et les sorties de l'humus.

Nous avons, dans un premier temps, calculé les temps de résidence des éléments étudiés et identifié les mécanismes associés. Ainsi, la forme des éléments dans les entrées (notamment l'importance de la forme soluble et des biominéraux dans les végétaux), la localisation de ces éléments dans les feuilles (limbe vs. nervure) et le recyclage biotique et abiotique ayant lieu dans l'humus influencent le temps moyen de résidence des éléments dans l'humus : plus l'élément arrivera sous forme soluble, plus son temps de résidence sera court.

Nous avons ensuite identifié et quantifié de nouveaux flux entrants et sortants de l'humus et évalué l'importance de ces différents flux dans la dynamique des éléments. Nous avons confirmé les deux hypothèses émises concernant ces flux : il existe un flux de particules en sortie de l'humus permettant de boucler et compléter les bilans des éléments et, selon l'élément, la part des différents flux est variable (par exemple influence principale des poussières et des particules pour Al et Fe). Nous avons également mis en évidence l'existence d'autres flux potentiels d'entrées (apport d'éléments du sol se complexant avec la matière organique) et de sorties (sédimentation, prélèvement par les hyphes, bioturbation) non quantifiés dans cette étude et qui permettraient d'équilibrer les bilans des éléments.

En étudiant la période de libération des éléments dans la solution de l'humus, nous avons montré qu'elle avait lieu principalement lors de la période de dormance de l'arbre et représentait un risque de perte d'éléments pour l'arbre. Ainsi le sol aura un rôle important pour retenir ces éléments. Nous avons également montré qu'en plus d'être influencée par les précipitations, la libération des éléments était influencée par l'action des microorganismes et par la forme des éléments dans les entrées.

A travers une étude expérimentale de la dégradation des litières en sachets, nous avons mis en évidence l'influence du type de sol sur la dégradation des litières. Nous avons également calculé des demi-vies de dégradations similaires aux demi-temps de résidence calculés pour les humus des mêmes sols. Lors de cette étude expérimentale, nous avons également testé la taille des mailles et le séchage ou non de la litière sur les vitesses de dégradations. Ces différents facteurs expérimentaux n'ont cependant pas eu d'effet sur les vitesses.

Enfin, à partir de l'étude des bilans entrées/sorties et d'une approche par corrélation, nous avons confirmé que les relations entre éléments étaient liées à la source de l'élément (végétaux, poussière, solution) mais également à leurs formes dans les entrées (soluble/non soluble).

2.2 Perspectives

Si nous avons réussi à répondre aux questions scientifiques àl'origine de cette thèse, de nouveaux questionnements sont apparus.

Lors de cette étude, les bilans pour C et N n'étaient pas équilibrés (plus d'entrées que de sorties). La quantification des flux gazeux en sortie de l'humus pourrait permettre d'équilibrer le bilan de ces éléments.

Bien que la quantification du flux de particules en sortie de l'humus ait permis d'équilibrer ou de compléter le bilan de certains éléments (notamment Si, Ca, Al et Fe), la conséquence de ce flux sur le fonctionnement du sol et le transfert des éléments (y compris le C) reste à déterminer. De plus, la dynamique saisonnière de transfert des éléments via ce flux reste à mesurer.

Nous avons pu observer au MEB la présence d'hyphes de champignons colonisant la litière et entourés de précipités minéraux. La présence de ces hyphes pourrait indiquer le prélèvement direct d'éléments dans l'humus, qui serait ainsi un nouveau flux de sortie à quantifier. Autour de ces hyphes, des précipités le plus souvent composés de Ca et Mn, pourraient-être mieux caractérisés et quantifiés pour mieux décrire la dynamique de ces éléments dans l'humus. Nous avons également observé au MEB des amibes à thèques dans les litières. Il serait intéressant de mieux connaître l'écologie de ces micro-organismes ainsi que de rechercher les mécanismes biotiques et abitotiques responsables de la cinétique de dissolution de leurs thèques.

L'humus est le siège d'une activité microbienne intense. Identifier les communautés présentes ainsi que l'apport de la biomasse morte aux flux d'élément pourrait compléter l'étude effectuée dans cette thèse.

Nous avons mis en évidence la sortie des éléments de l'humus pendant la période de dormance des végétaux. Cependant, le devenir des éléments une fois dans le sol et leur rôle dans la nutrition des arbres ne sont pas abordés dans cette thèse, ainsi que la forme directement assimilable ou non par les végétaux.

Le travail effectué dans cette thèse s'est concentré sur la dynamique des éléments dans des mulls. Cependant, cette dynamique pourrait être différente dans des moders ou des mors et impliquer d'autres mécanismes.

Si nous avons pu répondre à un certain nombre de questions liées à la dynamique des éléments dans l'humus, il est nécessaire de poursuivre les recherches sur ce niveau de l'écosystème souvent négligé pour des éléments autres que C, N ou P.

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Annexes

R^2 Solution entrée

DC	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.19									
Fe	0.13	0.02								
K	0.13	0.44	0.02							
Mg	0.18	0.49	0.05	0.82						
Mn	0.03	0.08	0.02	0.02	0.12					
P	0.09	0.31	0.02	0.84	0.67	0.00				
S	0.35	0.38	0.10	0.38	0.57	0.13	0.28			
Si	0.17	0.47	0.02	0.77	0.63	0.01	0.77	0.34		
N	0.16	0.02	0.03	0.02	0.00	0.01	0.04	0.23	0.01	
C	0.31	0.53	0.08	0.70	0.82	0.11	0.51	0.61	0.63	0.06

R^2 Solution humus

DC	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.33									
Fe	0.90	0.27								
K	0.40	0.52	0.36							
Mg	0.32	0.91	0.28	0.41						
Mn	0.13	0.08	0.07	0.12	0.09					
P	0.17	0.39	0.13	0.68	0.29	0.04				
S	0.33	0.77	0.31	0.68	0.76	0.11	0.48			
Si	0.70	0.61	0.62	0.63	0.47	0.07	0.40	0.53		
N	0.08	0.49	0.07	0.18	0.58	0.03	0.05	0.53	0.15	
C	0.53	0.84	0.43	0.70	0.71	0.14	0.41	0.72	0.82	0.33

R^2 Poussières

DC	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.20									
Fe	0.99	0.18								
K	0.95	0.40	0.93							
Mg	0.78	0.27	0.77	0.84						
Mn	0.76	0.64	0.72	0.92	0.76					
P	0.23	0.22	0.28	0.32	0.31	0.31				
S										
Si	0.93	0.23	0.90	0.87	0.58	0.72	0.12			
N	0.74	0.04	0.76	0.67	0.69	0.44	0.45		0.56	
C	0.15	0.14	0.16	0.04	0.01	0.00	0.01		0.20	0.22

R^2 particules humus

DC	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.79									
Fe	0.99	0.82								
K	0.75	0.95	0.81							
Mg	1.00	0.81	1.00	0.77						
Mn	0.85	0.61	0.83	0.56	0.84					
P	0.93	0.92	0.94	0.87	0.93	0.86				
S	0.95	0.91	0.96	0.83	0.96	0.84	0.99			
Si	0.99	0.84	0.97	0.77	0.99	0.86	0.96	0.99		
N	0.91	0.65	0.89	0.61	0.91	0.99	0.89	0.89	0.92	
C	0.92	0.86	0.91	0.76	0.93	0.88	0.97	0.99	0.97	0.91

* Pas assez de données pour obtenir de bonnes corrélations

R^2 Litière totale

DC	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.47									
Fe	0.16	0.39								
K	0.23	0.52	0.43							
Mg	0.53	0.72	0.50	0.55						
Mn	0.51	0.80	0.32	0.58	0.62					
P	0.51	0.67	0.44	0.84	0.83	0.72				
S	0.54	0.75	0.44	0.64	0.87	0.81	0.85			
Si	0.71	0.46	0.20	0.09	0.42	0.41	0.26	0.43		
N	0.18	0.45	0.33	0.52	0.67	0.51	0.65	0.76	0.11	
C	0.24	0.69	0.65	0.82	0.76	0.68	0.81	0.81	0.18	0.72

Annexe 1 : Corrélation entre les éléments dans les différents flux en entrées et en sorties de l'humus de DC. Vert : $r^2 \geq 0.7$. Bleu : $0.5 \leq r^2 < 0.7$

R^2 Solution entrée

RL	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.41									
Fe	0.17	0.07								
K	0.07	0.45	0.02							
Mg	0.27	0.60	0.09	0.65						
Mn	0.13	0.08	0.06	0.01	0.09					
P	0.05	0.40	0.03	0.92	0.59	0.02				
S	0.35	0.54	0.16	0.32	0.64	0.20	0.28			
Si	0.14	0.49	0.03	0.75	0.48	0.00	0.73	0.30		
N	0.34	0.08	0.05	0.02	0.03	0.09	0.03	0.21	0.00	
C	0.36	0.57	0.06	0.52	0.60	0.10	0.40	0.48	0.48	0.13

R^2 Solution humus

RL	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.27									
Fe	0.89	0.34								
K	0.27	0.62	0.34							
Mg	0.33	0.82	0.40	0.51						
Mn	0.34	0.27	0.32	0.16	0.28					
P	0.12	0.41	0.15	0.72	0.34	0.05				
S	0.11	0.78	0.17	0.53	0.55	0.19	0.30			
Si	0.69	0.65	0.76	0.62	0.56	0.32	0.34	0.47		
N	0.00	0.34	0.01	0.10	0.35	0.07	0.05	0.31	0.06	
C	0.36	0.84	0.42	0.67	0.71	0.35	0.42	0.62	0.73	0.20

R^2 Poussières

RL	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.20									
Fe	0.99	0.18								
K	0.95	0.40	0.93							
Mg	0.78	0.27	0.77	0.84						
Mn	0.76	0.64	0.72	0.92	0.76					
P	0.23	0.22	0.28	0.32	0.31	0.31				
S										
Si	0.93	0.23	0.90	0.87	0.58	0.72	0.12			
N	0.74	0.04	0.76	0.67	0.69	0.44	0.45		0.56	
C	0.15	0.14	0.16	0.04	0.01	0.00	0.01		0.20	0.22

R^2 particules humus

RL	Al	Ca	Fe	K	Mg	Mn	P	S	Si	N
Al										
Ca	0.64									
Fe	1.00	0.66								
K	0.73	0.80	0.75							
Mg	0.99	0.64	0.99	0.79						
Mn	0.77	0.60	0.76	0.51	0.73					
P	0.85	0.62	0.87	0.90	0.91	0.54				
S	0.74	0.67	0.75	0.96	0.80	0.55	0.92			
Si	0.95	0.71	0.95	0.66	0.92	0.82	0.71	0.63		
N	0.66	0.59	0.67	0.91	0.72	0.51	0.88	0.98	0.52	
C	0.81	0.76	0.83	0.97	0.86	0.64	0.94	0.98	0.72	0.95

* Pas assez de données pour obtenir de bonnes corrélations

R^2 Litière totale

RL	Al	Ca	Fe	K	Mg	Mn	P	S	Si	TN
Al										
Ca	0.01									
Fe	0.12	0.11								
K	0.12	0.55	0.24							
Mg	0.22	0.71	0.12	0.71						
Mn	0.02	0.24	0.02	0.14	0.22					
P	0.20	0.66	0.16	0.77	0.92	0.15				
S	0.05	0.80	0.09	0.70	0.79	0.10	0.84			
Si	0.51	0.00	0.00	0.00	0.07	0.00	0.03	0.01		
N	0.05	0.62	0.07	0.44	0.65	0.37	0.72	0.55	0.01	
C	0.02	0.72	0.15	0.64	0.63	0.43	0.66	0.56	0.03	0.79

Annexe 2 : Corrélation entre les éléments dans les différents flux en entrées et en sorties de l'humus de RL. Vert : $r^2 \geq 0.7$. Bleu : $0.5 \leq r^2 < 0.7$

Résumé

L'humus est le siège de processus clés pour la nutrition des arbres. Cependant, il reste un compartiment de l'écosystème encore peu étudié pour l'ensemble des nutriments et éléments bénéfiques autre que C et N. Ainsi, dans un contexte où les pressions exercées sur les forêts augmentent depuis quelques années, il est important de comprendre la dynamique comparée des éléments de leur arrivée dans l'humus à leur sortie dans le sol. Les objectifs de cette thèse sont (1) d'identifier les mécanismes et les facteurs responsables de la dynamique des éléments majeurs (C, N, P, K, Ca, Mg, Mn, S, Si, Al et Fe), (2) de déterminer les relations entre ces éléments et (3) de préciser la place de l'humus dans la nutrition de l'arbre. Pour répondre à ces objectifs, un modèle conceptuel basé sur l'identification des flux entrant et sortant de l'humus et des possibles mécanismes présents dans le niveau de l'humus a été élaboré.

Les résultats obtenus dans cette thèse suggèrent que la dynamique des éléments et leur disponibilité pour l'arbre à la sortie de l'humus dépendent d'un ensemble de processus et réactions accélérant ou retardant leur départ de l'humus. Les vitesses de libération des éléments dans le sol dépendent de leurs formes dans les entrées de l'humus (soluble, molécules organiques, biominéraux), de leur localisation dans les feuilles (limbe vs. nervure) et des tissus des végétaux plus ou moins facilement dégradables alimentant l'humus. La dynamique des éléments est également influencée par des réactions de recyclage à l'intérieur de l'humus, comme des précipitations biotiques ou abiotiques. Les éléments être utilisés par les amibes à thèques (Si, P, Ca et Mn), précipités autour des hyphes (Ca, Mn, P, et K) ou encore être précipité sous forme de plage amorphes (Si). Ces réactions contrôlent les temps moyens de résidence des éléments : les éléments les plus solubles, localisés dans des tissus plus dégradables et les moins recyclés seront lessivés plus rapidement et auront des temps de résidence plus bas. La période libération des éléments sous l'humus intervient sur leur disponibilité pour l'arbre de ces éléments. En période de végétation où les éléments sont directement disponibles pour les arbres, moins de 25% de K et P sont libérés. La capacité du sol à retenir les éléments produits en période de sénescence et de dormance sera fondamentale pour assurer la pérennité de l'écosystème. Lors de cette thèse, les particules en sortie de l'humus ont été quantifiées pour la première fois. Outre le rôle sur la pédogenèse en général et la dynamique du C, ce flux particulaire sous l'humus pourrait augmenter la capacité d'échanges cationique des sols et sa capacité à retenir ces éléments lessivés en période de dormance.

Mots clés : Humus; dynamique; bilan entrées/sorties; particules; formes des éléments; hêtraie; recyclage

Abstract

Humus is the place of key tree nutrition processes. However, it remains a compartment of the ecosystem still under studied for all major nutrients and beneficial elements other than C and N. Thus, in a context of increasing pressure on forests, it matters to understand the dynamics of the elements from their input into the humus to their output towards the soil. The aims of this thesis are: (1) to identify the mechanisms and factors responsible of the major element dynamics (C, N, P, K, Ca, Mg, Mn, S, Si, Al and Fe), (2) to determine the relationships between these elements and (3) to specify the place of the humus in tree nutrition. To answer these objectives, a conceptual model based on the identification of humus inputs and outputs and the possible mechanisms present at the humus level was elaborated.

The results of this thesis suggest that the element output fluxes depends on processes and reactions accelerating or delaying their release from the humus. The element release rates in the soil depend on their forms in the humus inputs (soluble, organic molecules, biominerals) and their location in the plant tissues (leaf blade vs. vein) supplying the humus. The element dynamics is also influenced by recycling reactions within the humus, such as biotic or abiotic precipitation through testate amoebae (Si, P, Ca and Mn), precipitates around hyphae (Ca, Mn, P, and K) or amorphous Si precipitates. These reactions control the mean residence times of the elements and their leaching gradient below the humus: the most soluble and least immobilized elements will be leached more rapidly and will have lower residence time. The release of these elements below the humus is another important factor that affects the availability of these elements to trees. During the growing season, when the trees upatke soil solution, less than 25% of K and P are released from humus. The ability of the soil to hold the elements produced during periods of senescence and dormancy is fundamental to ensure the sustainability of the ecosystem. In this thesis, the particles leaving the humus were quantified for the first time. In addition to their role on pedogenesis and the dynamics of C, this particle flux below the humus could increase the cation-exchange capacity of the soil and its ability to hold these leached elements during the dormant period.

Key words: Humus; dynamic; input/output budget; particles; element forms; beech forest; recycling