



Modèles agro-environnementaux de la fertilisation phosphatée dans les sols acides de pomme de terre de l'Est du Canada

Thèse

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Résumé

Le phosphore (P) est indispensable pour la pomme de terre, la principale culture des provinces de l'Est canadien. La diminution des réserves phosphatées et le risque de perte de cet élément dans les cours d'eau constituent des contraintes majeures pour la sécurité alimentaire et pour l'environnement. L'objectif de ce projet de recherche est d'optimiser la dose et le type d'engrais phosphaté en vue de minimiser le risque d'accumulation du P dans le sol, et de substituer les engrains de synthèse par des engrains recyclés.

Un total de 141 échantillons représentatifs des sols de l'Île-du-Prince-Édouard (ÎPE) a été d'abord analysé et a montré que l'acidité du sol influait grandement sur le pourcentage de saturation des sols en P. Ainsi, les seuils critiques environnementaux en terme d'indice de saturation en P, $(P/Al)_{M3}$ ont été de 19% pour les sols très acides ($pH_{eau} < 5,5$), et de 14% pour les sols moins acides ($pH_{eau} > 5,5$), au-dessus desquels la fertilisation en P devrait être limitée aux besoins de la culture. Six classes de risque environnemental ont été identifiées et leur répartition spatiale a montré que la classe de risque modéré était prédominante à l'ÎPE. Nous avons ensuite développé un modèle agro-environnemental de P pour la pomme de terre basé sur 42 essais de fertilisation conduits dans les provinces maritimes (ÎPE, Nouveau-Brunswick (NB) et Nouvelle-Écosse (NÉ)). Ce modèle, basé sur l'indice $(P/Al)_{M3}$, a été composé de sept classes de diagnostic et de recommandation pour la fertilisation phosphatée : (0-2,5%), (2,5%-5%), (5%-10%), (10%-14%), (14%-16%), (16%-23%) et $(P/Al)_{M3} > 23\%$ correspondant, respectivement, aux doses optimales en P de 105, 81, 70 kg P ha⁻¹ pour les trois premières classes, et à une dose de 21 kg ha⁻¹ pour les classes dont le $(P/Al)_{M3}$ est supérieur à 10%. Finalement, pour évaluer l'efficacité d'un engrain phosphaté recyclé (Crystal Green®), nous avons conduit 8 essais de fertilisation au Québec et dans les provinces maritimes. Il a été conclu que la fertilisation phosphatée avec le Crystal Green® pourrait substituer le triple superphosphate (TSP) jusqu'à 75% sans que les niveaux de rendements en pomme de terre, le prélèvement du P, et le statut du P dans le sol et dans la plante ne soient changés.

Abstract

Phosphorus (P) is essential for potatoes, the main crop in the Canadian eastern provinces. The depletion of phosphate reserves and the risk of loss of this element in watercourses are major constraints for food security and for environment. The objective of this study is to optimize the rate and type of phosphate fertilizer in order to minimize the risk of P accumulation in the soil, and to substitute synthetic fertilizers with recycled ones.

A total of 141 soil samples representative of Prince Edward Island (PEI) soils were first analyzed. It has been shown that soil acidity greatly influenced P soil saturation percentage. Thus, critical thresholds in terms of P saturation index, $(P/Al)_{M3}$, were 19% for very acid soils ($pH_{water} < 5.5$), and 14% for less acidic soils ($pH_{water} > 5.5$), above which P fertilization should be limited to the crop's requirement. Six environmental risk classes were identified and their spatial distribution showed that the moderate risk class was predominant in PEI. We then developed an agro-environmental model of P for potato based on 42 fertilization trials conducted in the Maritime provinces (PEI, New Brunswick (NB) and Nova Scotia (NS)). This model, based on the $(P/Al)_{M3}$ index, was composed of seven fertility and environmental risk classes: (0-2.5%), (2.5%-5%), (5%-10%), (10%-14%), (14%-16%), (16%-23%) and $(P/Al)_{M3} > 23\%$ corresponding, respectively, to the optimal P rates of 105, 81, 70 kg P ha^{-1} for the first three classes, and to a P rate of 21 kg ha^{-1} for classes with $(P/Al)_{M3}$ greater than 10%. Finally, to evaluate the effectiveness of a recycled P fertilizer (Crystal Green[®]), we conducted 8 fertilization trials in Quebec and in the Maritime provinces. It was concluded that P fertilization with Crystal Green[®] could substitute triple superphosphate (TSP) up to 75% without noticing any significant decrease in potato yield levels, P uptake, and P status in the soil and in the plant.

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| | |
|------------------------------------|---|
| α_m | Maximal sorption coefficient |
| AEMs | Anion exchange membranes |
| Al | Aluminium |
| APR | Apparent P recovery |
| CEC | Cation exchange capacity |
| Cryst | Crystal Green® |
| DAP | Day after planting |
| DPS | Degree of P saturation |
| DRP | Phosphore réactif dissous |
| Fe | Fer |
| FN | False negative |
| FP | False positive |
| ICP | Plasma emission spectroscopy |
| ISP/PSI | Phosphorus saturation index |
| ISP ₁ /PSI ₁ | P saturation index as expressed by the ratio of P over Al extracted with M3 |
| ISP ₂ /PSI ₂ | P saturation index as expressed by the ratio of P over the sum of (Al+Fe) extracted with M3 |
| ISP ₃ | Indice de saturation en P dans les sols organiques |
| M3 | Mehlich-3 extraction method or Mehlich-3-extractable |
| MY | Marketable yield |
| NB | New Brunswick |
| NPV | Negative predictive value |
| NS/NÉ | Nova Scotia/Nouvelle Écosse |
| OM | Organic matter |
| ox (as a subscript) | Ammonium-oxalate-extractable |
| P | Phosphorus |
| PEI/ÎPE | Prince Edward Island |
| pH _{SMP} | pH buffering capacity |
| PPV | Positive predictive value |
| PU | P uptake |
| SAA | Spectrophotométrie d'absorption atomique |
| SEP | Spectrométrie d'émission par plasma |
| P _w | Water-extractable P |
| QC | Québec |
| RF | Random forest |
| RK | Regression kriging |
| RP | Rock phosphate |
| RY | Relative yield |
| TN | True negative |
| TP | True positive |
| TSP | Triple superphosphate |
| TY | Total yield |

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Avant-propos

Cette thèse est composée d'une introduction, de quatre chapitres et d'une conclusion générale et recommandations. Le chapitre 1 est une revue de la littérature qui permet de mettre le projet de recherche dans son contexte, d'introduire les différents aspects à aborder, de dégager les problématiques et d'énoncer les hypothèses et les objectifs de recherche. Les chapitres 2, 3 et 4 sont présentés sous formes d'articles scientifiques. Le chapitre 2 est intitulé « Identifying environmental phosphorus risk classes at the scale of Prince Edward Island, Canada » et est consacré à la classification du risque environnemental de saturation des sols de l'ÎPE en phosphore. Ceci permet de prévoir le risque de pollution diffuse des sols agricoles en P, et de suivre l'évolution spatio-temporelle de ce risque au niveau de la province. Ce chapitre a été publié dans *Canadian Journal of Soil Sciences* en avril 2018. Le titre du chapitre 3 est « An Agro-Environmental Phosphorus Model for Potato in the Canadian Maritime Provinces ». Ce chapitre porte sur l'élaboration d'un modèle agronomique et d'un modèle de recommandation en phosphore pour la culture de pomme de terre dans les provinces maritimes. Il décrit notamment les niveaux de fertilité des sols en fonction de l'indice de fertilité correspondant, et permet d'en déduire les doses optimales à apporter en phosphore. Ce chapitre a été publié en septembre 2018 dans *Agronomy Journal*. Quant au chapitre 4, il concerne l'évaluation de l'efficacité d'un engrais à base de phosphore recyclé sur la culture de pomme de terre en comparaison avec un engrais phosphaté de synthèse. Cet article sera soumis pour publication sou peu. Ce document se termine par une conclusion générale sur les principaux résultats à retenir, et qui propose des perspectives de recherche. Pour le chapitre 2, des modifications ont été apportées au niveau de la présentation des références bibliographiques en comparaison à la version publiée, et ce, pour maintenir l'uniformité dans la présentation de la présente thèse.

Les résultats de ces études ont été partiellement présentés au 29^{ème} congrès annuel de l'Association Québécoise de Spécialistes en Sciences du Sol (AQSSS) en juillet 2015 à Montréal (communication orale).

Les trois articles insérés dans la thèse, dont je suis la rédactrice principale, ont été rédigés en anglais. Pour le chapitre 2 (article 1), j'ai réalisé toutes les analyses des sols dans les laboratoires du Centre de recherche d'Agriculture et Agroalimentaire Canada (AAC) situé à Charlottetown. Pour le chapitre 3 (article 2), j'ai été responsable du traitement des données.

Quant au chapitre 4 (article 3), j'ai participé à toutes les étapes de l'expérimentation, depuis le semis de la pomme de terre jusqu'à la récolte, passant par les divers échantillonnages des sols et des plantes effectués le long de la saison de croissance. Les travaux de terrain, la collecte des échantillons et l'analyse des sols et des plantes ainsi que les dosages ont été réalisés avec l'aide de nombreuses personnes (professionnels de recherche d'AAC et de l'Université Laval, et étudiants stagiaires). Le traitement des données, l'interprétation des résultats et leurs discussions ont été réalisés par moi-même sous la direction de mon directeur et de ma codirectrice de recherche.

Les co-auteurs sont le docteur Lotfi Khiari de l'Université Laval, la docteure Judith Nyiraneza du Centre de recherche d'AAC situé à Charlottetown, ainsi que d'autres chercheurs de différents centres de recherche d'AAC.

Introduction

Le phosphore (P) est un élément essentiel au développement des végétaux. La faible disponibilité du P dans les écosystèmes naturels est un facteur limitant des cultures. Cette déficience doit être corrigée par une fertilisation phosphatée nécessaire pour la croissance des plantes et l'optimisation de la production. Les engrains phosphatés chimiques couramment utilisés dans l'agriculture sont fabriqués à partir de la roche phosphatée (Talboys et al., 2016). Les réserves naturelles mondiales en roche phosphatée sont limitées et non renouvelables (Reijnders, 2014). Face à l'accroissement continu de l'industrie des engrains phosphatés, ces réserves risquent de s'épuiser d'ici les cent prochaines années (Reinhard et al., 2017). Ceci entraînerait de graves conséquences sur la production agricole et sur la sécurité alimentaire (Cordell et al., 2009).

Le P apporté dans le sol sous forme de fertilisant n'est qu'en partie assimilé par la plante. Le P non assimilé est fixé par les argiles ou les sesquioxides de fer et d'aluminium (Khiari et Parent, 2005). L'accumulation du P en excès dans les sols a un effet néfaste sur l'environnement, à savoir l'eutrophisation des cours d'eau. Ce phénomène est causé par l'enrichissement de l'eau en éléments nutritifs, soit l'azote, le carbone, et notamment le phosphore (Durrant et al., 1999) entraînant, à long terme, la croissance des algues et des plantes aquatiques. Il s'agit de l'une des principales causes de dégradation de la qualité des eaux de surface des régions humides. Le phosphore peut atteindre ces eaux naturellement par érosion, ruissellement et infiltration. Il peut aussi provenir des milieux agricoles, industriels et municipaux. L'apport des activités industrielles et municipales dans la contamination des eaux par le phosphore a été réduit grâce aux mesures d'assainissement et aux normes qui ont été établies. En effet, la réduction du flux de P en provenance des rejets des stations d'épuration des eaux usées pourrait constituer un moyen efficace de lutte contre ce phénomène (Le Corre et al., 2007). L'agriculture demeure toutefois l'activité ayant la majeure contribution à l'eutrophisation (Pierzynski et al., 2000; Parent et al., 2002) surtout dans les agrosystèmes qui nécessitent des apports excessifs en P tels que ceux supportant la culture de pomme de terre .

La pomme de terre est une culture particulièrement exigeante en P. Les doses apportées en engrais phosphatés dépassent généralement ce que cette culture est capable de prélever. Ces doses vont de 150 à 400 kg de P₂O₅ par hectare alors que moins de 50 kg ha⁻¹ seront prélevés réellement par la plante. C'est ainsi que cette culture est devenue une cause potentielle d'accumulation du P dans les sols et donc, de pollution diffuse. Dans les provinces de l'Est du Canada, la culture de la pomme de terre constitue une des principales productions agricoles. Pour adopter de bonnes pratiques agricoles liées à l'utilisation du P dans ces régions tout en optimisant la production, il est indispensable de développer des indicateurs de saturation des sols en P dans un double objectif de diagnostic à la fois agronomique et environnemental. En se basant sur des mécanismes de sorption, d'adsorption et de désorption du P dans les sols, il est nécessaire de développer une grille de fertilisation agroenvironnementale de P basée sur deux modèles. Le premier de nature environnementale pour déduire des valeurs critiques de saturation des sols en P pour l'ensemble des sols agricoles des maritimes quelles que soit leurs vocations. Le deuxième modèle est de nature agronomique pour déduire des valeurs critiques de la fertilité phosphatée des sols de pomme de terre dans ces mêmes provinces. En vue d'une gestion durable qui préserve le mieux les ressources en P, il est pertinent de mettre à l'essai la nouvelle génération d'engrais phosphatés issus du recyclage. Ces engrais pourraient constituer une alternative, à la fois économique et écologique, pour les engrais phosphatés chimiques.

Ce projet de doctorat a eu pour objectif de faire le diagnostic environnemental et d'élaborer des modèles agro-environnementaux de la fertilisation phosphatée dans les sols sableux acides de pomme de terre des provinces de l'Est canadien. Ce projet a également permis de tester une nouvelle génération d'engrais phosphatés issus du recyclage afin d'évaluer leur efficacité sous culture de pomme de terre.

Dans ce chapitre introductif, on passe en revue les différents indicateurs agronomiques utilisés pour mesurer la disponibilité du P dans le sol et dans le plant, les indicateurs environnementaux établis pour évaluer la saturation des sols en P, et les indices agro-environnementaux développés pour permettre une double interprétation agronomique et environnementale. Il décrit également les différentes étapes de l'élaboration d'un modèle de recommandation en P, et présente brièvement quelques exemples d'engrais P de synthèse

et de recyclage. Ce chapitre permet de présenter des connaissances générales, d'émettre les hypothèses et d'énoncer les objectifs de cette recherche.

1. Chapitre 1: Revue de littérature

1.1 Cycle, formes et disponibilité du phosphore dans le sol

1.1.1. Cycle du phosphore dans le sol

Le phosphore est un élément essentiel pour tous les organismes vivants. Chez les végétaux, le phosphore rentre dans la composition des structures membranaires et des acides nucléiques, et contribue au transfert d'énergie à l'intérieur des cellules (Morel, 1989).

Le P est présent dans le sol sous deux formes: organique et minérale. La première forme provient des résidus des cultures, des apports organiques ainsi que de l'activité des micro-organismes du sol (Morgan, 1997). La forme minérale qui représente de 50% à 70% du P total des sols agricoles (Pierzynski et al., 2000) se caractérise par une forte interaction avec les différentes composantes du sol. Ces deux formes de P peuvent subir diverses réactions chimiques et microbiologiques complexes.

Bien que le contenu en P total dans les sols soit généralement de 600 mg kg^{-1} en moyenne (Lindsay, 1979), plus de 90% de ce P se trouve sous formes peu solubles ou irréversiblement retenues par le sol (Stevenson, 1986). C'est pour cette raison que le P est considéré comme un facteur limitant des cultures. Les engrains phosphatés, apportés généralement sous forme entièrement soluble afin de corriger cette déficience, ne sont pas totalement utilisés par les cultures. En effet, la plante n'utilise que 5% à 30% du P ajouté (McLaughlin et al., 1988) laissant 70% à 95% de P résiduel. Au fil du temps, le P soluble des engrains accumulé dans les sols réagit avec les composantes du sol ce qui le rend de moins en moins disponible pour les plantes (Stevenson, 1986). Le cycle du P consiste en un système de transfert dynamique entre le sol, la plante et la biomasse microbienne (Fig. 1.1) (Pierzynski et al., 2000).

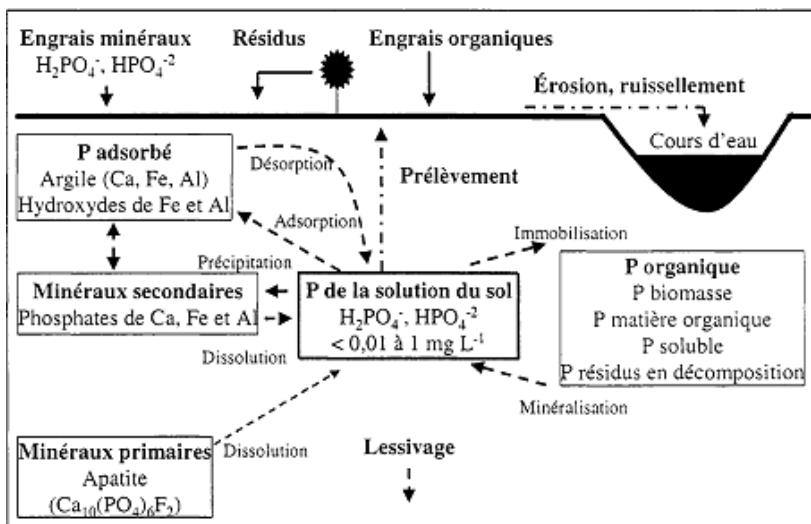


Figure 1.1. Cycle du phosphore (Adapté de Pierzynski et al., 2000)

1.1.2. Formes et disponibilité du P dans le sol

Les plantes sont capables d'absorber uniquement les formes minérales du P sous forme d'ions orthophosphates, principalement l'ion bivalent HPO_4^{2-} et l'ion monovalent $H_2PO_4^-$ (Morel 1989). La présence de ces formes minérales du P dépend du pH du sol. Pour un pH acide (pH 4.0 à 7.2), les ions $H_2PO_4^-$ prédominent dans la solution du sol. Les ions HPO_4^{2-} sont abondants plutôt dans les sols alcalins (pH > 7.2) (Pierzynski et al., 2005). La teneur en ions orthophosphates dans la solution du sol est très faible par rapport à la teneur totale du P dans les sols agricoles. Elle varie de moins de 0,01 à plus de 1 mg P L^{-1} . Le P est donc majoritairement lié à la phase solide du sol (Pierzynski et al., 2000) par des mécanismes d'adsorption réversibles sur les colloïdes du sol, ou fortement retenu sur les particules de sol.

L'adsorption des ions phosphates est considérée comme étant le phénomène principal contrôlant la migration du P minéral dans le profil du sol. L'adsorption du P réfère à des réactions d'échange du P ionique ($H_2PO_4^-$, HPO_4^{2-}) entre la solution et la phase solide du sol. Cette adsorption est assurée essentiellement par les colloïdes des sols, soit l'argile et la matière organique (Pierzynski et al., 2005). La liaison se fait également par l'intermédiaire d'un pont calcique formé par les ions calcium déjà adsorbés sur leur surface (Morel, 1989). Le P peut aussi être fixé sur les oxydes et hydroxydes de fer (Fe) et d'aluminium (Al), ou sur les carbonates de calcium (Ca) (Sample et al., 1980).

La biodisponibilité du P est régie par trois facteurs: le facteur « intensité » représente l'activité du P en solution, complètement et immédiatement disponible aux plantes; le facteur « quantité » représente la quantité de P soluble susceptible d'être assimilée; et le facteur «capacité» qui renseigne sur l'aptitude d'un sol à maintenir le facteur intensité constant lorsque le facteur quantité varie après une fertilisation ou suite à un prélèvement par la plante (Tran et Giroux, 1990).

Les voies de transformation et de solubilisation du phosphore et son interaction avec les composantes du sol sont complexes. Afin d'adapter des indicateurs de diagnostic du P, des méthodes d'extraction ont été développées et corrélées aux rendements et au prélèvement de cet élément par la plante.

1.2 Diagnostic agronomique du phosphore dans le sol au Canada

1.2.1. Indicateurs du diagnostic agronomique du P

Les analyses du sol constituent un indicateur agronomique nécessaire pour le diagnostic, l'interprétation et les recommandations en engrais phosphatés des cultures. Ces indicateurs sont couramment appelés indicateurs du phosphore biodisponible grâce à leur bonne corrélation avec le rendement et d'autres critères de performance comme le prélèvement du P par la culture.

Étant donné la grande variabilité entre les propriétés des différents types de sol, plusieurs méthodes d'analyse du P biodisponible ont été développées au Canada. Il est recommandé, pour une même région, d'opter pour la méthode d'analyse la plus efficace pour ses sols afin d'assurer le minimum de variabilité entre les laboratoires (Maguire et al., 2005). En général, la méthodologie d'analyse fait appel à une solution extractive avec un rapport sol/solution, un temps et une vigueur d'extraction, ainsi qu'une technique de dosage bien déterminés (CRAAQ, 2010).

Les indicateurs agronomiques du phosphore les plus connus au Canada sont P-Bray, P-Kelowna, P-Olsen et P-Mehlich-3 (Ziadi et al., 2013) (Tableau 1.1).

Tableau 1.1. Méthodes d'analyses du P biodisponible au Canada (Tiré et adapté de Ziadi et al., 2013).

| Méthodes d'analyses | Références | Extractifs | Types de sol | Provinces |
|-------------------------|---------------------------|--|---|--|
| Mehlich-3 | Mehlich (1984) | 0.2N CH ₃ COOH 0.25N NH ₄ NO ₃ 0.015N NH ₄ F 0.013N HNO ₃ 0.001M EDTA (pH 2.5) | Sols acides à neutres | Est du Canada : <ul style="list-style-type: none">• Île-du-Prince-Édouard• Nouvelle-Écosse• Nouveau-Brunswick• Québec |
| Olsen | Olsen et al. (1954) | 0.5M NaHCO ₃ (pH 8.5) | Sols calcaires | <ul style="list-style-type: none">• Ontario• Manitoba |
| Kelowna | van Lierop (1988) | 0.015N NH ₄ F 0.25N CH ₃ COOH (pH 3.2) | Sols acides et calcaires de pH neutre à alcalin | Ouest du Canada : <ul style="list-style-type: none">• Alberta• Colombie-Britannique• Saskatchewan |
| Kelowna modifiée | Qian et al. (1994) | 0.015N NH ₄ F 0.025N CH ₃ COOH 0.25N CH ₃ COONH ₄ (pH 4.9) | | |
| | Ashworth et Mrazek (1995) | 0.015N NH ₄ F 0.5N CH ₃ COOH 1N CH ₃ COONH ₄ | | |
| Bray-1 | Bray et Kurtz (1945) | 0.03N NH ₄ F 0.025N HCl (pH<3) | Sols acides et neutres | Terre-Neuve et Labrador |
| Bray-2 | | 0.03N NH ₄ F 0.1N HCl (pH<3) | | |

Dans les Maritimes (Terre-Neuve, Nouvelle-Écosse, Île-du-Prince-Édouard, Nouveau-Brunswick) et au Québec, on utilise la méthode d'extraction au Mehlich-3 (Mehlich, 1984) depuis la fin des années 1980, car c'est la méthode la plus adaptée aux sols acides à neutres. Elle se caractérise par une solution extractive acide qui permet d'extraire plusieurs éléments nutritifs du sol, en plus du P (Beaudin, 2008). Ce P extrait au Mehlich-3 (P_{M3}) est étroitement corrélé au P prélevé par la plante (Tran et Giroux, 1985; Tran et Giroux, 1990; Simard et al., 1991). Depuis le développement de l'indice de saturation (P/Al) $M3$ (Khiari et al., 2000), ce dernier a rapidement été adapté pour l'établissement des grilles de recommandations en P. En effet, le rapport entre le P et l'Al extraits au Mehlich-3 a montré une meilleure performance dans l'interprétation du comportement du P dans le sol que le P_{M3} (Khiari et al., 2000; Pellerin et al., 2006a). L'indice de saturation en P (P/Al) $M3$ est maintenant à la base des grilles de recommandations en P pour plusieurs cultures, notamment la pomme de terre, au Québec (CRAAQ, 2010) et au Nouveau-Brunswick (GNB, 2011) et a permis d'améliorer la gestion du P dans leurs sols.

La méthode Olsen (Olsen et al., 1954) est utilisée essentiellement en Ontario et au Manitoba pour les sols légèrement acides ($pH < 6.0$), légèrement alcalins ($6.0 < pH < 7.2$) et les sols calcaires ($pH > 7.2$) (Ziadi et al. (2013)). La solution extractive de bicarbonate de sodium ($NaHCO_3$) extrait le P par l'action d'hydrolyse de l'Al et du Fe, et par la précipitation du Ca (Tran et Giroux, 1985). Ainsi, en diminuant la concentration et l'activité des cations qui retiennent le P, la solubilité de ce dernier augmente et le P est ensuite plus facilement mesuré (Beaudin, 2008). Contrairement à l'extraction multiéléments Mehlich-3, la méthode Olsen extrait seulement le P biodisponible.

La méthode Kelowna a été proposée en 1988 par van Lierop pour extraire, simultanément, plusieurs éléments des sols acides et calcaires. Les modifications apportées à la méthode Kelowna (ajout d'acétate d'ammonium) ont permis d'améliorer la prévision du potassium disponible par rapport au Kelowna (Qian et al., 1994). Les méthodes Kelowna et Kelowna modifiée sont les mieux adaptées aux sols des Prairies canadiennes (Havlin et al., 1999).

La méthode d'extraction Bray-2 (Bray et Kurtz, 1945) a longtemps été utilisée pour extraire le P dans les sols acides et neutres du Québec et pour formuler les recommandations

en engrais phosphatés dans cette province (Tran et Giroux, 1985). En 1987, les travaux de Tran et Giroux ont conclu que cette méthode ne devait pas être utilisée pour les recommandations en P au Québec car elle extrait des formes de P non disponibles aux plantes et est moins bien corrélée aux rendements des cultures que la méthode Mehlich-3.

1.2.2. Indicateurs de disponibilité *in situ* du P par la technique des résines d'échanges ioniques

Bien qu'elles soient efficaces pour estimer la disponibilité des éléments nutritifs dans le sol, les méthodes d'extraction chimiques décrites dans la section précédente présentent certaines limitations. En plus d'être parfois coûteuses et longues à réaliser (Binkley et Matson, 1983), ces extractions chimiques fournissent seulement un indice et non un niveau absolu de disponibilité des éléments nutritifs du sol étant donné qu'elles ne tiennent pas compte des conditions réelles du champ (Cooperband et Logan, 1994; Qian et Schoenau, 2002).

Les membranes échangeuses d'ions, ou résines d'échanges ioniques, ont été proposées comme étant une alternative aux extractions chimiques pour évaluer la disponibilité du P dans le sol (Zheng et al., 2003). Ces résines prélèvent les ions de la solution du sol avec des mécanismes d'adsorption et d'échanges anioniques comparables à ceux des racines des plantes.

Les résines d'échanges ioniques accumulent les ions de la solution du sol par des réactions d'échanges similaires au mécanisme d'échange du système sol-racines. Elles simulent l'action des racines des plantes en absorbant continuellement les nutriments de la solution du sol (Yang et al., 1991). Les ions de la solution du sol sont redistribués sur les sites d'échanges ioniques des résines par diffusion. Donc, en plus de mesurer la concentration de la solution du sol en nutriments, les résines d'échanges ioniques renseignent également sur leur taux de diffusion à travers le sol (Abrams et Jarrel., 1992). La quantité d'ions adsorbés sur la surface de la membrane est donc fonction de leur concentration, de leur diffusion, et de la capacité du sol de les renouveler dans sa solution (Ziadi, 1999). Les résines sont dites anioniques ou cationiques selon la nature des ions de la solution du sol qu'on cherche à mesurer (anions ou cations). Dans le cas du phosphore, ce sont les ions orthophosphates

HPO_4^{2-} et H_2PO_4^- libres dans la solution du sol qui sont adsorbés sur la surface des résines. On parle donc de résines cationiques ou de résines d'échanges anioniques.

1.2.3. Indicateurs de P basés sur le plant de pomme de terre: P des pétioles

Les analyses du sol sont le principal outil utilisé pour évaluer les besoins en engrais phosphatés selon le niveau de fertilité du sol avant la plantation. Il existe cependant un autre moyen de détection et de correction de toute déficience en P pendant le cycle de croissance de la culture: l'analyse du P des pétioles (Rosen et al., 2014). Pour la pomme de terre, c'est le pétiole de la quatrième feuille mature sous le point de croissance qui est utilisé pour l'analyse de la composition foliaire (Westermann et Kleinkopf, 1985). En 1997, Rocha et al. ont montré que le rendement en tubercules a été plus corrélé au P des pétioles qu'au P des autres parties de la plante (tige, feuilles entières, folioles ou tubercules). Le suivi de la concentration en P des pétioles pendant le cycle de croissance permet d'avoir une idée sur l'état de suffisance de cet élément dans la plante. Westermann et Kleinkopf (1985) ont montré que le bon développement des plants et des tubercules a été assuré lorsque le seuil de suffisance en P dans les pétioles était $\geq 1.0 \text{ g de P kg}^{-1}$. Il demeure important de préciser que la concentration en nutriments dans les tissus végétaux, notamment dans les pétioles, est variable en fonction de plusieurs facteurs: stade de croissance de la plante, variété de pomme de terre, et méthode d'analyse (Rosen et al., 2014). Selon Walworth et Muniz (1993), le seuil de suffisance en P dans les pétioles était entre 1.5 et 3.1 g P kg⁻¹. Plus tard, Freeman et al. (1998) ont déterminé un seuil critique de suffisance suivant le stade de croissance des tubercules de la variété Russet Burbank: de 4.5 à 5.7 g P kg⁻¹ au stade 5-10 mm, de 3.5 à 4.7 g P kg⁻¹ au stade 35-45 mm et de 2.1 à 2.6 g P kg⁻¹ au stade 75-85 mm. De leur côté, Stark et Love (2003) ont proposé un seuil de suffisance $\geq 2.2 \text{ g P kg}^{-1}$.

1.2.4. Grilles de fertilisation phosphatée

Les indicateurs agronomiques servent à mieux comprendre les besoins des cultures et, par conséquent, à mieux gérer les recommandations en engrais phosphatés.

Les modèles de recommandations en P sont établis en quatre étapes : la corrélation, l'étalonnage ou calibration, l'optimisation et la discréétisation.

1.2.4.1. La corrélation

Afin d'élaborer un modèle de recommandations, il faut d'abord sélectionner la méthode d'analyse la plus utile pour mesurer les niveaux d'éléments nutritifs dans le sol. Pour le P, tous les modèles de recommandation ont été établis en se basant sur la teneur des sols en cet élément (Sbih et Khiari, 2005). Cependant, la méthode d'extraction du P diffère d'une région à une autre. À titre d'exemple et comme précédemment mentionné, la méthode Olsen est couramment utilisée au Manitoba et en Ontario. Au Québec et dans les Maritimes, c'est l'extraction au Mehlich-3 qui est privilégiée. Les indices $(P/Al)_{M3}$ et $(P)_{M3}/(Al+Fe)_{M3}$ ont montré une meilleure efficacité que le P_{M3} dans la prédition de la réponse des cultures à la fertilisation phosphatée et dans la prévention du risque environnemental de perte du P par ruissellement ou par lessivage au Québec (Khiari et al., 2000; Pellerin et al., 2006a) et aux États de la Mi-Atlantique (Maguire et Sims, 2002b; Sims et al., 2002). En effet, selon Giroux et Tran (1985), une bonne évaluation de la disponibilité du P dans le sol en vue de bien contrôler sa fertilisation doit tenir compte des éléments responsables de sa rétention (essentiellement l'Al pour les sols du Québec). L'indice actuellement utilisé pour la grille de fertilisation phosphatée de la pomme de terre au Québec est le $(P/Al)_{M3}$. Khiari et al. (2000) ont montré que la mise en commun du P_{M3} et de l' Al_{M3} sous forme d'un rapport décrivait mieux la réponse de la culture aux engrains phosphatés. Le risque environnemental de perte du P tel que décrit par le P extrait à l'eau (P_w) était mieux relié au $(P/Al)_{M3}$ qu'à la simple détermination du P_{M3} (Fig. 1.2). En plus de la pomme de terre, $(P/Al)_{M3}$ a été utilisé comme indicateur agronomique pour le maïs (Pellerin et al, 2006b) et pour la canneberge (Parent et Marchand, 2006) au Québec.

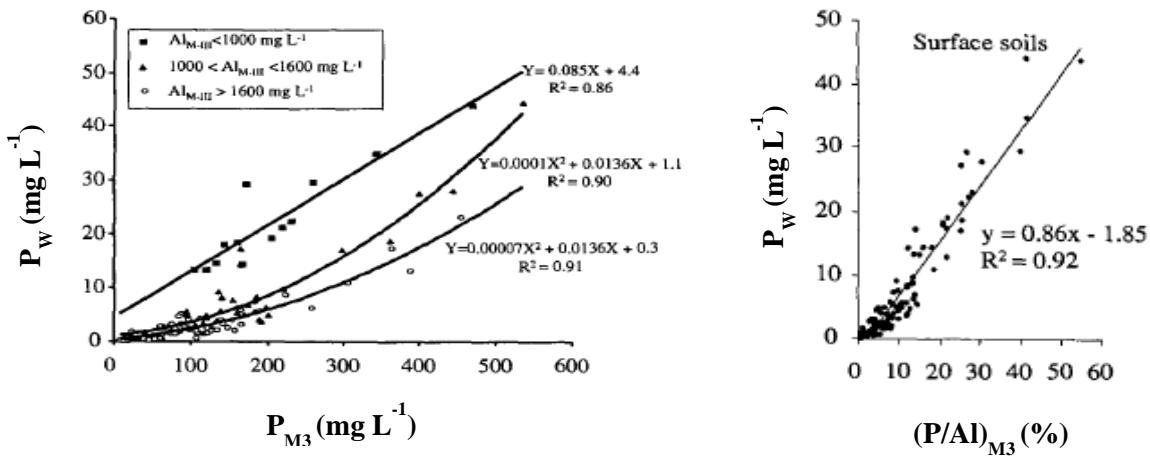


Figure 1.2. Relation entre le P désorbé (P_w), le P_{M3} et le $(P/Al)_{M3}$ dans des sols du Québec (Tiré et adapté de Khiari et al., 2000)

1.2.4.2. L'étalonnage ou calibration

Cette étape consiste à regrouper les sols ayant réagi de la même manière à l'application d'engrais dans une même classe de fertilité. Les intervalles qui délimitent les classes de fertilité sont déterminés à partir de seuils agronomiques et environnementaux. Le seuil agronomique est une valeur critique basée sur l'indice de fertilité au-delà duquel les rendements relatifs se stabilisent au-dessus de 80%. Il est obtenu d'une façon itérative selon la méthode de partition de Cate-Nelson (Cate et Nelson 1965; Nelson et Anderson, 1984) en exprimant dans un graphique les rendements relatifs (Giroux et al., 1984) obtenus dans les différents sites en fonction de l'indice de fertilité précédemment choisi dans l'étape de la corrélation. Le rendement relatif renseigne sur la capacité d'un site à répondre à l'ajout de l'engrais. Ainsi, si le rendement relatif est inférieur à 100%, on conclut que le site répond à l'engrais. Par contre, un rendement relatif supérieur ou égal à 100% indique que la culture ne répond pas à l'ajout d'engrais. Le rendement relatif est exprimé comme suit (Éq. 1):

$$\text{Rendement relatif (\%)} = \frac{\text{Rendement témoin (sans engrais P)}}{\text{Rendement maximal obtenu sur le même site}} \times 100 \quad (1)$$

La figure 1.3. illustre un exemple d'étude où la partition de Cate-Nelson a été appliquée pour évaluer le rendement relatif de 78 sites de pomme de terre au Québec en fonction de l'indice de fertilité de leurs sols exprimés sous forme de $(P/Al)_{M3}$. Selon cette figure, le seuil agronomique est $(P/Al)_{M3} = 7.3\%$ lorsque P_{M3} et Al_{M3} sont dosés par colorimétrie et par spectrophotométrie d'absorption atomique, respectivement. Cette valeur correspond à un $(P/Al)_{M3} = 8\%$ si le dosage est fait par spectrophotométrie d'émission par plasma (Voir section 1.4.2).

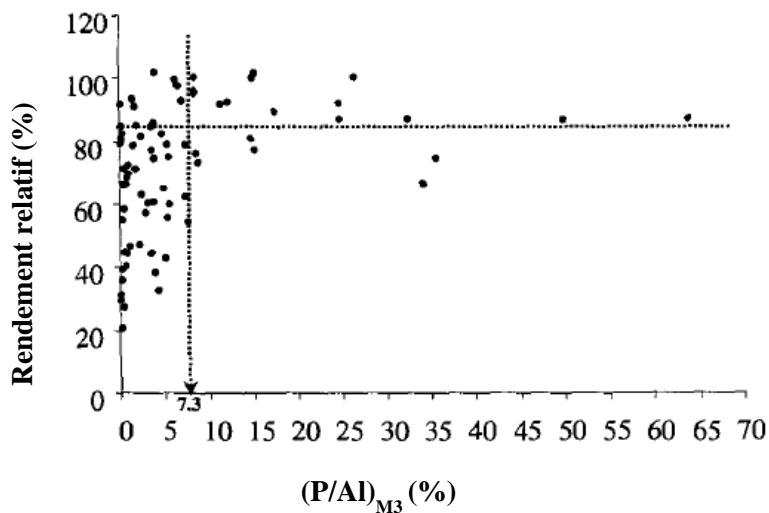


Figure 1.3. Relation entre le rendement relatif de pomme de terre et l'indice de fertilité $(P/Al)_{M3}$ pour des sols du Québec (Tiré et adapté de Khiari et al., 2000)

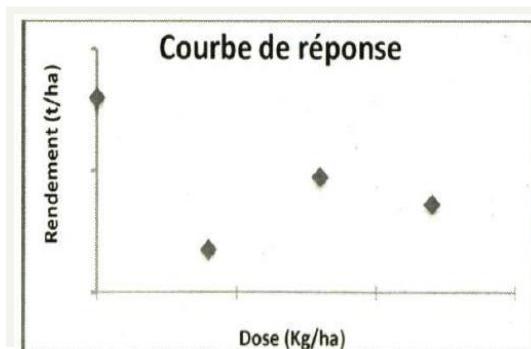
Le seuil environnemental est une valeur critique au-delà de laquelle le risque de perte du P dans les cours d'eau devient élevé (Breeuwsma et Silva, 1992). Au Québec, Pellerin et al. (2006a) ont établi des seuils environnementaux selon le groupe textural : $(P/Al)_{M3} = 7.6\%$ pour les sols de texture fine et $(P/Al)_{M3} = 13.1\%$ pour les sols de texture grossière. Pour les sols de pomme de terre (% argile < 20%), le seuil environnemental est $(P/Al)_{M3} = 15\%$ (Khiari et al., 2000). Ce seuil correspond à la limite supérieure de la classe de fertilité et de risque environnemental moyens. Le seuil agronomique de 8% correspond plutôt à la limite inférieure de la classe de risque moyen.

1.2.4.3.L'optimisation

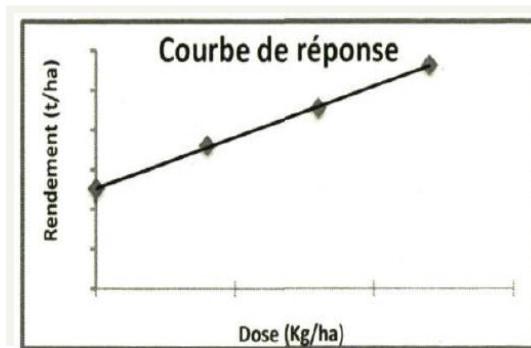
L'optimisation a pour objectif de déterminer la dose optimale de l'engrais dans chacun des sites. Cette dose est obtenue en étudiant les modèles de réponse mettant en relation les rendements vendables obtenus en fonction des doses appliquées (Samson et al., 2008). Selon la réponse de la culture aux doses d'engrais appliquées, on peut observer différents types de modèles. Le choix de l'un ou l'autre de ces modèles doit être fait de façon à bien décrire la réponse de la culture. Le coefficient de détermination (R^2) pourrait être considéré pour faire le choix du modèle (Cerrato et Blackmer, 1990).

Parmi les réponses couramment observées on retrouve (Samson et al., 2008) :

Aucune réponse : dans ce cas les rendements obtenus avec ou sans engrais ne sont pas significativement différents. La dose optimale retenue est de 0 kg ha^{-1} .



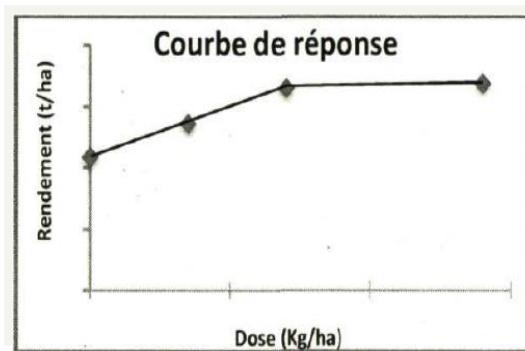
Réponse linéaire : rencontrée dans les sols dont le niveau de fertilité n'est pas élevé. La culture continue à répondre à l'engrais au fur et à mesure que la dose augmente. La dose optimale correspond à la dose maximale.



Réponse linéaire plateau : On doit tout d'abord vérifier que la pente de la partie linéaire ascendante correspond à l'inéquation suivante:

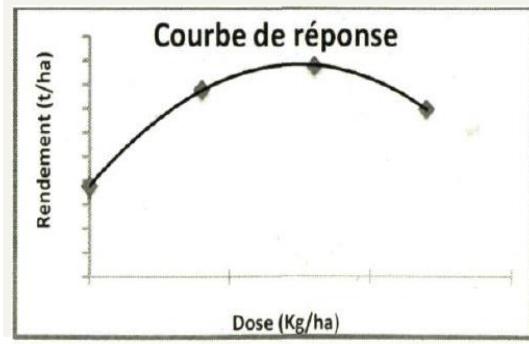
$$\frac{\Delta \text{ Rendement (kg ha}^{-1})}{\Delta \text{ Dose (kg ha}^{-1})} > \frac{\text{Prix de l'engrais (\$ kg}^{-1})}{\text{Prix de la récolte (\$ kg}^{-1})}$$

Si cette condition est respectée, la dose économique optimale est celle qui définit l'intersection entre les deux droites de la courbe de réponse. C'est en effet la plus petite dose pour atteindre un rendement optimal stable (Cerrato et Blackmer, 1990). Sinon, on déclare que la culture ne répond pas à l'engrais.

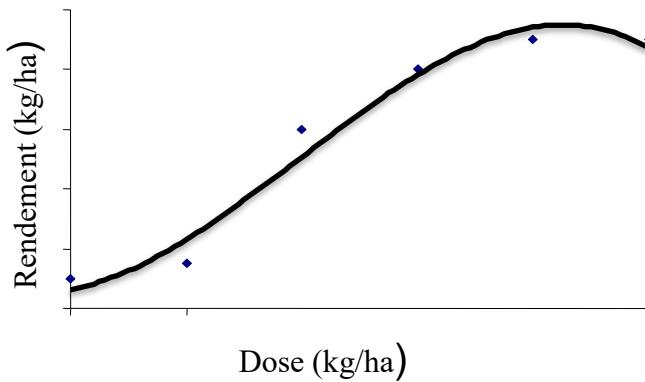


Réponse quadratique : le rendement augmente d'une façon moins que proportionnelle avec la dose de l'engrais jusqu'à un maximum. Tout de suite après ce maximum, on assiste à une déclinaison des rendements en fonction de la dose. Dans cette situation, la dose optimale est calculée en considérant le prix unitaire de l'engrais et celui de la récolte. Étant donné que les modèles quadratiques ont tendance à surestimer la dose économique optimale de P, Nelson et al. (1985) ont proposé que cette dose doit être déterminée en résolvant l'égalité entre la dérivée première de l'équation définissant le modèle de réponse et le rapport du prix de l'engrais par rapport à celui de la récolte (Éq. 2):

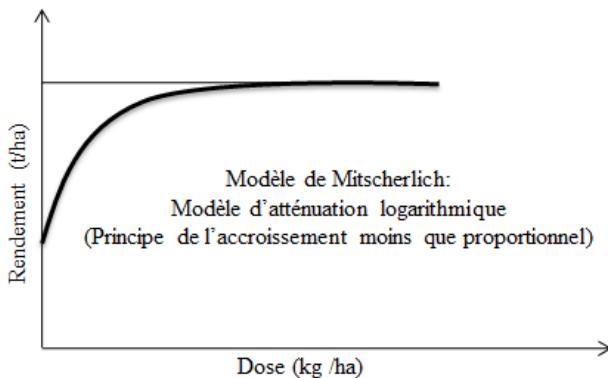
$$\frac{\delta \text{ Rendement (kg ha}^{-1})}{\delta \text{ Dose (kg ha}^{-1})} = \frac{\text{Prix de l'engrais (\$ kg}^{-1})}{\text{Prix de la récolte (\$ kg}^{-1})} \quad (2)$$



Réponse cubique: La réponse cubique est exprimée selon le polynôme $Y = ax^3 + bx^2 + cx + d = 0$ et un point d'infexion $-b/3a$ séparant deux patrons de concavité différents, généralement concaves ou plus que proportionnel pour des faibles doses et convexe ou moins que proportionnel pour des doses supérieures.



Modèle de Mitscherlich : Le modèle de Mitscherlich est parmi les modèles les plus couramment utilisés pour décrire la réponse des cultures à la fertilisation. Selon Gros (1960), la relation entre l'apport en engrais et le rendement est décrite selon la loi de Mitscherlich comme suit: «au fur et à mesure que la dose d'engrais utilisée s'élève, le supplément de récolte obtenu pour chaque unité fertilisante supplémentaires diminue jusqu'à devenir nul ou même négatif». Ceci signifie que l'augmentation des rendements est de moins en moins importante au fur et à mesure que les doses du fertilisant apporté augmente. Autrement dit, lorsque la réponse de la culture atteint son maximum, tout apport supplémentaire en fertilisant devient économiquement désavantageux.



1.2.4.4. La discréétisation

L'objectif de cette étape est de proposer une dose optimale commune valable pour tous les sols appartenant à un même groupe de fertilité phosphatée telle que mesurée par l'indicateur agronomique choisi afin d'établir le modèle de recommandation. La dose à recommander pour un groupe de fertilité donné peut être déterminée à l'aide des statistiques descriptives lorsque les données d'entrée ne sont pas volumineuses. Pour ce faire, les doses optimales correspondant aux sols ayant le même niveau de fertilité sont regroupées et classées par ordre croissant. La dose à recommander pourrait correspondre à l'une des tendances centrales suivantes: moyenne, mode ou médiane (50^{ème} centile), ou à d'autres centiles d'espérance conditionnelle (par exemple, 80^{ème} centile) (Isaaks et Srivastava, 1989; Khiari et al., 2000). Le 80^{ème} centile des doses optimales croissantes signifie que 80% des doses optimales dans la classe de fertilité en question sont inférieures ou égales à la dose de recommandation retenue (Pellerin et al., 2006b).

La figure suivante illustre la dispersion des doses optimales en P sur des sites de pomme de terre ainsi que le modèle de recommandation en P couvrant 50% et 80% des cas étudiés (Khiari et al., 2000).

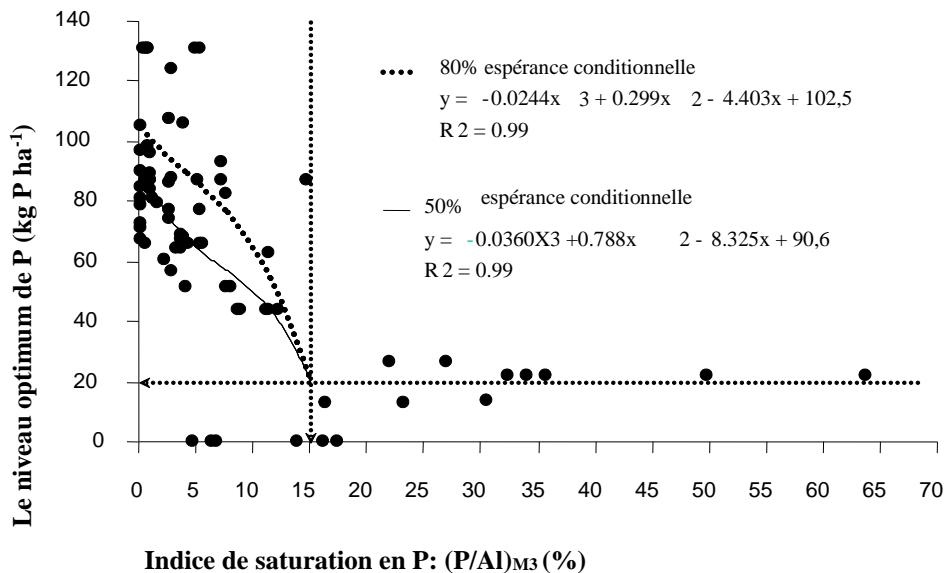


Figure 1.4. Relation entre $(P/Al)_{M3}$ et les doses optimales en P pour élaborer les modèles de recommandation pour la pomme de terre au Québec (Khiari et al., 2000)

1.2.4.5. Autres méthodes d’élaboration des modèles de recommandations

Les approches basées uniquement sur les rendements dans des sites spécifiques ne fournissent pas forcément de l’information fiable pour la détermination des recommandations (Kitchen et al., 2008). Ainsi, les doses recommandées obtenues à partir des modèles mathématiques décrits plus haut peuvent être assez imprécises et varier considérablement selon le modèle choisi (Kyveryga et al., 2007; Naud et al., 2008). De plus, plusieurs facteurs tels que les propriétés des sols, la température et les précipitations, peuvent avoir une influence sur la réponse aux engrains et sur la croissance des cultures (Anwar et al., 2009). Il en résulte que même si les essais sur le terrain peuvent identifier les doses optimales à recommander dans un site spécifique, ces mêmes doses ne peuvent pas être généralisées à des conditions différentes de celles du site en question (Kyveryga et al., 2007). Les méta-analyses (Valkama et al., 2009; Tremblay et al., 2012) et les modèles multi-niveaux (Parents et al., 2017) sont des méthodes qui combinent les résultats d’un grand nombre d’essais de fertilisation et qui tiennent compte du maximum de facteurs pouvant influencer la réponse de la culture aux engrains. Grâce aux outils d’apprentissage automatique, ces méthodes sont capables de générer des modèles de recommandations robustes et qui sont ajustées selon les spécificités locales de chaque site (Parent et al., 2017).

1.2.5. Engrais phosphatés de synthèse et de recyclage

1.2.5.1. Engrais de synthèse purement phosphatés

Les engrais phosphatés conventionnels sont tous issus du traitement du phosphate naturel par différents procédés. Les engrais phosphatés minéraux simples couramment utilisés en agriculture sont le superphosphate simple (SSP) et le triple superphosphate (TSP) produits, respectivement, du traitement du phosphate naturel par de l'acide sulfurique et de l'acide phosphorique. Le SSP contient de 16 à 22% de P₂O₅ soluble au citrate d'ammonium neutre (CAN). Environ 90% du P de cet engrais est soluble à l'eau. Le TSP contient de 44 à 52% de P₂O₅ (19 à 23% de P). Le P présent dans cet engrais est sous forme de phosphate monocalcique (Ca(H₂PO₄)₂) soluble à 95-98% dans l'eau (Havlin et al., 1999).

La grande solubilité de ces engrais phosphatés minéraux assure aux plantes leurs besoins en P. Cependant, une partie du P en excès (Khiari et Parent, 2005) peut se retrouver dans les eaux environnantes et perturber ainsi leur équilibre écologique (Liu et Chen, 2008).

La grande dépendance de l'agriculture à la fertilisation phosphatée minérale, la diminution des réserves des roches phosphatées, et les risques environnementaux liés à leur utilisation ont suscité un intérêt pour la récupération du P provenant des matières résiduelles fertilisantes (MRF).

1.2.5.2. Engrais phosphatés recyclés à partir des boues d'épuration

Les boues d'épuration sont un des principaux types de MRF. Ils proviennent du traitement des eaux usées, municipales ou industrielles riches en phosphore. Le P y est présent sous forme de struvite (NH₄MgPO₄ · 6H₂O) (Johnston et Richards, 2003). Même si elle contient une quantité importante d'azote et de magnésium, la struvite est considérée comme étant un engrais phosphaté. La struvite peut être récupérée moyennant différentes approches. Parmi ces approches on retrouve la technologie Pearl brevetée aux États-Unis et mise au point par l'Université de la Colombie-Britannique, au Canada. Cette technologie convient au traitement en aval des effluents contenant de fortes concentrations de phosphore et d'ammonium et permet de récupérer la struvite dans un réacteur à lit fluidisé. Les performances de la technologie ont également été modifiées par l'incorporation d'un procédé supplémentaire (WASSTRIP), dans lequel le phosphate est extrait des boues activées dans

une zone anaérobie avant la digestion, et ajouté à l'eau de rejet. Le procédé a une efficacité de récupération moyenne de 80% pour le phosphate et de 10-15% pour l'azote avec une production moyenne de 600 tonnes par an. Le produit de ce traitement est commercialisé sous le nom de Crystal Green® (5-28-0+10% Mg) (Kataki et al., 2016).

Contrairement aux engrains P conventionnels, la struvite est très peu soluble dans l'eau (Cabeza et al., 2011). La libération du P de la struvite est fortement liée aux acides organiques sécrétés par les exsudas racinaires (Talboys et al., 2016). Elle est considérée comme étant un engrais P à libération lente qui émet le P nécessaire à la plante au besoin et tout au long du cycle de croissance. Son utilisation pourrait donc réduire l'accumulation du P dans les sols agricoles, limitant ainsi les risques de contamination des eaux environnantes (Zhang et al., 2012). De plus, la struvite pourrait constituer une source de remplacement efficace pour les phosphates naturels. En effet, la récupération de la struvite à partir de boues d'épuration municipales à l'échelle mondiale devrait réduire d'environ 1,6% les activités d'extraction de phosphates dans le monde (Shu et al., 2006).

Ainsi, l'intégration d'un tel processus de récupération à partir du traitement des eaux usées contribuerait à la valorisation des éléments nutritifs en excès en transformant les MRF en matières fertilisantes (Burns et Moody, 2002) d'un côté, et à réduire la problématique de l'eutrophisation d'un autre côté.

1.3 Diagnostic environnemental du phosphore dans le sol

Les indices environnementaux du P ont été développés en se basant sur le fait que la désorption du P serait plus importante lorsque la quantité du P biodisponible augmente dans le sol. Ceci se traduit par une augmentation du P dans la solution du sol, réduisant d'une part le risque agronomique et accentuant d'autre part le risque environnemental de perte du P dans les eaux de ruissellement ou de lixiviation (Sharpley, 1995).

1.3.1. Indices environnementaux du P dans les sols

1.3.1.1. Le degré de saturation en P (DSP)

Le degré de saturation en P (DSP) fut l'un des premiers indices de la mobilité potentielle du P dans le sol. Il a été développé pour la première fois par van der Zee et al. (1987) et Breeuwsm et Silva (1992) pour les terres acides sableuses basses frontalières de

la mer du Nord (Hollande, Belgique et Allemagne), souvent surfertilisées en phosphore. Ces auteurs se sont basés sur le fait que dans les sols acides à neutres, la capacité de rétention des phosphates dépend essentiellement de leur contenu en oxydes et hydroxydes de fer (Fe) et d'aluminium (Al) extractibles à l'oxalate acide d'ammonium : $(Fe)_{ox}$ et $(Al)_{ox}$ (Khiari et al., 2000). Ces éléments représentent principalement les formes amorphes d'oxyde et d'hydroxyde de Fe et d'Al actives dans le processus de sorption du P biodisponible.

L'approche hollandaise est basée sur le principe que la fixation du P dans les sols non calcaires est la somme de deux réactions: une réaction d'adsorption rapide et réversible, et une réaction lente d'immobilisation irréversible (van der Zee et al., 1987).

La capacité maximale de sorption du P (CSP, en mmol kg^{-1}), réversible et irréversible, a été décrite, par les mêmes auteurs, comme étant la quantité maximale de P fixée suite à des réactions rapides ou lentes selon la relation suivante (Éq. 3):

$$CSP = b + S_m \quad (3)$$

Où b : la capacité maximale d'adsorption réversible du P selon l'équation de Langmuir ($\text{mmol P adsorbé kg}^{-1}$ de sol).

S_m : maximum de rétention de P dû aux réactions lentes ($\text{mmol P fixé kg}^{-1}$ de sol).

Or, pour les réactions lentes, il est difficile d'atteindre l'équilibre au laboratoire et dans un temps relativement court. van der Zee et al., (1987) ont donc démontré que la CSP peut être directement reliée à la somme de fer et d'aluminium extraits par l'oxalate acide d'ammonium selon la relation suivante (Éq. 4) :

$$CSP = \alpha_m (Fe_{ox} + Al_{ox}) \quad (4)$$

avec α_m : coefficient de fixation maximale (van der Zee et al., 1987, Breeuwsma et Silva, 1992). Lookman et al. (1995) ont défini α_m comme étant la fraction des sites réactifs de la phase solide du sol, représentée par le contenu en Al et Fe extractibles à l'oxalate, disponible pour fixer le P.

Le Fe_{ox} et Al_{ox} sont reliés aux formes amorphes d'oxydes et hydroxydes de Fe et d'Al et sont très actifs dans le processus d'adsorption rapide du P. La capacité maximale de sorption réversible (b) a été donc reliée au contenu en Fe_{ox} et Al_{ox} (van der Zee et al., 1987) (Éq. 5) :

$$b = \beta_m (\text{Fe}_{\text{ox}} + \text{Al}_{\text{ox}}) \quad (5)$$

où β_m est un coefficient maximal d'adsorption réversible.

Les mêmes travaux de van der Zee et al. (1987) ont montré que la relation qui existe entre α_m et β_m pour les sols sableux acides est comme suit (Éq. 6):

$$\alpha_m = 3\beta_m \quad (6)$$

En 1992, Breeuwsma et Silva ont rapporté que le DSP peut être défini comme étant le rapport reliant le P retenu sur les oxydes et hydroxydes de fer et d'aluminium et extrait à l'oxalate acide d'ammonium (P_{ox} exprimé en mmol kg^{-1} et qui représente une approximation de la quantité totale de P fixé) à la capacité totale de sorption du P, CSP (mmol kg^{-1}) (Éq. 7).

$$\text{DSP} (\%) = \frac{P_{\text{ox}}}{\text{CSP}} \times 100 \quad (7)$$

Selon les équations 4, 5 et 6, l'équation 7 peut alors s'écrire de deux autres façons (Éq. 8) et (Éq. 9) :

$$\text{DSP} = \frac{P_{\text{ox}}}{3b} \quad (8)$$

ou

$$\text{DSP} = \frac{P_{\text{ox}}}{\alpha_m (\text{Fe}_{\text{ox}} + \text{Al}_{\text{ox}})} \quad (9)$$

Les valeurs de α_m rapportées dans la littérature sont très variables. Pour les analyses courantes, Breeuwsma et Silva (1992) ont proposé une valeur moyenne $\alpha_m = 0.5$ et le DSP est défini selon l'équation suivante (Éq. 10):

$$\text{DSP} = \frac{P_{\text{ox}}}{0.5 (\text{Fe}_{\text{ox}} + \text{Al}_{\text{ox}})} \quad (10)$$

Ce $\alpha_m=0.5$ a été estimé en calculant la moyenne de α_m qui variaient de 0.4 à 0.6 rapportés respectivement par Schoumans et al. (1987) et par van der Zee et Riemsdijk (1988). Cependant, van der Zee et al. (1987) ont été les premiers à avoir proposé un $\alpha_m=0.6$ pour les sols podzoliques acides hollandais et à introduire le concept du DSP dans leur réglementation agricole et environnementale (Beauchemin et Simard, 1999). En effet, pour limiter la diffusion du P dans les eaux de surface et souterraines, la teneur en P sous forme d'orthophosphate ne doit pas dépasser 0.10 mg L^{-1} dans les eaux souterraines (van der Zee et al., 1987; van der Zee et al., 1990; Breeuwsma et Reijerink, 1992; Breeuwsma et Silva, 1992). Une valeur critique de DSP de 25% a été établie pour les sols sableux acides non calcaires (van der Zee et al., 1990; Breeuwsma et Silva, 1992) au-delà de laquelle le risque de contamination de l'eau par le P augmente et à partir de laquelle les apports en P ne doivent pas dépasser les besoins des cultures.

Le concept du degré de saturation en phosphore (DSP) est particulièrement significatif, car il tient en considération ces deux aspects: il donne une mesure de l'intensité de l'accumulation du P en considérant la capacité du sol à fixer le P, et il décrit le potentiel du P à se désorber de la phase solide du sol vers sa phase liquide (Sharpley, 1995).

1.3.1.2. Indice de désorption du P dans les sols

Le risque de contamination des eaux par le phosphore est évalué par sa concentration ou sa saturation dans le sol. En effet, la solubilisation et la perte du phosphore augmente rapidement avec l'accumulation du phosphore disponible (Maguire et Sims, 2002a). Ainsi, une fertilisation phosphatée organique ou minérale peut garantir de meilleurs rendements, mais peut aussi augmenter le risque environnemental de dissipation de P (eutrophisation).

Le P extrait à l'eau (P_w) (Sissingh, 1971) est l'un des meilleurs indicateurs de prédiction de la quantité potentielle de phosphore désorbée lors des événements pluvieux et perdue par ruissellement (Sharpley et al., 1996; Pellerin, 2005) ou par lixiviation (Maguire et Sims, 2002a). Le phénomène de désorption dépend, entre autres, de la texture du sol (Hooda et al., 2000). Il a été démontré au Québec que pour la même saturation en P, les sols de texture plus argileuse désorbent plus de P à l'eau que les sols les moins argileux (Pellerin et al., 2006a). Au Québec, une valeur critique de 9.7 mg L^{-1} en terme de P désorbé à l'eau et correspondant au DSP critique de 25% a été établie par Khiari et al., (2000). Cette valeur

critique est à l'origine des règlements sur les exploitations agricoles et de toutes les valeurs critiques environnementales de saturation des sols au Québec, quelle que soit leur nature (minérale ou organique) et leur texture (G1a : argile lourde, G1b : sols à texture fine (argileux), G2 : sols à texture moyenne (loameux), ou G3 : sols à texture grossière (sableux)) (Pellerin et al., 2006b; CRAAQ, 2010).

Il existe d'autres indicateurs de désorption du P tels que le P Self-Davis (procédure d'extraction à l'eau désionisée ou au CaCl_2) (Self-Davis et al., 2000) utilisé surtout pour les sols des États américains et le P-FeO extrait à l'aide de bandelettes enrichies d'hydroxyde de fer (van der Zee et al., 1987).

1.3.2. Indices environnementaux du P dans les eaux de dissipation

La perte du P de la terre vers les eaux peut se faire à travers plusieurs voies. On a longtemps considéré que la perte du P particulaire par érosion, ou celle du P dissous dans les eaux de ruissellement lors des événements pluvieux, sont les voies les plus importantes de perte du P des terres agricoles (Sims et al., 1998). Plus tard, plusieurs auteurs ont révélé l'importance de la perte du P par lixiviation, surtout dans les sols sableux ou dans les sols où l'activité des vers de terre et les fissures favorisent le drainage (Maguire et Sims., 2002a, Sims et al., 1998).

En général, les eaux de dissipation contiennent le P sous différentes formes. Le P peut se trouver sous forme dissoute ($< 0.45\mu\text{m}$), essentiellement les ions orthophosphates qui sont immédiatement disponibles pour les algues. S'il est retenu sur les particules de sol, on dit que le P est sous forme particulaire ($> 0.45\mu\text{m}$). Les deux formes, dissoute et particulaire, peuvent être réactives ou non réactives (Sims et Kleinman, 2005) (Figure 1.5).

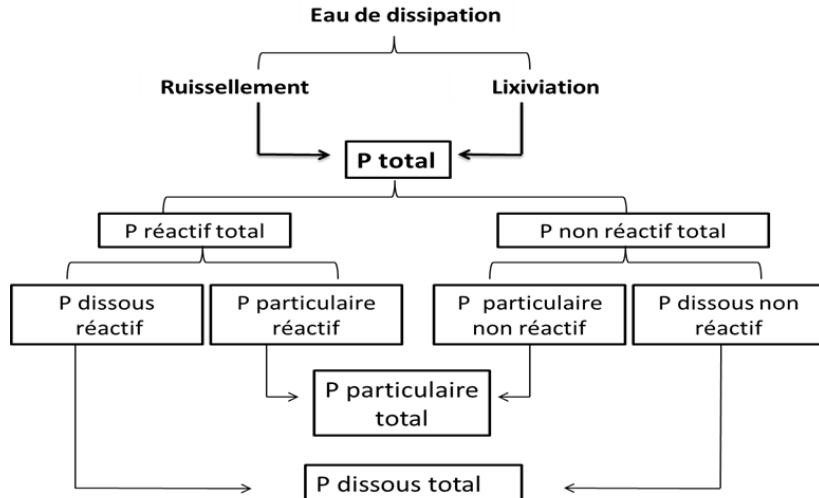


Figure 1.5. Différentes formes du P dans l'eau de dissipation

Des études ont été menées dans le but de prévoir les pertes potentielles du P par ruissellement et par lixiviation. Au Delaware, Maguire et Sims (2002a) ont effectué des tests de lixiviation de P sur 111 sols acides de texture grossière. Dans le même contexte, Sims et al. (2002) ont sélectionné des échantillons appartenant à cinq séries de sols différentes sur lesquels ils ont simulé des tests de ruissellement et de drainage. Ces travaux ont permis de construire un modèle simulant le risque d'accumulation du P dans les eaux de dissipation, ou le phosphore réactif dissous (DRP), selon les deux voies de ruissellement et de drainage.

Les indicateurs environnementaux requièrent des analyses longues, fastidieuses et exigeantes pour être déterminés. Ils ont été substitués par des indicateurs agroenvironnementaux d'interprétation à la fois agronomique (reliés aux critères de performance des cultures) et environnementale (reliés à la saturation des sols en phosphore et leur impact sur la pollution diffuse).

1.4 Indicateurs agro-environnementaux du P

1.4.1. Les indices de saturation des sols en P (ISP)

En se basant sur le même principe de DSP impliquant les mécanismes de rétention et de désorption du P par les sols, plusieurs études ont été menées afin de développer une

approche rapide et efficace qui utilise des méthodes d'extraction routinières et qui pourrait servir comme outil de prévoyance des risques de perte du P (Sharpley, 1995; Giroux et Tran, 1996).

Les travaux de Khiari et al. (2000) portant sur les sols acides de texture grossière du Québec ont abouti au développement d'un indice de saturation en phosphore (ISP) basé sur l'extraction au Mehlich-3, méthode d'analyse de routine adoptée au Québec et dans les provinces de l'Est canadien depuis 1989. L'ISP est calculé en considérant le rapport, molaire ou massique, entre les contenus en P, Al et Fe extraits au Mehlich-3. Il s'exprime généralement selon l'équation suivante (Éq. 11) :

$$\text{ISP} = \frac{\text{P}_{\text{M3}}}{[\text{Al} + \gamma\text{Fe}]_{\text{M3}}} \quad (11)$$

Khiari et al. (2000) et Maguire et Sims (2002b) ont trouvé que le Fe_{M3} ne représente qu'environ 12% et 9%, respectivement, de la somme $(\text{Al}+\text{Fe})_{\text{M3}}$ calculée sur base molaire. En effet, Solis et Torrent (1989) avaient montré que la solution Mehlich-3 n'est pas aussi efficace que la méthode à l'oxalate dans l'extraction de Fe. Pour cette raison, le Fe extractible au Mehlich-3 pourrait nécessiter un coefficient de pondération γ afin de prendre en considération l'efficacité de l'extraction, particulièrement dans les sols où le Fe joue un rôle plus important que l'Al dans la fixation du P.

Dans les sols minéraux du Québec, Khiari et al. (2000) et Pellerin et al. (2006a,b) ont utilisé $\gamma=0$ et l'ISP a été exprimé comme suit (Éq. 12):

$$\text{ISP}_1 = \frac{\text{P}_{\text{M3}} (\text{mg kg}^{-1})}{\text{Al}_{\text{M3}} (\text{mg kg}^{-1})} \quad (12)$$

D'autres auteurs ont considéré $\gamma=1$ pour les sols minéraux de la région Mi-Atlantique (Maguire et Sims, 2002b; Sims et al., 2002) et pour les sols sableux acides de canneberge du Québec (Parent et Marchand, 2006). L'ISP a donc été calculé en tenant compte du rôle du Fe dans la fixation du P dans le sol (Éq. 13):

$$\text{ISP}_2 = \frac{\text{P}_{\text{M3}} (\text{mmol kg}^{-1})}{[\text{Al}(\text{mmol kg}^{-1}) + \text{Fe}(\text{mmol kg}^{-1})]_{\text{M3}}} \quad (13)$$

Dans les sols organiques, Guérin et al. (2007) ont considéré un coefficient de pondération $\gamma=5$ afin de compenser l'inefficacité de la solution Mehlich-3 dans l'extraction du fer. L'expression de l'ISP devient comme suit (Éq. 14):

$$ISP_3 = \frac{P_{M3}(\text{mmol kg}^{-1})}{[Al(\text{mmol kg}^{-1}) + 5 \times Fe(\text{mmol kg}^{-1})]_{M3}} \quad (14)$$

Dans une étude sur des sols minéraux avec une teneur élevée en matière organique du Québec, Leblanc et al. (2013) ont défini $\gamma=3$, une valeur qui se situe entre $\gamma=1$ et $\gamma=5$ retenues, respectivement, pour les sols minéraux et les sols organiques.

Les indices ISP_1 et ISP_2 sont fortement corrélés entre eux (Khiari et al., 2000, Maguire et Sims, 2002b). Le lien qui a été établi entre ces deux indices est le suivant (Khiari et al., 2000) (Éq. 15):

$$ISP_1 = 1.34 \times ISP_2, \text{ où } R^2 = 0.99 \quad (15)$$

En effet, selon les résultats de recherche de Khiari et al. (2000), il s'est avéré que la présence du Fe dans le calcul de l'ISP n'est pas nécessaire, car il ne donne aucune amélioration significative dans la relation statistique entre le P soluble et la saturation du sol en P. Par contre, Sims et al., (2002) ont suggéré que dans les sols du Mi-Atlantique américain, le Fe amorphe joue un rôle considérable dans le mécanisme de rétention du P dans le sol et sa diffusion par ruissellement ou lessivage. Ils ont donc gardé le Fe extrait au Mehlich-3 dans l'équation d'estimation de la capacité de rétention du P et ils ont retenu l' ISP_2 plutôt que l' ISP_1 comme indicateur agroenvironnemental de saturation des sols en P.

L'indice de saturation des sols en phosphore est un indicateur agro-environnemental particulièrement intéressant. En prenant en considération les besoins phosphatés des cultures, l'ISP permet d'identifier le seuil agronomique à partir duquel on évalue la probabilité de réponse des cultures à l'application d'engrais. Il permet également de prévenir les risques environnementaux en identifiant le seuil environnemental au-delà duquel les apports en engrains phosphatés doivent se limiter aux besoins de la culture (Khiari et al., 2000).

Par analogie avec les sols acides, d'autres indicateurs de la saturation en P ont été développés. Il s'agit de la formule [P analysé / (b ou ISP_{18h})] traduisant le rapport entre le P extrait par une méthode normalisée (P-Mehlich, P-Olsen, P-FeO, P-Bray, P_w) et la capacité maximale d'adsorption à court terme (le b de Langmuir) ou l'indice d'adsorption à court terme (ISP_{18h}).

1.4.2. Considérations sur les unités et les méthodes de dosage pour les ISP

L'ISP₁ est calculé sur base massique d'unités courantes, soit en mg kg⁻¹ de sol, mg L⁻¹ de sol si on utilise une cuillère de laboratoire, ou en kg ha⁻¹. Lorsque le Fe est considéré dans le calcul de l'indice de saturation en P, l'ISP est calculé sur une base molaire (mmol kg⁻¹). Pour l'ISP₁, le plus utilisé au Québec, le dosage du P et de l'Al extraits par Mehlich-3 (P_{M3} et Al_{M3}) peut se faire selon deux méthodes: méthode Colorimétrie-SAA où le P est dosé par colorimétrie et l'Al par spectrophotométrie d'absorption atomique (SAA) et la méthode SEP (spectrométrie d'émission par plasma) qui permet de doser consécutivement le P et l'Al. D'ailleurs, la majorité des laboratoires recourent à la SEP. La méthode de dosage par Colorimétrie-SAA ne concerne que les formes minérales du P dissous (les orthophosphates H₂PO₄⁻ et HPO₄²⁻) alors que le dosage par SEP renseigne sur le P total dissous dans la solution Mehlich-3 (les orthophosphates H₂PO₄⁻ et HPO₄²⁻ ainsi que le P organique). Le P organique représente environ 9% du P total. Khiari et al., (2000) ont établi le lien entre ces deux méthodes de dosage (Éq. 16) et (Éq. 17):

$$P_{M3} \text{ (SEP)} = 1.09 P_{M3} \text{ (C)} \quad (16)$$

$$\frac{P_{M3}}{Al_{M3}} \text{ (SEP)} = 1.12 \frac{P_{M3}}{Al_{M3}} \text{ (Colorimétrie-SAA)} \quad (R^2 = 0.99; n=100) \quad (17)$$

1.5 Valeurs critiques des indicateurs environnementaux de P

Plusieurs auteurs ont démontré que l'ISP est fortement corrélé au DSP (Khiari et al., 2000; Sims et al., 2002; Maguire et Sims, 2002b; Pellerin et al., 2006a; Guérin et al., 2007). La valeur critique du risque environnemental de perte du P définie à 25% par les Hollandais correspond à 15% en terme de ISP₁ exprimé sur base massique ou 13% si on l'exprime sur base molaire (mmol kg⁻¹ pour P_{M3} et Al_{M3}) et à 11% en terme de ISP₂ dans les sols sableux acides à texture grossière du Québec (Khiari et al., 2000).

Pour les sols de canneberge, Parent et Marchand (2006) ont déterminé une valeur critique d'ISP₂ égale à 12%.

Après avoir établi le lien entre le DSP et l'ISP₁ pour les sols sableux acides du Québec, Khiari et al. (2000) ont établi le lien entre l'ISP₁ et le P_w. La valeur critique environnementale de 15% en terme d'ISP₁ correspond à une valeur critique de 9.7 mg P L⁻¹ en terme de P désorbé à l'eau (Figure 1.6).

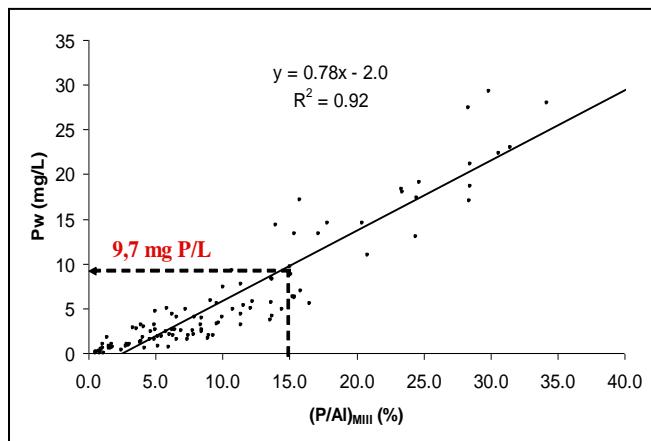


Figure 1.6. Évolution du P_w en fonction de (P/Al)_{M3} (Khiari et al., 2000)

Cette nouvelle valeur critique environnementale a été utilisée par Pellerin et al. (2006a) pour être transposée sur les différentes classes allant des sables jusqu'à l'argile lourde. Les ISP₁ critiques varient de 5.8% à 15.3% pour les sols acides à neutres. À toutes fins pratiques, deux valeurs critiques ont été retenues dans la réglementation québécoise sur les exploitations agricoles, soit 13.1% pour les sols de texture grossière (G2 et G3 dont le pourcentage d'argile<30%), et 7.6% pour les sols de texture fine (G1 dont le pourcentage d'argile>30%) (Figure 1.7).

La relation entre la teneur des sols en P extrait selon l'une ou l'autre des méthodes d'analyse officielles et le DRP a été établie et approuvée suite à plusieurs analyses. En effet, il a été démontré que le DRP dissipé dans les eaux de ruissellement et de drainage est fortement corrélé au P extrait à l'eau (P_w), au P extrait au Mehlich-3 ainsi qu'aux indices de saturation des sols en phosphore (Sharpley, 1995; Pote et al., 1996; Khiari et al., 2000; Maguire et Sims, 2002a; Beauchemin et al., 2003).

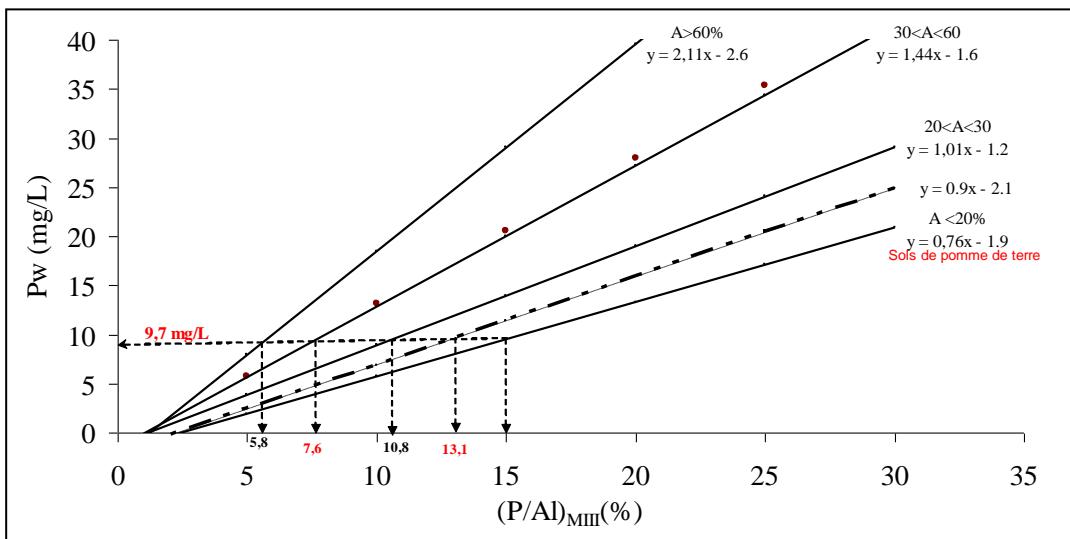


Figure 1.7. Évolution du P désorbé à l'eau (P_w) en fonction du rapport $(P/Al)_{M3}$ (base massique) et de la teneur en argile des sols de Québec (Pellerin et al., 2006b)

Les meilleurs prédicteurs du DRP sont le rapport $[P/(Al+Fe)]_{M3}$ et le P_w (Maguire et Sims, 2002a; Sims et al., 2002). Yli-Halla et al. (1995) et Pote et al. (1996) ont démontré que la concentration du P dissous dans les eaux de ruissellement est hautement corrélée au P extrait à l'eau des sols environnants.

Un point de changement de comportement diffusif du P en profondeur est défini comme étant le point à partir duquel la concentration du DRP augmente significativement avec le contenu en P de la solution du sol (Maguire et Sims, 2002a). Autrement dit, le point de changement correspond à un certain niveau de saturation du sol en P à partir duquel une hausse rapide et considérable de la concentration du P dans les eaux de dissipation est observée. En reliant l'ISP au DRP, Sims et al., (2002) ont déterminé deux points de changement de comportement et de recharge des eaux de dissipation en P en fonction de la saturation des sols en P. Il s'agit de deux valeurs critique de saturation ISP_2 de 21% et de 14% au-delà desquels les eaux de drainage et de ruissellement, respectivement, se chargent considérablement en P (Figure 1.8).

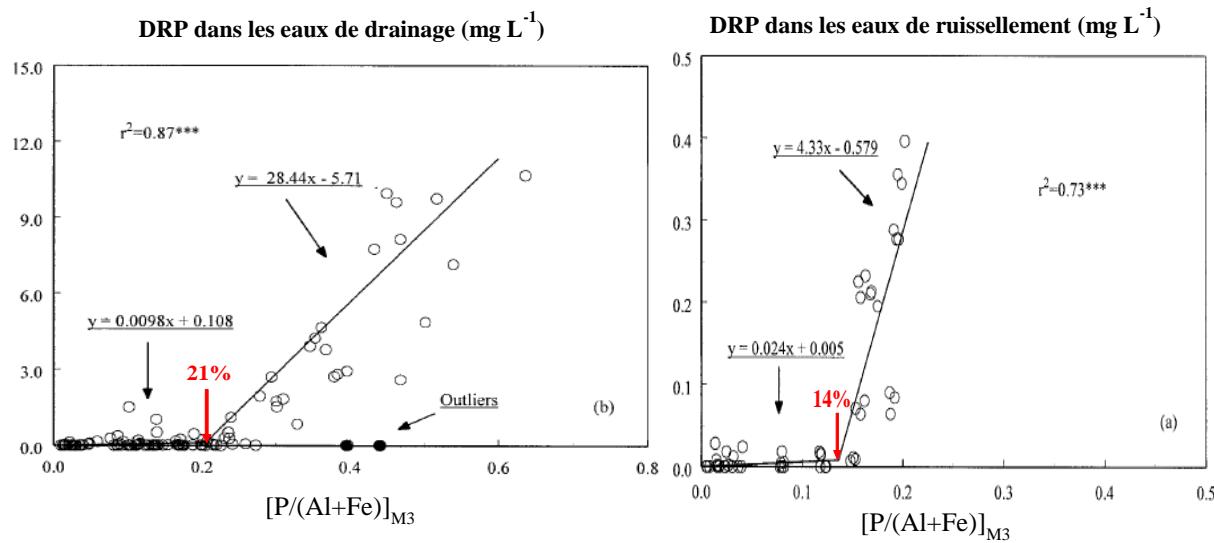


Figure 1.8. Évolution de la teneur en phosphore réactif dissous dans les eaux de drainage et de ruissellement en fonction de l'indice $[P/(Al+Fe)]_{M3}$ (Sims et al., 2002)

1.6 Grilles de recommandations phosphatées pour la pomme de terre dans l'Est du Canada

Les différents indices de fertilité et de diagnostic agro-environnemental présentés dans les sections précédentes sont utilisés pour proposer les grilles de recommandations adéquates en fertilisants phosphatés. Ceci permet d'optimiser les rendements tout en préservant l'environnement.

Dans les provinces de l'Est du Canada, les recommandations en P pour la pomme de terre sont très variables. Au Québec et au Nouveau-Brunswick, les grilles de fertilisation phosphatée ont été actualisées pour prendre compte du rôle de l'Al dans la fixation du P des sols acides de pomme de terre de ces provinces (CRAAQ, 2010; GNB, 2011). Ainsi, pour les sols dont l'indice $(P/Al)_{M3}$ est compris entre 10.1 et 15.0%, les doses recommandées en P sont de 52.4 kg P ha⁻¹ au Québec et comprises entre 43.6 et 65.5 kg P ha⁻¹ au NB. Pour les sols de fertilité en P moyenne ($(P/Al)_{M3} < 10\%$), ces doses varient de 65.5 à 87.3 kg P h⁻¹ et de 65.5 à 91.7 kg P ha⁻¹ au Québec et au NB, respectivement. Au-delà d'un $(P/Al)_{M3}$ de 15%, les doses apportées sont de moins en moins élevées et s'approchent du niveau d'exportation du P par la pomme de terre. À l'Île-du-Prince-Édouard (ÎPE), les doses recommandées en P sont variables en fonction de la variété de pomme de terre. Cependant, quelle que soit la

variété, la dose appliquée ne doit pas dépasser 174.6 kg P ha⁻¹ (PEI Department of Agriculture and Fisheries, 2017). Les doses de P appliquées à l'ÎPE sont plus élevées que celles appliquées au Québec et au NB. Ceci est dû à la grande capacité de fixation du P par les sols podzoliques acides de l'ÎPE réputés pour leur teneur élevée en Fe et en Al (Whiteside, 1965). En Nouvelle-Écosse, aucune grille de recommandations n'a été développée pour la culture de pomme de terre et on suit les recommandations des provinces voisines (Nyiraneza et al., 2017).

1.7 Évaluation spatio-temporelle des sols

Le sol est une ressource naturelle hétérogène dont les processus et les mécanismes sont complexes et difficiles à comprendre (Morellos et al., 2016). La collecte d'informations précises sur les sols aux niveaux régional et national est essentielle, car elle permet une gestion améliorée des sols en fonction de leur potentiel (Odeh et McBratney, 2000). En effet, une meilleure compréhension des propriétés des sols et leur effet sur l'agriculture peut conduire à la mise en œuvre d'une gestion agricole et environnementale durable (Viscarra and Webster, 2012 ; Wang et al., 2009; Yasrebi et al., 2009). Les analyses de laboratoire ont été la clé principale pour comprendre le système de sol et pour évaluer sa qualité et ses fonctions (Viscarra et al., 2006). Cependant, se contenter des méthodes d'analyse traditionnelles, fastidieuses, coûteuses, parfois longues à réaliser et nécessitant un opérateur de laboratoire expert (Morellos et al., 2016 ; Liakos et al., 2018), ne permet pas de répondre aux exigences d'une agriculture de précision qui requiert une résolution d'échantillonnage élevée. Ainsi, pour adapter la gestion des éléments nutritifs aux conditions locales requises par l'agriculture de précision (Inman et al., 2005), des nouvelles technologies ont été développées au cours des dernières décennies afin d'estimer les différentes propriétés des sols et d'étudier leur variabilité spatiale et temporelle. Il s'agit, entre autres, des méthodes d'analyse géostatistiques basées sur des données géoréférencées (krigeage ordinaire, pondération inverse à la distance, etc.) (Guan et al., 2017). Ces techniques d'interpolation fournissent des cartes qui permettent de détecter les zones aux caractéristiques particulières, comme par exemple les zones déficientes en nutriments (Guan et al., 2017). Les chercheurs peuvent ainsi avoir une meilleure compréhension de la dynamique des éléments nutritifs au fil du temps (Hartemink 2006) et détecter les effets directs des pratiques d'utilisation des

terres sur les processus écosystémiques qui ne sont pas apparents dans les études à court terme (Tilman, 1989).

1.8 Hypothèses et objectifs

1.8.1. Hypothèses de recherche

En se basant sur la littérature, les hypothèses de recherche sont les suivantes :

- Il existe une relation entre le degré de saturation des sols en P (DSP) et des indicateurs de saturation en P (ISP) dont la mesure est facile, rapide, moins coûteuse et se prête bien à des analyses de routine.
- Les relations entre les différents indicateurs de sorption, d'adsorption et de désorption du P permettent d'élaborer un modèle environnemental et d'en déduire des valeurs critiques environnementales de saturation des sols en phosphore à l'ÎPE.
- La réponse de la culture de la pomme de terre à la fertilisation phosphatée est fonction des indices de fertilité phosphatée des sols. Cette dernière permet d'établir des modèles de recommandation en P pour les sols des provinces maritimes.
- La substitution partielle de l'engrais P conventionnel par un engrais P issu du recyclage n'entraîne pas la baisse des rendements de la culture de pomme de terre dans les provinces de l'Est du Canada.

1.8.2. Objectifs

1.8.2.1. Objectif général

L'objectif général de cette étude est de développer un système agro-environnemental de diagnostic et de recommandation pour la fertilisation phosphatée de la culture de pomme de terre dans l'Est du Canada.

1.8.2.2. Objectifs spécifiques

En vue d'atteindre cet objectif général, les objectifs spécifiques sont comme suit :

- 1- Développer un modèle environnemental basé sur la détermination des valeurs critiques environnementales de saturation des sols en P prévoyant le risque de pollution diffuse

du P, et déterminer la distribution spatio-temporelle des indices de saturation en P à l'échelle de l'ÎPE.

2- Développer des modèles agronomique et de recommandation en P pour la culture de pomme de terre dans les Maritimes.

3- Étudier l'efficacité d'engrais à base de P recyclé en comparaison aux engrains phosphatés de synthèse sur la culture de pomme de terre.

2. Chapitre 2: Identifying environmental phosphorus risk classes at the scale of Prince Edward Island, Canada

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Abbreviations: α_m , maximal sorption coefficient; DPS, degree of P saturation; M3, Mehlich-3 or (as a subscript) Mehlich-3 extractable; ox (as a subscript), ammonium oxalate-extractable; PEI, Prince Edward Island; pH_{SMP}, pH buffering capacity measured using the Shoemaker–McLean–Pratt method; PSI, P saturation index; P_w, water-extractable P; RF, random forest; RK, regression kriging.

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2.1 Résumé

La perte de phosphore (P) par les terres cultivées pose un risque majeur pour l'environnement. La présente étude avait deux grands objectifs : i) adapter un simple indicateur de la saturation en P reposant sur 141 sols à concentration de P contrastante et s'en servir pour déduire la teneur en P critique pour l'environnement et ii) identifier des classes de risque environnemental, puis en établir la répartition spatiale et temporelle à l'Île-du-Prince-Édouard (Canada). L'indice de saturation en P (ISP) est fortement influencé par l'acidité du sol et les auteurs en ont identifié deux valeurs critiques : celle correspondant à un rapport $(P/Al)_{M3}$ de 19,2% dans les sols très acides ($pH < 5,5$) et celle de 14,2% (l'équivalent de 200 mg de P_{M3} par kg) dans les sols légèrement à moyennement acides ($pH > 5,5$). Au-dessus de ces valeurs, il conviendrait de restreindre les applications de P aux besoins de la culture. Six classes de risque environnemental allant de très faible à extrêmement élevé ont été établies pour le phosphore.

Les auteurs ont vérifié la distribution de ces classes dans l'espace en recourant aux données sur le sol géoréférencées, recueillies entre 2003 et 2015. La classe de risque modéré ($(P/Al)_{M3}$ de 7 à 14% pour les sols au pH supérieur à 5,5) prédomine puisqu'elle couvre environ 70% de la région. Les auteurs ont découvert des points chauds à risque très à extrêmement élevé dans environ 10% de l'île. Il faudrait échafauder des stratégies d'atténuation afin de réduire l'apport de P et de contrôler les risques d'eutrophisation des eaux voisines qui s'y associent.

Mots-clés : échantillonnage du sol avec géoréférencement, krigage de régression, classes de risque environnemental, modèles de désorption/saturation du P du sol, phosphore, indice de saturation du phosphore.

2.2 Abstract

Phosphorus (P) loss from agricultural land poses a major risk to the environment. The main objectives of this study were (i) to adapt a simple P saturation indicator using 141 soils that had contrasting P levels and to deduct critical environmental P values, and (ii) to identify environmental risk classes and their spatial and temporal distribution at the scale of Prince Edward Island, Canada. The P saturation index (PSI) was greatly influenced by the soil acidity, and two critical P saturation indices were identified (i) a PSI (P/Al)_{M3} of 19.2% for very to extremely acidic soils ($pH < 5.5$), and (ii) a PSI of 14.2% (corresponding to 200 mg $P_{M3} \text{ kg}^{-1}$) for slightly to moderately acidic soils ($pH > 5.5$). Above these critical values, P fertilization should be limited to crop requirements. Six environmental P risk classes from very low to extremely high were identified. Spatial distribution of the identified classes was performed using georeferenced soil data collected between 2003 and 2015. The moderate risk class ((P/Al)_{M3} ratio from 7% to 14% for soil pH above 5.5) was the predominant class, covering approximately 70% of the total area. Hot spots in the very high to extremely high range were found in about 10% of the total area, and mitigation strategies are needed to reduce P inputs to control P-related eutrophication risks in surrounding waters.

Key words: georeferenced soil sampling, regression kriging, environmental risk classes, soil phosphorus desorption/saturation models, phosphorus, phosphorus saturation index.

2.3 Introduction

In wet regions, agricultural non-point source pollution is a significant problem from both economic and environmental perspectives. Excessive and repeated inputs of inorganic and organic phosphate fertilizers, which often exceed crop removals, can translate into P loss from agricultural soils and P mobilization and can also transport P to adjacent watercourses through runoff, drainage, or erosion (Sharpley et al. 2013). The result can be surface water eutrophication, particularly in areas where the P retention capacity of the soil is poor (Nair 2014). Regulations governing the management of P fertilization have been based on models that describe the relationship between soil P saturation and the soil's capacity to release this element [Ministère du Développement durable, de l'Environnement et de la Lutte contre les changements climatiques (MDDELCC) 2017].

It was reported that the higher the soil P saturation level, the greater the amount of desorbed P (Lookman et al. 1995). Among the common indicators of P desorption are water-extractable P (P_w) determined by the method of Sissingh (1971) or of Self-Davis et al. (2000) and dissolved reactive P in runoff or drainage water (Breeuwsma and Silva 1992; Sims et al. 2002). Water-extractable P represents a fast and simple method that mimics the P that can be dissolved from soil to runoff water. Two soils may have the same soil test levels of P but have different abilities to release P in runoff, as a result of the soils' different P sorption or P saturation capacities.

Different methods are used to estimate soil P saturation. Traditionally, soil P isotherms were constructed (Nair et al. 1984) to assess the amount of P sorbed to the P in solution at equilibrium. This method was considered time-consuming and not appropriate for routine soil analyses (Sharpley et al. 1994). Alternative soil P saturation indicators include the well-known test for the degree of P saturation (DPS) (van der Zee et al. 1987), which was developed in the Netherlands in acidic soils using ammonium oxalate extractant in the dark. The DPS is calculated as the ratio of oxalate-extractable P to total P sorption capacity derived from oxalate-extractable Al and Fe. However, oxalate extraction is not routinely used for soil analyses in acidic soils of North America, where Mehlich-3 (M3) extraction (Mehlich 1984) has been widely adopted. The critical DPS value of 25% was defined as an international benchmark above which the risk of P loss and water contamination increases. This DPS value

was used to derive P saturation indices (PSI) using M₃ in acidic soils (Sims et al. 1998, 2002; Khiari et al. 2000; Maguire et al. 2001; Nair et al. 2004; Pellerin et al. 2006; Bastounopoulou et al. 2007; Guérin et al. 2007). These indices can be expressed in various ways: PSI₁ (P_{M_3}/Al_{M_3}) for mineral soils (Khiari et al. 2000), PSI₂ [$P_{M_3}/(Al+Fe)_{M_3}$] for soils from the mid-Atlantic region (Sims et al. 2002), and PSI₃ [$P_{M_3}/(Al+5Fe)_{M_3}$] for organic soils (Guérin et al. 2006) [Centre de référence en agriculture et agroalimentaire du Québec (CRAAQ) 2010]. The DPS developed using ammonium oxalate and the PSI developed using M₃ were found to be strongly correlated in acidic to neutral soils (Khiari et al. 2000; Maguire and Sims 2002b; Sims et al. 2002).

Two approaches have been used to describe the relationship between P desorption and saturation indices: one approach is based on the notion of change points in P transport at the surface and in drainage water, and the other is based on the notion of a critical threshold for desorbed P. The first approach was used in Delaware, USA, by Maguire and Sims (2002a) and Sims et al. (2002). Those authors defined two change points in terms of PSI₂ beyond which the dissolved reactive P concentration in surrounding waters increases considerably. The change points were 21% for drainage water and 14% for surface water. Nair et al. (2004) and Wang et al. (2012) used the same approach in order to define P saturation thresholds for soils in Florida, USA, and Ontario, Canada, respectively.

The second approach uses the relationship between P desorption and saturation indices to establish a critical environmental threshold. In Quebec, Canada, the desorption indicator was P_w determined by the method of Sissingh (1971), with a critical P_w of 9.7 mg P L⁻¹ (Khiari et al. 2000; Pellerin et al. 2006). This value corresponded to PSI₁ values of 15, 11, 8, and 6% for coarse-, medium-, fine-, and heavy-textured soils, respectively (Pellerin et al. 2006). For organic soils, the desorption/saturation curve generated a critical value of 5% for PSI₃ (Guérin et al. 2007). All these critical saturation indices were incorporated into the Quebec government's environmental regulations pertaining to agricultural land (MDDELCC 2017). The dynamics of P desorption and saturation in soils can be impacted by certain soil properties.

Besides texture (Jalali and Jalali 2016) and organic matter, pH is a property that has a considerable influence on P desorption/saturation in soils. The more acidic the soil, the

higher the exchangeable Al (Al^{3+}) concentration and the greater the retention of P (Guibaud and Ayele 2000; Iqbal 2012), through precipitation as Al-P (Haseman et al. 1950; Randall and Vose 1963; Hsu 1965; Wright 1989; Daly et al. 2015). Consequently, the amount of P_w decreases as P sorption increases (Pratt 1961; Bowden et al. 1980).

To date, neither a soil P saturation indicator nor a soil P desorption indicator has been developed for the soils of the province of Prince Edward Island (PEI), Canada. Results from a survey conducted across North America by the International Plant Nutrition Institute (2010) revealed that 92% of PEI soils had P levels greater than 50 ppm (Bray P-1 equivalent), in comparison with 64% for New Brunswick, 76% for Newfoundland, 55% for Nova Scotia, 40% for Quebec, 41% for Ontario, 18% for Manitoba, and an average of 26% for North America. The main objectives of the present study were (i) to adapt a simple P saturation indicator and deduct critical environmental P values, and (ii) to identify environmental risk classes and their spatial and temporal distribution in the province of PEI. The findings will help estimate and monitor the environmental risk of P mobilization from farmland in PEI and will identify areas at high risk of P mobilization.

2.4 Materials and methods

2.4.1. Study area

Prince Edward Island is characterized by January and July mean temperatures of -7 and 18.7°C , respectively, and annual mean precipitation of 1100 mm, 25% of which falls as snow (Nyiraneza et al. 2017). The cold temperatures and humidity that characterize the climate of PEI have favoured the development of podzolic soils in the province (Whiteside 1965). Besides the podzolic group, which is predominant, brunisolic, gleysolic, and luvisolic soils are also found but cover smaller amounts of the land area (Nyiraneza et al. 2017). Local differences in soils are attributable to topographic relief and drainage patterns. Podzolic soils are coarse-textured, have low biological activity and fairly high levels of iron and aluminum oxides, and display an acidic reaction (Legros 2007).

2.4.2. Soil selection and analyses

A total of 141 soil samples that had contrasting P levels were selected from the PEI Department of Agriculture soil archive. The majority of the samples were from the Alberry

or Charlottetown soil series (Orthic Humo-Ferric Podzol) (Agriculture and Agri-Food Canada 2013). The remaining soils were from other soil series found in PEI.

Soil samples were taken at a depth of 0 to 17 cm, and five subsamples were mixed to constitute each composite sample. The composite samples were air-dried and passed through a 2-mm sieve. Soil pH was determined on the basis of a soil-to-water solution ratio of 1:1. The soil pH buffering capacity was measured using the Shoemaker–McLean–Pratt method (pH_{SMP}) (Shoemaker et al. 1961). Total organic C was determined by the dry-combustion method (Matejovic 1997). Soil texture was determined by the hydrometer method (Sheldrick and Wang 1993).

P, Al, and Fe were extracted using the M3 method (Mehlich 1984). A 2.5-mL scoop was used to weigh the samples, and extraction was performed in 25 mL of M3 extraction solution. It should be noted that the M3 method is not effective for Fe extraction (Solis and Torrent 1989). To determine P_w, the water extraction procedure of Sissingh (1971) was carried out with a soil-to-solution ratio of 1:60. For extraction with acid ammonium oxalate, a portion of each sample was ground and sieved to pass through a 100-mesh screen. This method can be used to extract the amorphous and weakly crystallized forms of Al and Fe oxides and hydroxides (Ross and Wang 1993) as well as organic complexes of Al and Fe. This extraction was performed using the method of McKeague and Day (1966). Exchangeable Al was extracted using a solution of ammonium chloride (NH₄Cl) (Bertsch and Bloom 1996) and determined by atomic absorption spectrophotometry (PerkinElmer AAnalyst 200; PerkinElmer Inc., Waltham, MA). Quantification of P_w and M3-extractable or oxalate-extractable P, Al, and Fe was carried out by inductively coupled plasma emission spectroscopy (Varian 820-MS spectrometer; Varian Scientific Instruments Inc., Mulgrave, Victoria, Australia).

The DPS was determined as follows:

$$DPS = \frac{P_{ox}(\text{mmol kg}^{-1})}{\alpha_m[\text{Al}(\text{mmol kg}^{-1}) + \text{Fe}(\text{mmol kg}^{-1})]_{ox}} \quad (1)$$

where the subscript *ox* is ammonium-oxalate-extractable, and α_m is the maximal sorption coefficient (van der Zee et al. 1987; Breeuwsma and Silva 1992). In this study, the

DPS was calculated using an α_m value of 0.6, as first proposed by van der Zee et al. (1987) for acidic sandy soils in the Netherlands.

Soil P saturation indices were calculated on a mass basis (mg kg^{-1}) or on a molar basis (mmol kg^{-1}) using the following equations (CRAAQ 2010):

$$\text{PSI}_1 \left(\text{mg kg}^{-1} \right) = \frac{\text{P}_{\text{M3}} \left(\text{mg kg}^{-1} \right)}{\text{Al}_{\text{M3}} \left(\text{mg kg}^{-1} \right)} \quad (2)$$

$$\text{PSI}_2 \left(\text{mmol kg}^{-1} \right) = \frac{\text{P}_{\text{M3}} \left(\text{mmol kg}^{-1} \right)}{\left[\text{Al} \left(\text{mmol kg}^{-1} \right) + \text{Fe} \left(\text{mmol kg}^{-1} \right) \right]_{\text{M3}}} \quad (3)$$

2.4.3. Statistical analyses

The basic descriptive statistics (range, standard deviation, mean, and coefficient of variation) of the measured parameters as well as the relationships between the different P saturation and desorption indicators were determined by simple regression in Excel (Microsoft 2010).

2.4.4. Determination of environmental P risk classes

Environmental P risk classes were established on the basis of two concepts: the desorption criterion (P_w) and the PSI_1 . The identified critical P_w was used to separate the mean P mobility risk class from the highest risk class. Below this critical value, three risk classes were established using the procedure developed by Cope and Rouse (1983): moderate, low, and very low, corresponding to 100, 50, and 25% of the critical P_w threshold. For desorption levels higher than the critical P_w , three classes with an elevated environmental risk were derived to delineate the high, very high, and extremely high environmental risk classes based on the studies by Sims et al. (2002), who identified two critical soil P saturation values of 14 and 21% as PSI_2 , beyond which the risk of water contamination from runoff and drainage, respectively, reaches a high level.

2.4.5. Collection of spatially distributed training and testing data

Georeferenced soil samples have been collected since 1998 by the PEI Department of Agriculture and Fisheries as part of the PEI Soil Quality Monitoring Project. However, Al has only been included in the standard soil analysis package since 2003. The sampling

procedure for that project was described in detail by Douglas et al. (2000). Briefly, sampling points on agricultural lands were located on intersecting points (i.e., nodes) of a 4×4 -km National Forest Inventory grid system (Canadian Forest Inventory Committee 1998), and additional soil samples were taken at 100 m from the central point in each cardinal direction, for a possible total of five sampling points per grid intersection (central point and points north, south, east, and west from the central point). Soil sampling was carried out, by means of an Edelman soil auger, in the spring before tillage or fertilization, with an approximately 1- to 3-kg sample taken from each sampling point at a depth of 0 to 17 cm. The total number of sampled sites varied from year to year ($n = 592$ in 2003–2006; $n = 538$ in 2007–2009; $n = 496$ in 2010–2012; and $n = 490$ in 2013–2015), and some sites were lost because of changes in ownership and in land-use (i.e., conversion from agricultural land to a cemetery, residential area, commercial area, or another use). Because of the island's area, which is 5665 km^2 , the georeferenced samples were taken at each site once every 3 yr for the whole study period.

In the prediction process, the 4×4 intersecting points were used as training data. The non-intersecting points were used to calculate inference residuals and to evaluate P and Al inference accuracy. The basic statistics of the training and testing data used in each cycle are listed in Table 2.1.

2.4.6. Environmental covariates

Environmental covariates used as predictors for this study included various topographic indices and surficial geology information. The topographic indices were calculated in SAGA GIS software (Conrad et al. 2015) based on a DEM (digital elevation model) data set downloaded from Natural Resources Canada. The DEM data were first resampled to 30 m using the bilinear interpolation method with projection of WGS84 UTM-20N (Universal Transverse Mercator coordinate system, zone 20N) before the calculation of topographic indices. Random forest (RF) was applied to identify important covariates.

The covariates used in the inference process for P were valley depth (Rabassa 2017), flow accumulation (Goodchild et al. 1996), topographic wetness index (Sørensen et al. 2006), slope length factor (Desmet and Govers 1996), convergence index (Köthe and Lehmeier 1996), relative slope position (Weiss 2001), slope in gradients, and surficial deposit layer.

The covariates used in the inference process for Al were plan curvature (de Smith et al. 2015), multiresolution index of the ridge top flatness index (Gallant and Dowling 2003), slope length factor, valley depth, slope in gradients, mass balance index (Möller et al. 2012), topographic wetness index, and surficial deposit layer.

2.4.7. Regression kriging

In this study, the P saturation index was calculated in two steps: P and Al values were inferred separately, and then PSI_1 was calculated. For each cycle, the following sequence was applied: first, RF was applied to estimate soil P and Al attributes (i.e., regression) based on the training data (i.e., intersecting points); next, ordinary kriging was used to interpolate RF regression residuals calculated at the non-intersecting locations; and lastly, the interpolated residuals were added to the RF regression result. This inference approach is called regression kriging (RK) (Odeh et al. 1994). Before soil property inference, the normality of training and testing data was examined using the Shapiro–Wilk test (Shapiro and Wilk 1965) in R software (R Core Team 2013). A square-root transformation was applied to soil P attributes, and a square transformation was applied to Al attributes, in order to meet the normality requirement of ordinary kriging on data values (Hengl 2009).

The performance of RK was evaluated by comparing the RK-derived P and Al values to the sampled values at the testing point (i.e., non-intersecting) locations. The accuracy indicators included coefficient of determination (R^2), coefficient of concordance correlation (ρ), root mean square error (RMSE), and bias (mean error of prediction).

2.5 Results and discussion

2.5.1. Soil characteristics

The pH levels varied between 4.5 and 7.1. Among the 141 soils studied, 38% were very acidic to extremely acidic ($\text{pH} < 5.5$). The rest of the soils were slightly to moderately acidic, on the basis of the classification of PEI soils by Whiteside (1965) (Table 2.2). More than half of the soils (56%) had pHSMP below 6.7 and were considered buffered against alkalinity on the basis of the assessment of Shoemaker et al. (1961). The organic matter contents were low, with a maximum of 48 g kg^{-1} , and 65% of the soils had an organic matter

content below 30 g kg^{-1} (Table 2.2). Phosphorus extracted with M3 extractant ranged from 38 to 455 mg kg^{-1} with a coefficient of variation of 58%, whereas PSI_1 and PSI_2 ranged from 2.3 to 32% and from 1.9 to 25% with coefficients of variation of 56 and 55%, respectively (Table 2.2).

The majority of the soils ($n = 124$) were sandy loam. All the soils had low clay content not exceeding 150 g kg^{-1} . The PSI_1 and P_w had coefficients of variation higher than 55%. The greatest variation was observed for the exchangeable Al content of soils, which ranged from 0.6 to 68.3 mg kg^{-1} , with a coefficient of variation higher than 100% (Table 2.2).

Aluminum extracted with M3 ranged from 561 to 2102 mg kg^{-1} (Table 2.2). For acidic soils in Quebec, the P fixation capacity of the soil is low at an Al_{M3} content below 1100 mg kg^{-1} , moderate at a content between 1100 and 1600 mg kg^{-1} , and high at levels above 1600 mg kg^{-1} (Tran et al. 1990). Alternatively, the P retention capacity of soils can be estimated from the sum of $(\text{Al}+\text{Fe})_{\text{ox}}$. In acidic soils of Quebec, Déziri (2009) established six classes of soil P fixing capacity (very low, $<70 \text{ mmol kg}^{-1}$; low, $70\text{--}116 \text{ mg kg}^{-1}$; medium, $116\text{--}148 \text{ mmol kg}^{-1}$; high, $148\text{--}191 \text{ mmol kg}^{-1}$; very high, $191\text{--}244 \text{ mmol kg}^{-1}$; and excessive, $>244 \text{ mmol kg}^{-1}$) as a function of soil $(\text{Al}+\text{Fe})_{\text{ox}}$ contents. According to this classification, 3% of the soils analyzed in this study had low P-fixing capacity, 8% had moderate P-fixing capacity, and 89% had high, very high, and extremely high P-fixing capacity. In cases where M3 extractant is used for routine soil analyses, P retention capacity can be assessed by simple determination of Al_{M3} . Indeed, the greater the Al_{M3} content, the greater the P retention capacity and the lower the risk of soil P saturation (Daly et al. 2015). The values of Al_{M3} ranging from 561 to 2102 mg kg^{-1} are comparable to those found in Quebec ($650\text{--}2382 \text{ mg kg}^{-1}$) (Khiari et al. 2000).

2.5.2. Desorption/saturation model: Relationship between DPS and P_w

The desorption/saturation model expressed in P_w/DPS showed an ascending straight line where P_w was strongly correlated with DPS ($R^2 = 0.70$) (Fig. 2.1). A significant positive relationship between P solubility in the soil and the P saturation indicator was reported in previous studies (Kuo et al. 1988; Lookman et al. 1996; Sharpley 1996; Khiari et al. 2000; Pautler and Sims 2000; Leclerc et al. 2001; Sims et al. 2002; Nair et al. 2004; Pellerin et al. 2006; Bortolon et al. 2016). The higher the P saturation level, the greater the tendency for

soil to desorb the excess P (Lookman et al. 1995). The critical P_w corresponding to the critical DPS value of 25% obtained in acidic soils of the Netherlands was $9.7 \text{ mg } P_w \text{ L}^{-1}$, and the same value was obtained by Khiari et al. (2000) for acidic sandy soils in Quebec. This critical value of $9.7 \text{ mg } P_w \text{ L}^{-1}$ distinguishes effectively between soils that pose a high environmental risk and those that pose a lower risk. Approximately one third of the soils under study presented a high environmental risk, with a P_w concentration higher than the critical level of $9.7 \text{ mg } P_w \text{ L}^{-1}$. However, when this critical value was applied across the confidence interval, the corresponding DPS diverged considerably from the critical DPS value of 25%, ranging from 17 to 33% (Fig. 2.1). This wide range does not provide the required precision for decision making related to the critical environmental threshold. In addition, determining the DPS is a labour-intensive procedure, because oxalate extraction requires specific experimental conditions in terms of darkness and very tight time constraints. Therefore, oxalate extraction was replaced with the M3 method (Sharpley 1995; Beauchemin and Simard 1999; Khiari et al. 2000; Sims et al. 2002), which is faster, easier, and more suitable for routine analyses. To verify the correlation between soil saturation values as determined by these two methods, we studied the relationship between DPS and the PSI_1 and PSI_2 using the 141 soils used in this study (eqs. 4, 5).

$$\text{DPS (\%)} = 0.79 \text{ PSI}_1 (\text{mg kg}^{-1}) + 12.05 \quad (n = 141; R^2 = 0.90) \quad (4)$$

$$\text{DPS (\%)} = \text{PSI}_2 (\text{mmol kg}^{-1}) + 11.99 \quad (n = 141; R^2 = 0.88) \quad (5)$$

The PSI_1 and PSI_2 indices were strongly correlated (eq. 6).

$$\text{PSI}_1 (\text{mg kg}^{-1}) = 1.26 \times \text{PSI}_2 (\text{mmol kg}^{-1}) \quad (n = 141; R^2 = 1.00) \quad (6)$$

The PSI_1 value ranged from 2.3% (soils with a very low P saturation level) to 32.4% (soils with an extremely high P saturation level). For acidic mineral soils in Quebec, Khiari et al. (2000) obtained PSI_1 values varying between 0.5 and 55.1% and selected a PSI_1 value of 15% as a critical environmental threshold. On the basis of the classification developed by these authors, the environmental risk for our soils ranged from low to high. The regression equation ($\text{PSI}_1 = 1.26 \times \text{PSI}_2$) is very close to the equation of Khiari et al. (2000) ($\text{PSI}_1 = 1.34 \times \text{PSI}_2$) and to that of Pellerin et al. (2006) ($\text{PSI}_1 = 1.39 \times \text{PSI}_2$) for acidic soils in Quebec. On a molar basis, Sims et al. (2002) and Maguire and Sims (2002b) found the

regression equations $\text{PSI}_1 = 1.13 \times \text{PSI}_2 - 0.003$ and $\text{PSI}_1 = 1.21 \times \text{PSI}_2 - 0.015$, respectively. Even though PEI soils are known to be rich in Fe (Whiteside 1965), the limited effectiveness of the M3 solution for extracting this element meant that Fe played a negligible role in the calculation of the P saturation index. Mehlich-3-extractable Fe represented only 8% ($\pm 3.52\%$) of the sum of $(\text{Al}+\text{Fe})_{\text{M3}}$ (mmol kg^{-1}). Thus, including Fe_{M3} in the calculation of the P saturation index did not improve the environmental desorption/saturation model. Consequently, the model in which P_w is a function of PSI_1 seems to be the most suitable for establishing environmental limits for PEI soils.

2.5.3. Desorption/saturation model: Relationship between PSI_1 and P_w

A positive linear relationship between P_w and PSI_1 was found ($R^2 = 0.79$; Fig. 2.2a). The critical P_w of 9.7 mg P L^{-1} (Fig. 2.2a) corresponded to a PSI_1 of 16%, which is close to the PSI_1 of 15% previously selected for coarse-textured acidic soils in Quebec (Khiari et al. 2000). However, it was observed that the higher the P saturation index, the greater the variation of data points from the linear model (Fig. 2.2a). Beauchemin and Simard (1999) recommended forming homogeneous groups in order to reduce the variation, and Jalali and Jalali (2016) identified soil texture as an important factor associated with P sorption. The soils were grouped according to several criteria (pH, texture, and organic matter), and the correlation between P_w and PSI_1 showed a significant increase only when the soils were grouped according to their pH_{water} . Two groups were created: highly to extremely acidic soils with pH_{water} below 5.5, and slightly to moderately acidic soils with pH_{water} above 5.5. The pH_{water} value appears to have a considerable influence on the desorption/saturation model, with the slope increasing from 0.60 for highly to extremely acidic soils to 0.73 for moderately acidic soils (Fig. 2.2b).

The PSI_1 corresponding to a critical environmental threshold of $9.7 \text{ mg P}_w \text{ L}^{-1}$ was 14.2% for soils with pH above 5.5 and 19.2% for soils with pH below 5.5 (Fig. 2.2b). Beyond these two critical PSI_1 values, the risk of P loss is high, and inputs of P fertilizer should be limited to the plants' requirements (Khiari et al. 2000).

The content of active or mobile forms of Al, known as exchangeable aluminum (Al^{3+}), increased as the soil pH declined (Fig. 2.3), as was previously reported (Skwira et al. 2012; Daly et al. 2015). The presence of exchangeable Al is one of the main chemical

characteristics of strongly acidic soils (Ségalen 1973; Bache 1985). The pH values at which the amount of exchangeable Al increases exponentially and becomes measurable depend on the soil properties. This point generally occurs at a pH level between 5 and 6, typically at pH 5.5 (Boyer 1976). One of the most visible signs of the presence of exchangeable Al in soil is a P deficiency. Exchangeable Al binds to P within the roots, slowing P availability to the plant (Boyer 1976).

We observed that, at the same saturation level of 16%, soils with pH above 5.5 desorb more P ($P_w = 11 \text{ mg P L}^{-1}$) than soils with pH below 5.5 do ($P_w = 7.8 \text{ mg P L}^{-1}$) (Fig. 2.2b), and thus the risk of P loss is higher. Therefore, soil acidity was identified as the main criterion influencing the dynamics of desorption/saturation in PEI soils. According to Bache (1985) and Syers et al. (2008), P retention capacity is greater at low pH levels than at higher pH levels. According to Deb and Datta (1967) and Bolan et al. (1986), this retention capacity is explained by an increase in the positive charges of colloids and an increase in Al activity (Daly et al. 2015). At a neutral pH, surface charge is low, so phosphate ions are adsorbed with difficulty (Razaq 1989).

2.5.4. Environmental P risk classes

The critical P_w of 9.7 mg P L^{-1} separated the mean P mobility risk class from the highest risk class. Below this critical value, three classes corresponding to 100, 50, and 25% of critical P_w (Cope and Rouse 1983) were identified: moderate ($9.7 \text{ mg P}_w \text{ L}^{-1}$), low ($4.8 \text{ mg P}_w \text{ L}^{-1}$), and very low ($2.4 \text{ mg P}_w \text{ L}^{-1}$), respectively. The threshold level of $9.7 \text{ mg P}_w \text{ L}^{-1}$ was very close to the values of 10.0 and $10.9 \text{ mg P}_w \text{ L}^{-1}$ corresponding to the high fertility level of German soils (Landwirtschaftskammer Hannover 1996) and Dutch soils (Breeuwsma and Silva 1992), respectively. This method of establishing classes below the environmental limit is based on an interpretation that is both agronomic and environmental. Since plant response models are non-linear, it makes more sense to use the partitioning approach of Cope and Rouse (1983). However, for desorption levels higher than $9.7 \text{ mg P}_w \text{ L}^{-1}$, findings by Sims et al. (2002) were used to derive three classes with an elevated environmental risk. Sims et al. (2002) identified two critical soil P saturation values of 14 and 21% as PSI_2 , beyond which the risk of water contamination from runoff and drainage, respectively, reaches a high level. By using a double conversion of PSI_2 to PSI_1 values (eq. 6; $\text{PSI}_1 = 1.26 \times \text{PSI}_2$) and

then PSI_1 values to P_w values (equation in Fig. 2.2a; $y = 0.67x - 1.01$), we obtained critical P_w values of 10.8 mg P L^{-1} for runoff and 16.7 mg P L^{-1} for drainage water, corresponding to 14 and 16% PSI_2 , respectively. We used these two thresholds to delineate the high, very high, and extremely high environmental risk classes (Table 2.3).

The desorption/saturation model approach (Fig. 2.2b) was used to assess environmental risk classes in two groups created on the basis of soil acidity (Fig. 2.2b; Table 2.3). For more acidic soils, the proposed critical environmental values were less severe than those for less acidic soils were. For soils with pH_{water} below 5.5, the critical limits of the intervals were three or four percentage points of PSI_1 higher in the very low and low risk classes, five points higher in the moderate and high risk classes, and seven points higher in the very high risk class (Table 2.3). When the pH level is very low, the various fixing agents that are present favour greater P retention in soils than in less acidic soils, and the environmental risk level is reached slowly. The moderate risk class is of particular interest, since it corresponds to soils whose P level is sufficient for crops and does not pose a risk to the environment. The moderate risk class (7–14%) for soils at pH_{water} above 5.5 was close to the class established for coarse-textured acidic soils in Quebec (8–15%) (Khiari et al. 2000) and comparable to the moderate fertility class for German soils (8.9–15.3%) (Landwirtschaftskammer Hannover 1996). The upper limit of the moderate risk class was close to the PSI_1 of 16% obtained for all soils in PEI (Fig. 2.2a). The PSI_1 value of 14% also corresponded to a P_{M3} content of 200 mg kg^{-1} (eq. 7), and the same value was reported by Sharpley et al. (2002) in several U.S. states (Kansas, Oklahoma, and Pennsylvania).

$$\text{PSI}_1 (\text{mg kg}^{-1}) = 0.07 P_{M3} (\text{mg kg}^{-1}) \quad (n = 141; R^2 = 0.88) \quad (7)$$

2.5.5. Statistics associated with P and Al interpolation

As discussed previously, P and Al were inferred first, and then their ratio was calculated. Instead of discussing spatiotemporal trends of P and Al, we will focus only on calculated PSI_1 , which is linked to the objective of the present study. Averaged across testing and training data and across cycles, P values were 372 and 1397 mg kg^{-1} for P and Al respectively (Table 2.1). As measures of dispersion, the coefficients of variation were 20% for Al and 60% for P (Table 2.1). Coefficients of variation ranging from 16 to 35% were

previous deemed to be moderate (Wilding and Drees 1983; Nielsen and Bouma 1985); thus, Al showed low variation, whereas P showed high variation, probably as a result of annual P fertilizer applications to meet P crop requirements.

Semivariogram parameters are presented in Table 2.4. As measures of spatial structural variation, nugget ratio (nugget/sill) values were below 50%, which again indicates moderate spatial dependence (Cambardella et al. 1994; Cambardella and Karlen 1999) throughout the study period for both Al and P. The range reflects the distance where variable are spatially autocorrelated and was 7557 to 8457 m for P and 2660 to 7100 m for Al (Table 2.4), mirroring the wider sampling grid dimension. The model accuracy evaluation indicators are reported in Table 2.5. Values for the coefficient of variation, which measures the strength of the relationship between observed and predicted soil properties, ranged from 64 to 78% for P and from 70 to 79% for Al. On the basis of concordance as a measure of goodness of fit, values above 70 and 80% indicate a good model and a strong model, respectively (Altman 1991). Values of 100% indicate that predicted and observed data match. Bias evaluates the mean difference between the predictions and observations. Overall, overestimation bias was observed for P, and underestimation bias was observed for Al. In sum, on the basis of the concordance values (mostly >80%), the model for interpolating P and Al was deemed acceptable (Altman 1991), and we could therefore derive the P/Al ratio. The RMSE reflects the deviation of the predicted and observed values, and the smaller the RMSE, the closer both values are. These values were in the same range for P and Al for all cycles (Table 2.5), with trends toward higher values in cycle 5 for P and in cycles 5 and 6 for Al.

2.5.6. Spatiotemporal distribution of environmental P risk classes

Given the environmental P risk classes identified in Table 2.3, it is very important to identify areas of high risk in order to recommend mitigation strategies. Maps of identified classes were developed using georeferenced soil samples collected between 2003 and 2015. Risk classes for soil with pH above 5.5 were used, as this pH range is more likely to be encountered in most agricultural areas. Overall, we observed that the moderate risk class was the most predominant (approximately 70% of the total area), followed by the low risk class (12–14% of the total area) (Table 2.6; Fig. 2.4). The area within the risk classes identified as

high or extremely high was below 10% of the total area (Table 2.6; Fig. 2.4). No significant changes were observed from year to year, which implies that, although we observed slight increases in Al over time (data not reported), which would enhance P sorption, regular P inputs have been supplied to different crops at the same time, which explains the absence of any trend across years. We observed some, although not many, hot spots within the risk class categorized as very high or extremely high, which were concentrated mainly in the western part of the island, with a few patches in the rest of the province. The risk of P mobilization and transport to adjacent watercourses is expected to be high around these areas, and P inputs should be limited.

2.6 Conclusion

By combining P adsorption and desorption models, we obtained two critical P saturation index (PSI_1) values of 19.2 and 14.2% for soils with pH below 5.5 and above 5.5, respectively, from which environmental risk classes within each soil group were calculated. For soils with pH above 5.5, six risk classes were determined on the basis of PSI_1 : very low risk (0–4%), low risk (4–7%), moderate risk (7–14%), high risk (14–16%), very high risk (16–23%), and extremely high risk (>23%). Using a georeferenced long-term data set, we performed a spatial and temporal distribution of identified environmental P risk classes across PEI, and the results showed that the moderate risk class was the predominant class across the island and corresponds to soils whose P level is sufficient to meet crop P requirements and does not pose a risk to the environment. Hot spots within the risk class categorized as very high or extremely high were concentrated mainly in the western part of PEI, with a few patches in the rest of the province. The results of this study provide much needed information for PEI to support environmental assessment at the provincial scale for identifying risks for P build-up and release in high-P fields.

2.7 Acknowledgments

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Table 2.1. The basic statistics (count, minimum [min], maximum [max], mean, and coefficient of variation [CV]) of the training and testing data used in each cycle of P and Al interpolation.

| Soil property | Cycle | Training data | | | | | Testing data | | | | |
|-------------------------------------|---------------|---------------|-----|------|------|-----|--------------|-----|------|------|-----|
| | | Count | Min | Max | Mean | CV | Count | Min | Max | Mean | CV |
| P_{M3} (mg kg ⁻¹) | 3 (2004-2006) | 120 | 65 | 934 | 368 | 0.6 | 474 | 43 | 1115 | 355 | 0.6 |
| | 4 (2007-2009) | 105 | 47 | 1098 | 379 | 0.6 | 436 | 28 | 1106 | 389 | 0.6 |
| | 5 (2010-2012) | 99 | 21 | 1053 | 350 | 0.6 | 399 | 27 | 1643 | 377 | 0.6 |
| | 6 (2013-2015) | 100 | 35 | 1256 | 381 | 0.6 | 392 | 16 | 1248 | 375 | 0.6 |
| Al_{M3} (mg kg ⁻¹) | 3 (2004-2006) | 120 | 565 | 1866 | 1335 | 0.2 | 474 | 368 | 1925 | 1355 | 0.2 |
| | 4 (2007-2009) | 105 | 705 | 1877 | 1406 | 0.2 | 435 | 424 | 1912 | 1421 | 0.2 |
| | 5 (2010-2012) | 99 | 318 | 1833 | 1406 | 0.2 | 399 | 93 | 1936 | 1412 | 0.2 |
| | 6 (2013-2015) | 100 | 599 | 1960 | 1412 | 0.2 | 392 | 469 | 2075 | 1428 | 0.2 |

P_{M3} , Mehlich-3-extractable P; Al_{M3} , Mehlich-3-extractable Al.

Table 2.2. Selected properties of the 141 Prince Edward Island soil samples used for the environmental model.

| Soil properties | Range | Standard deviation | Mean | Coefficient of variation (%) |
|--|--------------|--------------------|-------|------------------------------|
| pH | 4.5–7.1 | 0.6 | 5.7 | 10.3 |
| pH _{SMP} | 6.1–7.3 | 0.2 | 6.6 | 3.5 |
| Organic matter (g kg ⁻¹) | 16–48 | 6.4 | 28 | 22.8 |
| Sand (g kg ⁻¹) | 335.8–763.5 | 62.3 | 595.8 | 10.5 |
| Clay (g kg ⁻¹) | 25–150 | 22 | 71.3 | 30.9 |
| Silt (g kg ⁻¹) | 186.5–539.3 | 54 | 332.9 | 16.2 |
| Al ³⁺ (mg kg ⁻¹) | 0.6–68 | 16 | 16 | 101 |
| P _{M3} (mg kg ⁻¹) | 38–455 | 105 | 181 | 58 |
| Al _{M3} (mg kg ⁻¹) | 561–2 102 | 265 | 1 394 | 19 |
| Fe _{M3} (mg kg ⁻¹) | 98–517 | 91 | 240 | 38 |
| P _w (mg kg ⁻¹) | 0–22 | 4.7 | 6.7 | 69.7 |
| PSI ₁ (%) | 2.3–32.4 | 7.3 | 13.0 | 55.6 |
| PSI ₂ (%) | 1.9–25.4 | 5.7 | 10.4 | 54.6 |
| P _{ox} (mg kg ⁻¹) | 396–1 669 | 274 | 916 | 30 |
| Al _{ox} (mg kg ⁻¹) | 1 045–7 374 | 1 198 | 3 444 | 35 |
| Fe _{ox} (mg kg ⁻¹) | 3 029–12 715 | 1 176 | 5 499 | 21 |
| (Al+Fe) _{ox} (mmol kg ⁻¹) | 101–500 | 59 | 226 | 26 |
| DPS ($\alpha_m = 0.6$) (%) | 11–36 | 6.0 | 22.4 | 27 |

pH_{SMP}, pH buffering capacity measured using the Shoemaker–McLean–Pratt method; Al³⁺, exchangeable aluminum extracted with ammonium chloride; M3 (as a subscript), Mehlich-3-extractable; P_w, water-extractable P; PSI₁, P saturation index (P/Al)_{M3}; PSI₂, P saturation index [(P/Al+Fe)]_{M3}; ox (as a subscript), ammonium-oxalate-extractable; DPS, degree of P saturation; α_m , maximal sorption coefficient.

Table 2.3. Environmental risk classes for Prince Edward Island soils.

| Environmental risk classes | P_w (mg P L ⁻¹) | PSI ₁ (%) (pH > 5.5) | PSI ₁ (%) (pH < 5.5) |
|----------------------------|----------------------------------|------------------------------------|------------------------------------|
| Very low | 0–2.4 | 0–4 | 0–7 |
| Low | 2.4–4.8 | 4–7 | 7–11 |
| Moderate | 4.8–9.7 | 7–14 | 11–19 |
| High | 9.7–11 | 14–16 | 19–21 |
| Very high | 11–16 | 16–23 | 21–30 |
| Extremely high | >16 | >23 | >30 |

P_w , water-extractable P; PSI₁, P saturation index (P/Al)_{M3}; M3 (as a subscript), Mehlich-3-extractable.

Table 2.4. The fitted semivariogram parameters used in ordinary kriging for P and Al extracted with Mehlich-3 (M3).

| | Cycle | Sill | Nugget | Range (m) |
|-----------|---------------|-----------------|-----------------|-----------|
| P_{M3} | 3 (2004-2006) | 24 | 10 | 8319 |
| | 4 (2007-2009) | 31 | 14 | 8547 |
| | 5 (2010-2012) | 32 | 16 | 7615 |
| | 6 (2013-2015) | 33 | 13 | 7557 |
| Al_{M3} | 3 (2006-2009) | 339 132 173 558 | 136 524 903 506 | 7100 |
| | 4 (2007-2009) | 373 437 095 543 | 162 495 725 674 | 7021 |
| | 5 (2010-2012) | 370 922 947 266 | 181 718 884 826 | 2660 |
| | 6 (2013-2015) | 484 764 628 394 | 218 992 281 036 | 2843 |

Table 2.5. Model accuracy evaluation indicators.

| | Cycle | R^2 | Concordance | RMSE | Bias |
|-----------|---------------|-------|-------------|---------|---------|
| P_{M3} | 3 (2004-2006) | 0.750 | 0.835 | 100.909 | -11.958 |
| | 4 (2007-2009) | 0.728 | 0.814 | 124.223 | -16.286 |
| | 5 (2010-2012) | 0.644 | 0.735 | 149.795 | -19.35 |
| | 6 (2013-2015) | 0.788 | 0.859 | 111.452 | -15.508 |
| Al_{M3} | 3 (2006-2009) | 0.792 | 0.860 | 124.972 | 10.277 |
| | 4 (2007-2009) | 0.766 | 0.834 | 128.387 | 10.463 |
| | 5 (2010-2012) | 0.706 | 0.754 | 165.032 | 17.054 |
| | 6 (2013-2015) | 0.790 | 0.836 | 143.057 | 14.568 |

Note: These values are derived by comparing the testing point values to the final regression kriging results. The indicators are calculated at the original scales of the P and Al values. RMSE, root mean square error; P_{M3} , Mehlich-3-extractable P; Al_{M3} , Mehlich-3-extractable Al.

Table 2.6. Areas (ha) and percentages (in parentheses) of environmental P risk classes for soils with pH above 5.5 per cycle.

| Environmental P risk classes | Cycle 3 (2004–2006) | Cycle 4 (2007–2009) | Cycle 5 (2010–2012) | Cycle 6 (2013–2015) |
|---------------------------------|------------------------|------------------------|------------------------|------------------------|
| <4 (very low) | 2 057.04 (0.4) | 6 858.36 (1.3) | 5 605.38 (1.1) | 7 410.33 (1.4) |
| 4–7 (low) | 73 948.68 (14.1) | 66 226.05 (12.6) | 69 106.14 (13.2) | 77 085.09 (14.7) |
| 7–14 (moderate) | 369 725.94 (70.6) | 364 733.64 (69.6) | 376 655.94 (71.9) | 372 308.4 (71.1) |
| 14–16 (high) | 41 060.25 (7.8) | 44 312.13 (8.5) | 38 968.11 (7.4) | 36 470.79 (7) |
| 16–23 (very high) | 35 538.3 (6.8) | 39 378.15 (7.5) | 31 868.1 (6.1) | 28 505.3 (5.4) |
| >23 (extremely high) | 1 632.15 (0.3) | 2 454.57 (0.5) | 1 759.14 (0.3) | 2 182.68 (0.4) |

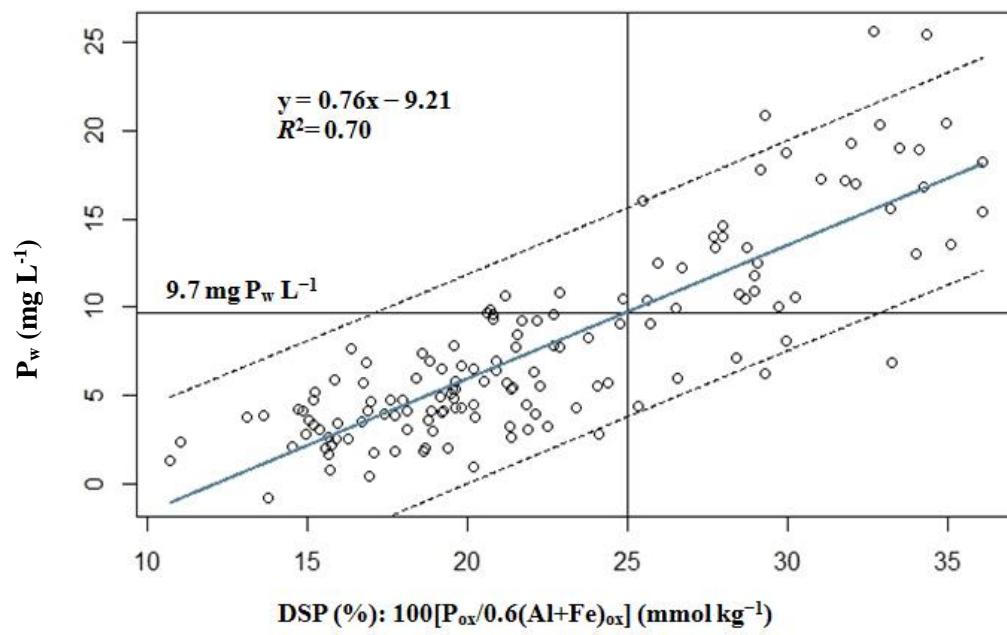


Figure 2.1. Linear correlation between water-extractable P (P_w) and the degree of P saturation (DPS).

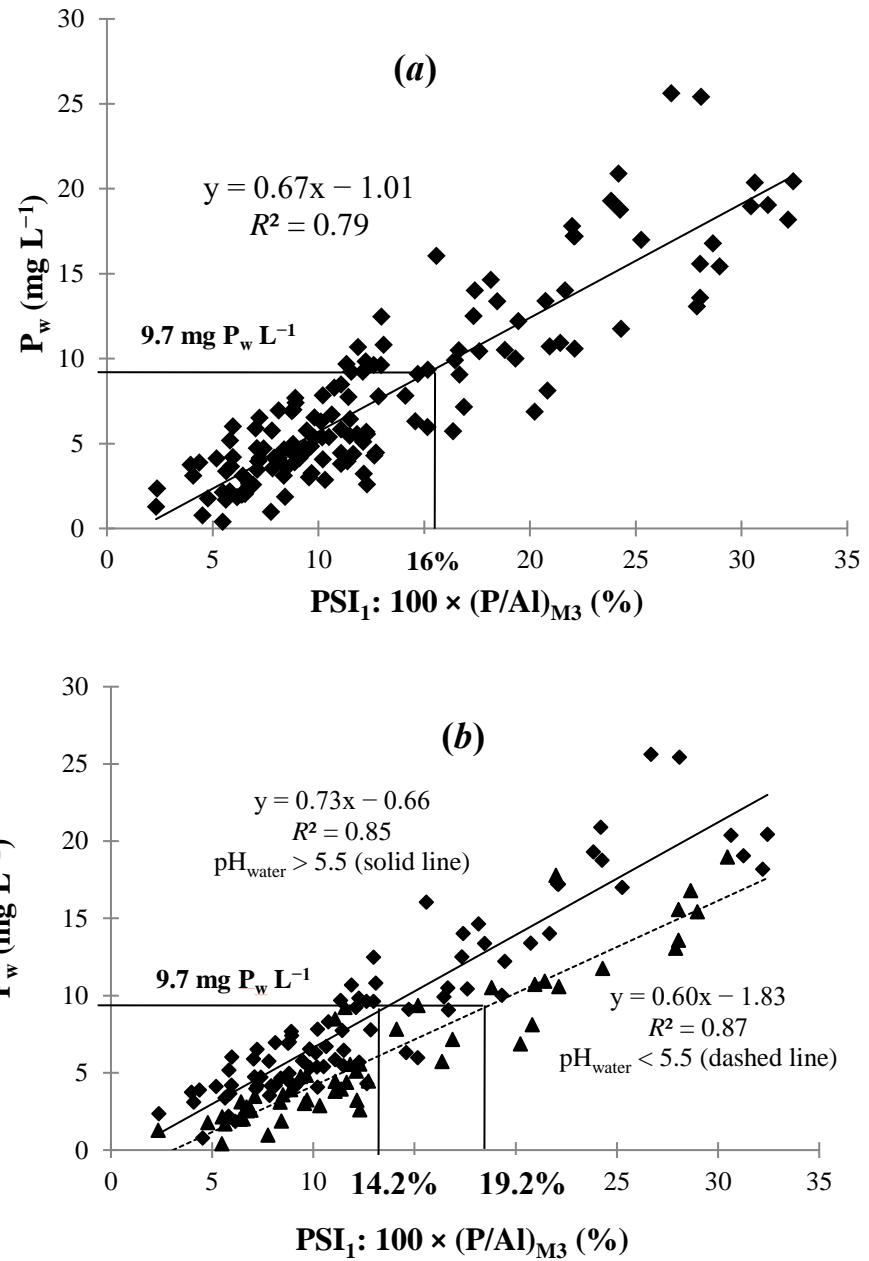


Figure 2.2. Correlation between water-extractable P (P_w) and P saturation index (PSI_1) (a) for the whole data set and (b) when data are grouped by soil pH.

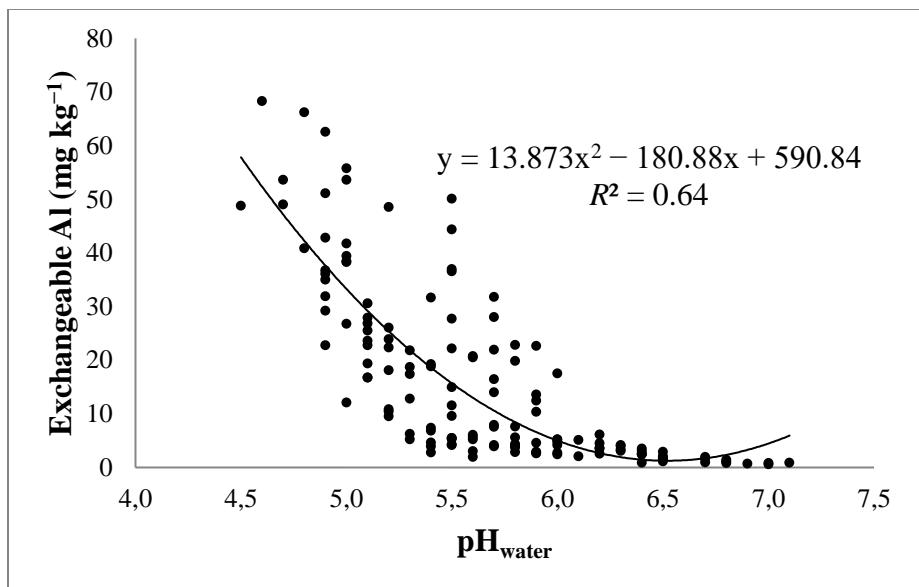


Figure 2.3. Relationship between exchangeable Al and pH_{water} in the studied soils.

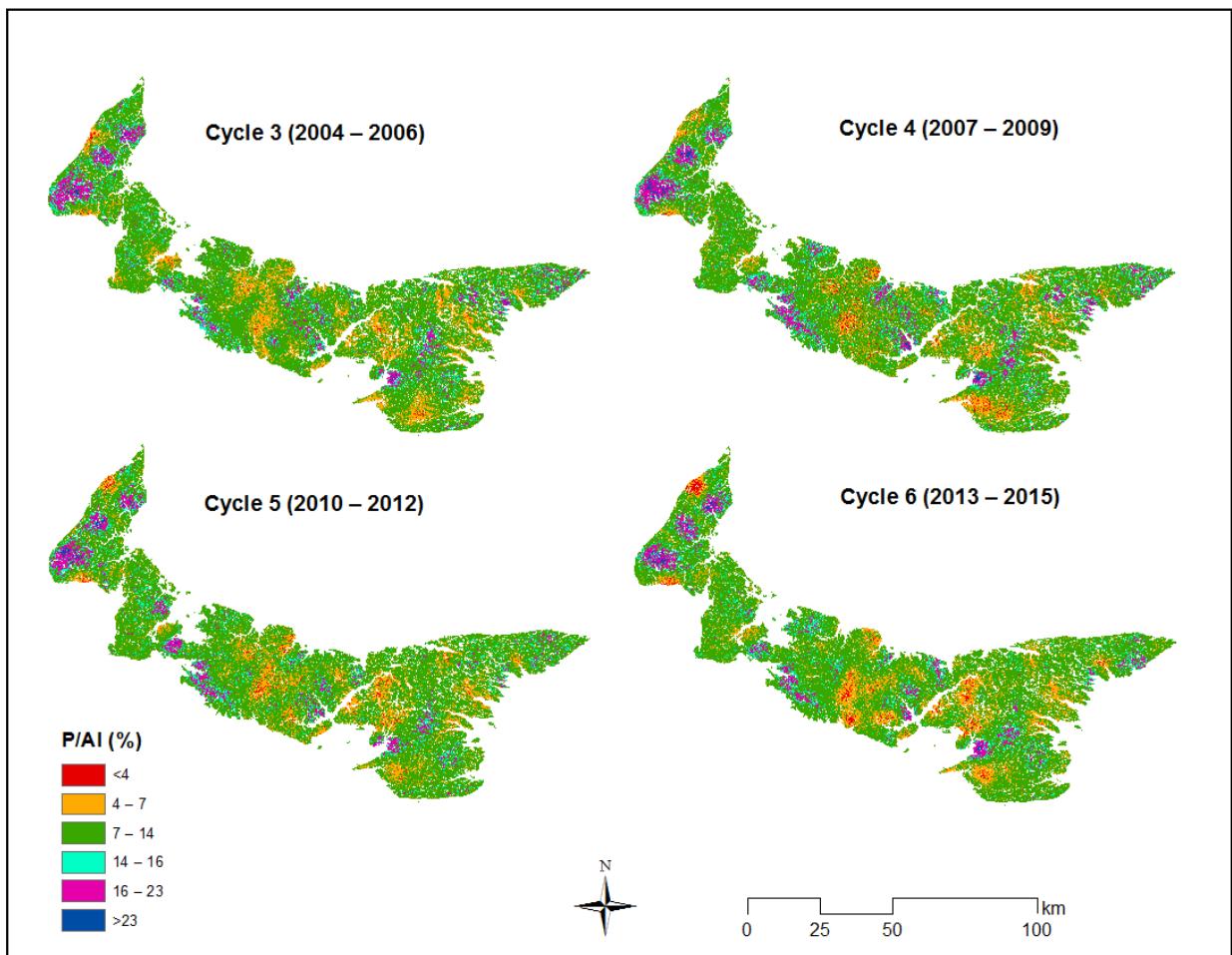


Figure 2.4. Spatiotemporal distribution of identified environmental P risk classes for soils with pH above 5.5.

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3. Chapitre 3: An Agro-Environmental Phosphorus Model for Potato in the Canadian Maritime Provinces

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Abbreviations: FN, false negative; FP, false positive; M3, Mehlich-3; NB, New Brunswick; NPV, negative predictive value; NS, Nova Scotia; PEI, Prince Edward Island; PPV, positive predictive value; PSI₁, phosphorus saturation index as expressed by the ratio of P over Al extracted with Mehlich-3; PSI₂, phosphorus saturation index as expressed by the ratio of P over the sum of (Al+Fe) extracted with Mehlich-3; RY, relative yield; TN, true negative; TP, true positive.

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Core Ideas

- Data from 42 field trials were used to develop an agro-environmental P model for potato based on soil P saturation index.
- A critical agronomic PSI (P/Al)_{M3} of 10% was identified, which corresponded to 85% of relative yield.
- Seven agro-environmental P risk classes were identified with rates ranging from 21 to 105 kg P ha⁻¹.

3.1 Résumé

Le phosphore (P) est un élément essentiel pour la pomme de terre (*Solanum tuberosum* L.), l'une des principales productions des provinces maritimes. La perte de cet élément dans les cours d'eau constitue un risque majeur pour l'environnement. L'objectif de cette étude a été d'élaborer un nouveau modèle agro-environnemental de recommandation en P de la pomme de terre basé sur l'indicateur de saturation en phosphore exprimé en $(P/Al)_{M3}$ dans les provinces maritimes. Pour ce faire, les résultats de 42 essais de fertilisation phosphatée réalisés à l'Île-du-Prince-Édouard (ÎPE), au Nouveau-Brunswick (NB) et en Nouvelle-Écosse (NE) entre 2000 et 2015 ont été analysés. Il ressort de cette étude un modèle agronomique avec deux valeurs critiques à 85% de rendement relatif et à 10% de saturation en phosphore, et un modèle de recommandation en phosphore subdivisé en sept classes de fertilité phosphatée et de risque environnemental. Les recommandations en P varient de 21 à 105 kg P ha⁻¹, soit en moyenne de un à cinq fois le niveau d'exportation du P par la pomme de terre et correspondant respectivement, aux niveaux de saturation en P des plus élevés: $(P/Al)_{M3} \geq 14$ aux plus faibles: $(P/Al)_{M3} \leq 2.5$. Il s'agit d'une première ébauche de modèle de recommandations en P pour les sols de pomme de terre des Maritimes et nécessite d'être validée par d'autres essais de fertilisation.

3.2 Abstract

Phosphorus is essential for potato (*Solanum tuberosum* L.), an economically important crop for the Canadian Maritime provinces. Nutrient loss, including P, into waterways and estuaries has contributed to reduced water quality such as algae blooms. The objective of this study was to develop an agro-environmental model for potato based on the phosphorus saturation indicator (PSI) expressed as the ratio between Mehlich-3 extractable P and Al (P/Al)_{M3}. A total of 42 field trials (6 conducted from 2000 to 2002 and 36 from 2013 to 2015) were conducted in Prince Edward Island (PEI), New Brunswick (NB), and Nova Scotia (NS) with six increasing P rates. A critical agronomic PSI of 10% was identified, which corresponded to 85% of relative yield. The P recommendation model was subdivided into seven P fertility and environmental risk classes with P rates ranging from 21 to 105 kg P ha⁻¹, i.e., on average from one to five times the level of P removal by the potato crop, corresponding to the highest ($[P/Al]_{M3} \geq 14$) and lowest ($[P/Al]_{M3} \leq 2.5$) PSI levels, respectively. Only one site was in the very low fertility class. Additional trials are needed within this class to build a more robust and representative model. This is the first agro-environmental P model for sustainable potato production that reduces the risk of P loss to watercourses in the region. The new P rates were developed at plot scale and need to be validated at larger scale.

3.3 Introduction

Potato (*Solanum tuberosum* L.) produced in the Canadian Maritime Provinces (Prince Edward Island, PEI; New Brunswick, NB; and Nova Scotia, NS) represented 38.5% of national production in 2017 (Statistics Canada, 2017). This crop is grown on acidic soils that have a high P fixing capacity owing to their high content of Fe and Al oxides and hydroxides (Hooda et al., 2000; Parent et al., 2003). The P requirements are particularly high for potato, which has a poorly developed root system resulting in low soil P utilization efficiency (Fixen and Bruulsema, 2014; Rosen et al., 2014). Phosphorus inputs generally exceed crop requirements in these regions. Excess P accumulated in the soil poses a threat to the environment, since P can be transported to watercourses and cause eutrophication of surface water (Sharpley et al., 1993; Sims et al., 1998; Beauchemin et al., 2003). The major difficulty in managing the economic and environmental issues is to determine the probabilities of crop response to added P and the risk of transport of P to the environment for different P fertility levels (Alberta Agriculture and Forestry, 2003). To ensure economically viable yields while protecting the environment, a P recommendation model must consider the agronomic and environmental aspects.

For soils in the Maritimes, Quebec, and the US Mid-Atlantic Region, P fertility is measured by means of the Mehlich-3 extraction method (Mehlich, 1984). Of the numerous P extraction methods, this one is the best adapted to soils in the acid to neutral range, which are common in these regions (Beegle and Oravec, 1990; Khiari et al., 2000; New Brunswick Department of Agriculture, Aquaculture and Fisheries, 2001; Maguire and Sims, 2002a; Sims et al., 2002; Heckman et al., 2006; Pellerin et al., 2006a), and P_{M3} was formerly considered the best diagnostic indicator of P fertility (Tran et al., 1990; Simard et al., 1991). Recently, several research studies have concluded that the saturation indices (Eq. [1] and [2]) showed stronger correlations with crop performance criteria than did P_{M3} , and were therefore more effective in preventing the environmental risk of P loss by runoff or drainage in Quebec (Khiari et al., 2000; Pellerin et al., 2006a) and in the Mid-Atlantic States (Maguire and Sims, 2002b; Sims et al., 2002). These agronomic and environmental P saturation indices were rapidly adopted and used as P fertility indices for developing the P recommendation tables for several crops in Quebec, including potato (Khiari et al., 2000),

corn (*Zea mays* L.) (Pellerin et al., 2006b), and cranberry (*Vaccinium macrocarpon* Aiton) (Parent and Marchand, 2006).

$$\text{PSI}_1 (\text{mg kg}^{-1}) = \frac{\text{P}_{\text{M3}} (\text{mg kg}^{-1})}{\text{Al}_{\text{M3}} (\text{mg kg}^{-1})} \quad (\text{Eq. 1})$$

$$\text{PSI}_2 (\text{mmol kg}^{-1}) = \frac{\text{P}_{\text{M3}} (\text{mmol kg}^{-1})}{[\text{Al}(\text{mmol kg}^{-1}) + \text{Fe}(\text{mmol kg}^{-1})]_{\text{M3}}} \quad (\text{Eq. 2})$$

These indices were also used to estimate critical values of the risk of P transport to the environment (PEI Department of Agriculture and Fisheries, 2017a; Benjannet et al., 2018). The recommendations for application of P fertilizers to potato could exceed 92 kg P ha⁻¹ in NB (New Brunswick Department of Agriculture, Aquaculture and Fisheries, 2011) and 175 kg P ha⁻¹ in PEI (PEI Department of Agriculture and Fisheries, 2017b). These levels significantly exceed P removal by the crop. A long-term study conducted by the PEI Department of Agriculture revealed that applying P fertilizer in excess of the crop P requirements caused an increase in the P concentration of soils in the region and in the risk of contamination of the surrounding water (Agriculture and Agri-Food Canada, 2016). As it is desirable to continuously update and re-evaluate information on soil testing and fertilizer recommendations (Peck and Soltanpour, 1990), we concluded that a revised P fertilization recommendation was needed to meet the crop P requirements while monitoring soil P saturation. Our objective was to develop a combined agronomic/agro-environmental P fertilization recommendation model for potato in the three Canadian Maritime Provinces by conducting multiple field trials to measure potato response to six increasing P fertilizer rates on soils of varying P content.

3.4 Theory

Phosphorus recommendation models are built by following four main steps: correlation, calibration, optimization, and recommendation (Fig. 3.1) (Dahnke and Olson, 1990; Khiari et al., 2000; Korndorfer et al., 2001; Mallarino, 2003; Pellerin et al., 2006b; Samson et al., 2008).

3.4.1. Soil test correlation

The choice of the fertility index, or soil test correlation (Dahnke and Olsen, 1990), is based on selecting an index that best describes the soil P fertility level and is highly correlated to crop response. As previously mentioned, PSI_1 (Eq. 1) and PSI_2 (Eq. 2), calculated based on the Mehlich-3 extraction, are the most common indices used for P recommendations for several crops and regions (Khiari et al., 2000; Maguire and Sims, 2002b; Sims et al., 2002; Parent and Marchand, 2006; Pellerin et al., 2006b; Guérin et al., 2007). However, many studies have reported a high correlation coefficient between PSI_1 and PSI_2 (Khiari et al., 2000; Maguire and Sims, 2002b; Sims et al. 2002; Pellerin et al., 2006a; Benjannet et al., 2018) and concluded that PSI_1 could be the best fertility index to use when building P recommendation models.

3.4.2. Soil test calibration

Soil test calibration is the process that aggregates soils into fertility classes or response categories (Dahnke and Olson, 1990). This step describes the changes in relative yield (RY) based on the fertility index. Relative yields are generally used to standardize the variability of crop response to fertilization due to soil type, site, season, or variety (Whitney et al., 1985; Belanger et al., 2002). To consider all the cultivars and all the sites in the same model, total yields (wet basis) are converted to relative yields (Giroux et al., 1984):

$$\text{RY}(\%) = \frac{\text{Average yield in the unfertilized treatment}}{\text{Maximum yield obtained on the same site}} \times 100 \quad (\text{Eq. 3})$$

For each trial, the RY is computed as the mean yield of the unfertilized treatment divided by the highest yield, then multiplied by 100 (Nelson and Anderson 1984) (Eq. [3]). A relative yield of 100% means that the addition of fertilizer did not increase crop yield. However, a relative yield of <100% means that the crop responded positively to fertilization (Nelson and Anderson, 1984; Samson et al., 2008). The limits of the soil fertility classes can be derived from the critical agronomic value determined iteratively according to the Cate–Nelson partitioning method (Nelson and Anderson, 1984) by linking

the relative yields of all sites to the selected fertility index. The critical agronomic threshold is the value above which no significant increase in yield is expected with additional inputs of the fertilizer (Dahnke and Olson, 1990; Korndorfer et al., 2001; Zamuner et al., 2017); it separates crop response from non-response (Beegle et al., 1998). The partitioning approach of Cope and Rouse (1983) suggests that three fertility classes could be identified below the critical agronomic value and three classes above it. The upper limits of the six fertility classes are 25, 50, 100, 200, 400, and 900% of the value of the critical agronomic threshold, respectively. For example, if the critical agronomic value (PSI_1) is 10%, the fertility groups would be 0 to 2.5, 2.5 to 5, 5 to 10, 10 to 20, 20 to 40, and 40 to 90%.

3.4.3. Soil test optimization

Optimum P rates are computed for each independent experimental site (Black, 1993) by plotting total yield (kg ha^{-1}) against increasing P rates (kg P ha^{-1}), and from response curves described by the most common mathematical models. The optimum P rate is zero when potato yield is not improved (plateau model) or is reduced with added P (regressive yield pattern). For the linear model, optimum P rate is the maximum rate of added P if the slope of the linear curve ($\Delta \text{Yield} [\text{kg ha}^{-1}] / \Delta \text{Rate} [\text{kg P ha}^{-1}]$) is higher than the maximum productivity (Nelson et al., 1985) (ratio of the unit price of the fertilizer to the crop price). For a linear–plateau model, optimum P rate corresponds to the intersection between the linear response and yield plateau if the slope of the linear trend exceeds maximum productivity, and is zero if it does not (Cerrato and Blackmer, 1990; Pellerin et al., 2006b). Quadratic models are very commonly used for describing the crop response to fertilizers, but they tend to overestimate optimal P rates (Cerrato and Blackmer, 1990; Dahnke and Olson, 1990). Therefore, their optimum P rates are constrained by assuming that the first derivative of the equation defining the response model is equal to the maximum productivity:

$$\frac{\delta \text{ Yield} (\text{kg ha}^{-1})}{\delta \text{ Rate} (\text{kg P ha}^{-1})} = \frac{\text{Fertilizer price} (\$ \text{kg P}^{-1})}{\text{Crop price} (\$ \text{kg}^{-1})} \quad (\text{Eq. 4})$$

The Mitscherlich model, or quadratic plateau model, is among the most commonly used models for describing crop response to fertilization, particularly crop response to P

(Bray, 1963). It also has been considered satisfactory for the site-by-site analysis of N fertilizer trials (Valkama et al., 2013). The Mitscherlich model is more realistic, as it mimics soil and environmental conditions related to the fertilization trials and can be tested as a predictive model in precision farming by combining many factors affecting the crop pattern (Parent et al., 2017). According to this model, as the amount of added fertilizer increases, the supplement yield gain for each additional fertilizer unit (the rate) (Parent et al., 2017) decreases (Rajsic and Weersink, 2008). In other words, when crop response reaches its maximum, any additional fertilizer input becomes economically disadvantageous (Gros, 1960). The Mitscherlich equation used to calculate optimum P rate is as follows:

$$\text{Log } (A-Y) = \text{Log } (A-Y_0) - CX \quad (\text{Eq. 5})$$

where A is optimal yield, Y is yield at any rate, Y_0 is yield of the control without fertilizer, C is effect factor of Mitscherlich, and X is fertilizer rate.

3.4.3. Soil test recommendation

In each soil fertility class, a single fertilizer recommendation rate is selected. The recommendation rates for a given soil fertility group can be determined using descriptive statistics when input data are not large. In such cases, the P recommendation rate could be determined by calculating the mean of all the optimum P rates in that fertility group, or by ranking the optimum P rates in ascending order across experimental sites, and then considering their median value, or could correspond to other conditional expectation percentiles (e.g., 80th percentile) (Isaaks and Srivastava, 1989; Khiari et al., 2000). For larger input data, meta-analysis (Tremblay et al., 2012) and multilevel modeling (Parent et al., 2017) have been developed to model crop yield and fertilizer requirements.

3.5 Materials and methods

3.5.1. Selection of experimental sites

To study the response of potato to P fertilization, 42 field trials were established in the three Canadian Maritime Provinces: six trials in NB from 2000 to 2002, and 36 trials from 2013 to 2015 with 23 in PEI ($46^{\circ}20'$ to $46^{\circ}40'$ N, $63^{\circ}0.9'$ to $64^{\circ}24'$ W), 7 in NB ($45^{\circ}55'$ N, $66^{\circ}36'$ W), and 6 in NS ($45^{\circ}03'$ to $45^{\circ}07'$ N, $64^{\circ}29'$ W).

The experimental sites were chosen to ensure a wide gradient of soil P fertility. Prior to the establishment of each trial, the soil at the site was sampled randomly using a 2-cm diameter stainless auger by combining and thoroughly mixing four to six subsamples taken at a depth of 0 to 15 cm. Samples were subsequently air-dried and passed through a 2-mm sieve. A subsample was ground using a mortar and pestle, then passed through a 500 mesh for total C and N analyses. Soil texture was determined by the hydrometer method (Sheldrick and Wang 1993). Soil pH_{water} was measured using a 1:1 soil/solution ratio (10 g soil:10 mL water). Cation exchange capacity was estimated as the sum of Mehlich-3 extractable bases, whereas soil acidity was estimated based on the buffer pH. Soil organic C was measured by dry combustion using an Elementar analyzer (Vario Max, Element Analyzer, Hanau, Germany), and percent soil organic matter (%OM) was estimated as the product of organic C and a conversion factor of 1.72. Phosphorus, Al, and Fe were determined by the Mehlich-3 method (Mehlich, 1984). A 2.5-mL scoop was used to weigh the samples, and the extraction was performed in 25 mL of extracting solution. The elements were quantified by plasma emission spectroscopy (ICP) (Varian 820-MS).

The main soil series encountered were Charlottetown in PEI (Nyiraneza et al., 2017a), Holmesville in NB (Nyiraneza et al., 2017b), and Queens in NS (Nyiraneza et al., 2017c). Charlottetown and Queens soil series correspond to Haplorthods in the United States soil classification system; Holmesville series correspond to Dystrochrepts.

3.5.2. Experimental design and treatments

Each trial consisted of six treatments and three replications in a randomized complete block design. Treatments were six increasing rates of P applied in the form of triple superphosphate (0–46–0). Rates were assigned to sites based on the value of the P saturation index (PSI_2) defined by Sims et al. (2002) (Table 3.1). The experimental units were composed of four 8-m long rows spaced 0.91 m apart. Experimental units were spaced 2 m apart, and blocks at least 6 m apart. Seed potato spacing within rows was 0.381 m. The furrows were opened manually or mechanically, depending on the equipment available at the experimental sites. Nitrogen and K were applied as ammonium nitrate (34–0–0) and muriate of potash (0–0–60), respectively, to meet recommended potato production requirements at rates ranging from 150 to 200 kg ha⁻¹ for both fertilizer products (New

Brunswick Department of Agriculture Aquaculture and Fisheries, 2001; Nyiraneza et al., 2017b, 2017c; PEI Department of Agriculture and Fisheries 2017b). The fertilizers were weighed for each row to ensure accuracy and were applied by hand or machine in the spring at the time of planting, banded 5 cm below the seed pieces. No additional fertilizer was applied during the growing season. Planting was performed in May and/or June, and harvest was in September and/or October each year. The details on the phytosanitary treatments are provided in Nyiraneza et al. (2017b, 2017c).

The potato varieties grown were Russet Burbank (grown on 50% of the experimental sites), Shepody (26% of the sites), Superior (14% of the sites), and Alta Cloud and Alta Strong (5% of the sites each). The yields (wet basis) were determined in one of the middle rows of each plot. The tubers were harvested, weighed, and sorted by categories according to the market criteria of each province. The marketable yield in each province corresponded to tubers measuring 3.8 to 8.9 cm in diameter in PEI, 5.1 to 11.4 cm in NB, and 4.8 to 9.5 cm in NS. Tubers that were misshapen, green, rough, or with blackheart or hollow heart were discarded.

3.5.3. Agro-environmental classification and phosphorus recommendation model

Agronomic (this study) and environmental (Benjannet et al., 2018) thresholds as PSI_1 were combined to build an agro-environmental model. Three fertility classes (medium-low, low, and very low) were defined below the critical agronomic threshold, according to the procedure of Cope and Rouse (1983). Their respective upper limits corresponded to 100, 50, and 25% of the value of this critical threshold. The six environmental P risk classes identified by Benjannet et al. (2018) for PEI soils with pH above 5.5 were as follows: very low risk, $(\text{P}/\text{Al})_{M3}$ ratio from 0% to 4%; low risk, $(\text{P}/\text{Al})_{M3}$ ratio from 4 to 7%; moderate risk, $(\text{P}/\text{Al})_{M3}$ ratio from 7 to 14%; high risk, $(\text{P}/\text{Al})_{M3}$ ratio from 14 to 16%; very high risk, $(\text{P}/\text{Al})_{M3}$ ratio from 16 to 23%; and extremely high risk, $(\text{P}/\text{Al})_{M3}$ ratio $> 23\%$. Based on this study, three critical environmental thresholds (14, 16, and 23%) delimited four environmental risk classes: medium-high, high, very high, and extremely high. Seven classes were identified to assess the level of fertility and environmental risk.

The optimum P rates at each experimental site were obtained by equating the first derivatives of the response curve to the maximum productivity (Nelson et al., 1985) and assuming that the unit price of the fertilizer is Can\$2.75 kg⁻¹ P and that the crop price is Can\$0.3 kg⁻¹ (Eq. [5]). The mathematical model chosen to calculate the optimal P rate in each trial was the most significant model with the highest R^2 . A single P recommendation rate was determined in each of the seven fertility and environmental risk classes by considering the mean of the optimal P rates of the sites within each class.

3.5.4. Statistical analysis

The critical agronomic threshold and the corresponding RY were determined iteratively according to the Cate–Nelson partitioning method (Nelson and Anderson, 1984) using the R companion package (Mangiafico, 2013) of the R software (R Development Core Team, 2013). The Cate–Nelson method makes it possible to determine the critical fertility for development of the fertilization tables (Dahnke and Olson, 1990). The method involves identifying these critical values based on an analysis of the distribution of the values of the sum of squares (SS). It endeavors to determine the value of PSI_1 that maximizes the SS, which is then used to divide the points into two groups (Nelson and Anderson, 1984). Mangiafico (2013) found that the RY of the model corresponded to the value that minimized the number of points in the error quadrants. The performance criteria of a partitioning model are R^2 (accuracy), specificity, sensitivity, positive predictive value (PPV), and negative predictive value (NPV). Binary partition becomes more efficient as its performance criteria approach unity (Parent et al., 2012; Parent, 2013).

The equation of the fertilization response model for each site was determined by using the Excel software program (Microsoft Excel 2010), either the trend curve function for linear and polynomial models or the solver function for the Mitscherlich model (Eq. [4]).

3.6 Results and discussion

3.6.1. Soil characteristics and crop yield

The majority of the soils had a sandy loam texture, low clay content ranging from 5 to 15%, and OM content ranging from 1.6 to 4.1%, and they were slightly to moderately

acidic according to the Whiteside (1965) classification, with pH ranging from 5.0 to 6.8 (Table 3.2). These soils are suitable for potato crops, which are often grown on acid, coarse-textured soils (FAO, 2008). Since the sites were chosen to ensure coverage of a wide spectrum of P fertility, the P_{M3} and PSI_1 indicators had the largest coefficients of variation (51.1 and 51.9%, respectively). The PSI_1 ranged from 2.3 to 19.2% (Table 3.2). A previous study in PEI established environmental P risk classes based on soil P saturation in PEI and reported that 2% of the soils had a very low risk, $0 < PSI_1 < 4\%$; 43% a low risk, $4\% < PSI_1 < 7\%$; 38% a medium risk, $7\% < PSI_1 < 14\%$; 2% a high risk, $14\% < PSI_1 < 16\%$; and 14% a very high risk, $16\% < PSI_1 < 23\%$. No soil presented an extremely high environmental risk, $PSI_1 > 23\%$ (Benjannet et al., 2018).

The marketable yields obtained at the 42 sites ranged from 3 to 61 Mg ha⁻¹ (Table 3.3). The lowest yields were obtained in NS with the Superior variety. At the highly P-saturated sites ($PSI_2 > 11\%$), neither Russet Burbank nor Shepody responded to the addition of P fertilizer. The greatest response to P fertilization was observed in soils having a $PSI_2 < 6\%$, with a significant response at 85% of the sites (Table 3.3).

3.6.2. Agro-environmental model

The distribution pattern of the RY based on the fertility indices was almost the same whether P_{M3} or PSI_1 was used, and a RY of 85% separated the two P fertilization response groups, high response and low response (Fig. 3.2a, 3.2b).

The Cate–Nelson partitioning method reports the ascending section located in the True Positive (TP) quadrant, where the responses of the potato crop to the P fertilizers were high and the RY values ranged from 44 to 83% (Fig. 3.2b). The yield stability plateau is located in the True Negative (TN) quadrant, where the responses were rather low and the RY values ranged from 85 to 100% (Fig. 3.2b). This yield stability interval exhibits a range lower than the 75 to 100% proposed by Cope and Rouse (1983). The minimum number of points in the error quadrants, False Positive (FP) and False Negative (FN) representing the sites with deviation from expected behavior (Beegle, 1995), obtained from this partition was six (Fig. 3.2b); this corresponded to a relative yield critical value of 85% (Fig. 3.2a, 3.2b). The Cate– Nelson method is similar to an analysis of variance, which maximizes the SS between two groups (Nelson and Anderson, 1984). This maximum SS allowed the

experimental sites to be divided into two P saturation groups: a group of soils with low P saturation having a $\text{PSI}_1 < 10\%$ (27 experimental sites), and a higher saturation group having a $\text{PSI}_1 > 10\%$, above which potato response to P fertilizer is unlikely (15 experimental sites) (Fig. 3.2b, 3.2c). This Cate–Nelson procedure exhibited robustness (R^2) of 86%, which is the ratio between the true trends (TN+TP) relative to the total of the observations (Fig. 3.2d). This means that out of 100 diagnoses, 86 will be correctly classified in one or the other of the two groups of high or low response to P fertilization.

Specificity ($\text{TN}/[\text{TN}+\text{FP}]$) and sensitivity ($\text{TP}/[\text{TP}+\text{FN}]$) are measures that give the percentages of positive observations in the model (Fig. 3.2d). As an example, a specificity of 76% means that the probability of correctly diagnosing the sites that have a P saturation $> 10\%$ and grouping them together in the same TN quadrant that did not respond to P fertilization is 76% (Fig. 3.2d). Similarly, a sensitivity of 92% indicates that, in the majority of cases, the sites whose P saturation level is $<10\%$ and which showed a high response to the P fertilizer ($\text{RY} < 85\%$) were correctly diagnosed as TP.

The PPV ($\text{TP}/[\text{TP}+\text{FP}]$) and NPV ($\text{TN}/[\text{TN}+\text{FN}]$) values were comparable (Fig. 3.2d). The PPV indicates the probability that the crop will respond to P fertilization ($\text{RY} < 85\%$) when the fertility index is $<10\%$, whereas the NPV indicates the probability that no response to fertilization is present when the soil PSI_1 is $>10\%$.

Critical agronomic thresholds are quite variable among crops, and the threshold for potato is generally higher than for others (Howard, 2006), since potato is a high-nutrient-demanding crop grown in acid soils with high P-fixing capacity. In Quebec and North Carolina, for example, a PSI_1 threshold of 10% is twice that of corn (McCollum, 1991; Khiari et al., 2000; Pellerin et al., 2006b). This is normal, since corn has a more developed and deeper root system, giving it a greater root-to-soil exchange surface for P absorption. This PSI_1 threshold of 10% for potato crops in the Canadian Maritimes was slightly higher than the 8% threshold in Quebec (Khiari et al., 2000). This can be attributed to the OM content of the soils in the two regions and/or higher Fe contents in PEI soils. The coexistence of OM with P makes this element less fixed and therefore more available to the plant (Beaudin et al., 2008). Since Quebec soils are on average richer in OM than those of the Maritimes, their agronomic thresholds are likely to be reached at lower P levels.

Conversely, the soils of the Maritimes have an abundance of iron oxides with higher P-fixing capacity, and the plant consequently reaches agronomic threshold at higher P -values. Despite the high P requirements of the potato crop, the PSI_1 agronomic threshold of 10% remains below the critical environmental thresholds of 15% in Quebec (Khiari et al., 2000) and 14% in PEI (Benjannet et al., 2018; PEI Department of Agriculture and Fisheries 2017a). In Delaware (USA), Sims et al. (2002) established two PSI_1 environmental thresholds of 16% and 23%, above which runoff and drainage waters, respectively, reach a critical P enrichment level. With the exception of organic soils (Guerin et al., 2007), several research studies had concluded that agronomic thresholds often tend to be significantly lower than the critical environmental values in mineral soils (Khiari et al., 2000; Sharpley et al., 2002; Parent and Marchand, 2006; Pellerin et al., 2006b). In the yield stability plateau, three environmental thresholds of soil P saturation of 14, 16, and 23% of PSI_1 were established based on the study of Benjannet et al. (2018), to delimit four fertility and environmental risk classes for the Maritimes: medium-high (10–14%), high (14–16%), very high (16–23%), and extremely high (>23%).

The 10 to 14% class represents the medium-high risk category, because its upper limit of 14% corresponds to the limit of sufficiency for several P fertility systems for potato. Benjannet et al. (2018) demonstrated that a soil saturated to 14% can desorb P into water by up to 9.6 mg water extractable P L⁻¹, a value close to the thresholds of sufficiency of 9.7 mg P_{water} L⁻¹ in Quebec and PEI (Khiari et al., 2000; Benjannet et al., 2018), 10.0 mg P_{water} L⁻¹ in Germany (Landwirtschaftskammer, 1996), and 10.9 mg P_{water} L⁻¹ in the Netherlands (Breeuwsma and Silva, 1992). According to several authors, a response to fertilization in high-fertility soils is unlikely (Beegle, 1995; Sims et al., 2002; Pellerin et al., 2006b), but not impossible (Nelson and Hawkins, 1947; Dyson and Watson, 1971; Rosen et al., 2014). Above the saturation threshold of 14%, the environmental risk begins to increase progressively. Caution must be exercised when managing P applications to these soils, since excessive fertilization could result in eutrophication of the surrounding water (Sims et al., 2002).

In the ascending section (TP) of response to P fertilization (Fig. 3.2b), the two agronomic thresholds of Cope and Rouse (1983), i.e., 2.5 and 5% soil P saturation, were

considered to delimit three fertility and environmental risk classes: very low, 0 to 2.5%; low, 2.5 to 5%; and medium-low, 5 to 10%. These three potato P fertility classes in the Maritimes were very comparable to the first three classes of the fertilization table for Quebec (CRAAQ, 2010). Because the probability of response to P fertilization below the agronomic threshold is generally medium to high (Bundy et al., 2005), fertilization of these soils is essential and producers can apply P fertilizer with little risk to the environment. The crop response to fertilization in these classes can be explained by the fixing capacity of soils in the TP quadrant (Fig. 3.2b). In fact, 57% of these soils had a medium fixing capacity ($1100 \text{ mg kg}^{-1} < \text{Al}_{M3} < 1600 \text{ mg kg}^{-1}$) and 35% had a high fixing capacity ($\text{Al}_{M3} > 1600 \text{ mg kg}^{-1}$) according to the classification of Tran et al. (1990). Because of the predominance of Al in these soils, they act as P sinks. Consequently, even if high rates are applied, P is rapidly fixed and becomes less available to the crop. In this category, it is preferable to apply P in bands close to the seed to maximize the efficiency of P utilization and minimize exposure to the P-fixing agents in the soil matrix and transport in runoff (Gerwing and Gelderman, 2005; Beaudin et al., 2008; Ruark et al., 2014). The low responses to P fertilization of the four sites in the FP quadrant (Fig. 3.2b) despite their low P saturation level could be due to environmental factors or to the presence of limiting factors other than P (Havlin et al., 1999).

3.6.3. Recommendation model

The P recommendation model, which presents different optimal rates (kg P ha^{-1}) based on the PSI_1 index, produced somewhat scattered points, with a general descending pattern (Fig. 3.3). The optimum rates ranged from 21 to 105 kg P ha^{-1} . The very low class (0–2.5%) included only one site whose RY was among the lowest (approximately 50%) with a recommended rate of 105 kg P ha^{-1} . Its fertilization response curve was linear with a slope of 157 (Fig. 3.4), much higher than the maximum productivity of 9 (Eq. [5]). This means that crop yields would have been higher if a higher rate of fertilizer had been applied. This could be explained by the very high fixing capacity of this site, which had one of the highest Al_{M3} contents ($\text{Al}_{M3} = 1915 \text{ mg kg}^{-1}$). However, because this is only one site and extrapolation is not possible, the rate of 105 kg P ha^{-1} was maintained as the representative rate of this interval. This rate exceeds the recommended rate for potato in Quebec

(CRAAQ, 2010) and Ontario (Ontario Ministry of Agriculture, Food and Rural Affairs, 2012) by 18 kg P ha⁻¹, and the rate recommended in NB (New Brunswick Department of Agriculture Aquaculture and Fisheries, 2011) by 13 kg P ha⁻¹, for the same P saturation level. In Idaho (Stark et al., 2004), the recommended rate is much higher when P fertility is low, and it is approximately 156 kg P ha⁻¹. In the very low fertility class, P fertilization of up to five to six times the level of P removal by the potato crop (21 kg P ha⁻¹) (CRAAQ, 2010) is necessary to maximize crop response. However, we suggest increasing the number of experimental sites in this class to obtain more robust statistics and a more representative model.

The low fertility class (2.5–5%) included seven sites with optimal rates ranging from 0 to 105 kg P ha⁻¹ and an average of 81 kg P ha⁻¹ (Fig. 3.3; Table 3.4), which is comparable to the recommended rates in NB (79–92 kg P ha⁻¹) (New Brunswick Department of Agriculture, Aquaculture and Fisheries, 2011) and a little higher than the recommended rate in Quebec (65 kg P ha⁻¹) (CRAAQ, 2010). In this class, the recommended rate was four times the level removed by the crop (21 kg P ha⁻¹, CRAAQ, 2010).

The optimal rates of the 19 sites in the medium fertility class (5–10%) showed wide variability, ranging from 0 to 105 kg P ha⁻¹ (Fig. 3.3; Table 3.4). The average of 70 kg P ha⁻¹ was close to the 66 kg P ha⁻¹ recommended in Quebec (CRAAQ, 2010) for the same fertility interval. The variability could be associated with differences in weather (rainfall and temperature) and soil properties. Thus, as the critical saturation of 10% is approached, we observe less difference between the quantity of P fertilizer applied and removed by the crop.

Above the critical agronomic threshold, the optimal rates stabilized at zero at 13 of the 15 sites whose P saturation level exceeded 10% (Fig. 3.3). This indicates that no P fertilization is necessary in these soils. Nonetheless, it is very rare for a zero rate to be recommended for potato, even in soils with high P fertility levels (Davenport et al., 2005). However, there are exceptions, such as in Idaho (Stark et al., 2004) and South Dakota (Gerwing and Gelderman, 2005). Under the cold, wet conditions of the Maritimes, characterized by a short growing season, it is more prudent to apply a starter P fertilizer.

Indeed, when the soil P saturation level is high, the starter fertilizer usually increases crop yields, since the crop responds efficiently to the starter (Bundy et al., 2005). The efficiency of a starter fertilizer at high levels of P saturation has been proven in several regions and for various crops (Teare and Wright, 1990; Gordon et al., 1997). A rate equivalent to the rate of removal by the potato crop, i.e., 21 kg P ha^{-1} , is therefore recommended (Fig. 3.3). This rate matching potato P uptake does not pose an environmental threat, even in highly fertile soils.

3.7 Conclusion

Based on an initial calibration, it was found that substituting the PSI for the P_{M3} fertility indicator yielded the same agronomic interpretation. The PSI_1 was calibrated again with the relative yields of the 42 experimental sites in the Canadian Maritime Provinces. This calibration resulted in a critical agronomic value of 10% for PSI_1 , from which seven classes of agronomic and environmental interpretation were established: 0 to 2.5, 2.5 to 5, 5 to 10, 10 to 14, 14 to 16, 16 to 23, and $\text{PSI}_1 > 23\%$. An optimization step for each site showed that the economically advantageous rates of P fertilization ranged from 0 to 105 kg P ha^{-1} . These three steps of initial calibration, re-calibration, and optimization enabled us to develop a preliminary agro-environmental P recommendation model for potato crops in the Maritimes. Below an agronomic critical PSI_1 of 10%, P fertilization was required. Between the PSI_1 agronomic threshold of 10 and 14%, the agronomic P sufficiency level for potato was met while remaining below the level of environmental risk. This provides a comfort zone, but would require frequent monitoring of soil P test levels. Above a PSI_1 of 14%, the P recommendations become more restrictive and are limited to the level of P removal by the potato crop. The new P rates were developed at plot scale and need to be validated at larger scale.

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Table 3.1. Phosphorus rates (treatments) applied according to the soil phosphorus saturation index (PSI_2).

| PSI_2^\dagger | Treatments | | | | | |
|-------------------------|----------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| | T₀ | T₁ | T₂ | T₃ | T₄ | T₅ |
| kg P ha^{-1} | | | | | | |
| > 11% (high P site) | 0 | 9 | 17 | 26 | 35 | 52 |
| 6–11% (medium P site) | 0 | 9 | 17 | 35 | 52 | 79 |
| $\leq 6\%$ (low P site) | 0 | 17 | 35 | 52 | 79 | 105 |

† PSI_2 saturation index according to Sims et al. (2002).

$$\text{PSI}_2 (\text{mmol kg}^{-1}) = \frac{\text{P}_{M3} (\text{mmol kg}^{-1})}{[\text{Al} (\text{mmol kg}^{-1}) + \text{Fe} (\text{mmol kg}^{-1})]_{M3}}$$

Table 3.2. Range of soil properties of the potato fertilizer trials.

| Soil property | Min.–Max. | Standard deviation | Mean | Coefficient of variation, % |
|---|------------|--------------------|--------|-----------------------------|
| pH | 5.0–6.8 | 0.4 | 6.0 | 6.9 |
| CEC, cmol _c kg ⁻¹ † | 4–13 | 2.2 | 9.0 | 23.9 |
| Organic matter, % | 1.6–4.1 | 0.6 | 2.9 | 21.3 |
| Sand, % | 24.9–72.9 | 11.1 | 54.6 | 20.2 |
| Clay, % | 5.0–15.0 | 2.4 | 8.8 | 27.4 |
| Silt, % | 14.6–64.5 | 11.3 | 36.6 | 30.8 |
| P _{M3} , mg kg ⁻¹ | 43.2–296.5 | 62.6 | 122.4 | 51.1 |
| Al _{M3} , mg kg ⁻¹ | 999–2175 | 278.1 | 1461.8 | 19.0 |
| Fe _{M3} , mg kg ⁻¹ | 80–382 | 69.8 | 190.0 | 36.8 |
| PSI _I (%) = (P/Al) _{M3} | 2.3–19.2 | 4.4 | 8.5 | 51.9 |

† CEC, cation exchange capacity

Table 3.3. Overview of the number of trials, varieties tested, response to phosphorus (P) fertilization and yield intervals in each fertility group.

| PSI ₂ | Number of trials | Number of sites per cultivar type | Response to P fertilization | Yield (wet basis) |
|------------------|------------------|---|--------------------------------|---------------------|
| | | | | Mg ha ⁻¹ |
| > 11% | 6 | 5 Russet Burbank 1 Shepody | Significant in 0% of cases | 11–51 |
| 6–11% | 16 | 6 Russet Burbank 7 Shepody 3 Superior | Significant in 38% of cases | 5–55 |
| < 6% | 20 | 10 Russet Burbank 3 Shepody 3 Superior 2 Alta Cloud 2 Alta Strong | Significant in 85% of cases | 3–61 |

Table 3.4. Statistics associated with optimal P rates (kg P ha^{-1}) within each P saturation index (PSI_1) class.

| PSI_1 (%) | Number of sites | Min. | Max. | Mean | Median | Standard deviation |
|--------------------|-----------------|------|------|-----------------|--------|--------------------|
| 0–2.5% | 1 | - | - | - | - | - |
| 2.5–5% | 7 | 0 | 105 | 69 ^Y | 82 | 37.7 |
| 5–10% | 19 | 0 | 105 | 63 ^Y | 73 | 37.7 |
| 10–14% | 10 | 0 | 79 | 10 | 0 | 27.8 |
| 14–16% | 2 | 0 | 0 | 0 | 0 | 0 |
| 16–23% | 5 | 0 | 37 | 7 | 0 | 16.4 |
| >23% | 0 | - | - | - | - | - |

^Y These values correspond to the real means calculated in both low and medium fertility classes with PSI_1 ranging, respectively from 2.5 to 5% and from 5 to 10%. However, for practical reasons, three experimental trials corresponding to an optimal P rate of 0 kg P ha^{-1} have been removed from the calculation. Thus the P recommended values have been adjusted in order to meet the real crop requirement in these fertility classes.

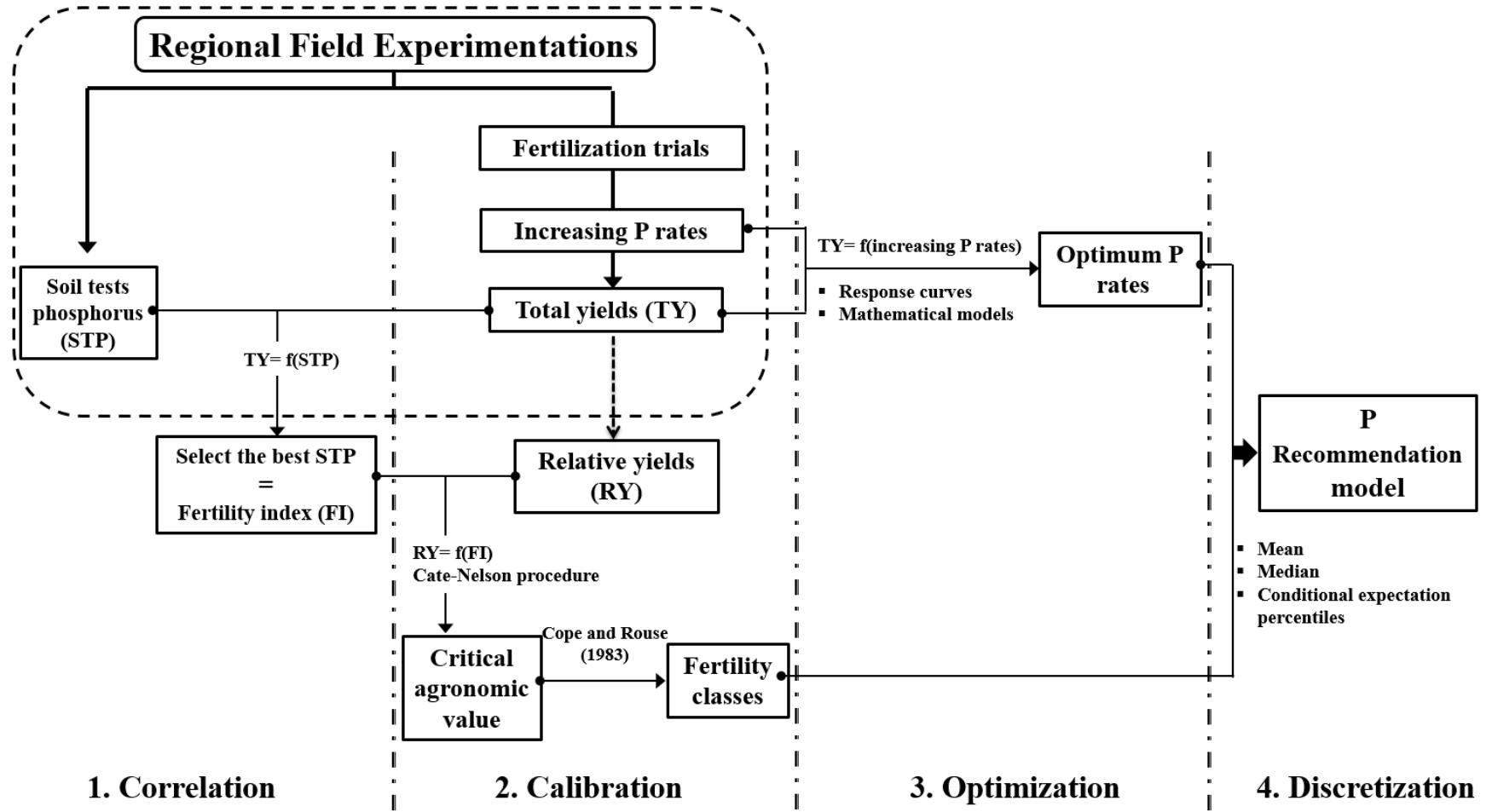


Figure 3.1. The four major steps of building a P recommendation model.

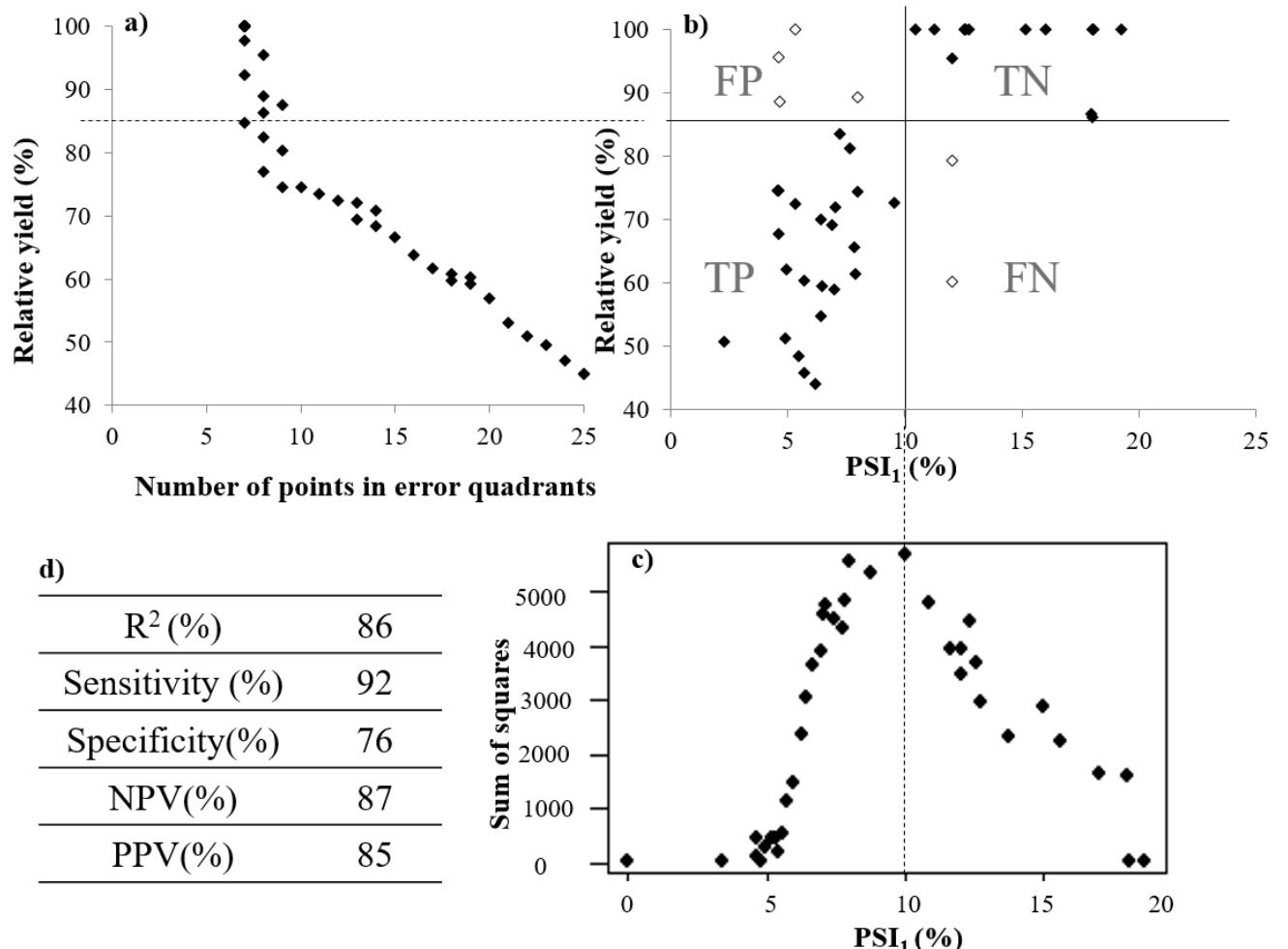


Figure 3.2. Construction of the diagnostic and statistical model of the Cate–Nelson classification showing the (a) number of points outside the model for determination of the critical relative yield (RY); (b) Cate–Nelson graph for the critical thresholds identified. FP, false positive; TP, true positive; TN, true negative; FN, false negative; (c) sum of squares for determination of the critical PSI_1 ; and (d) performance indices of the partition model. R₂, Accuracy; NPV, negative predictive value; PPV, positive predictive value.

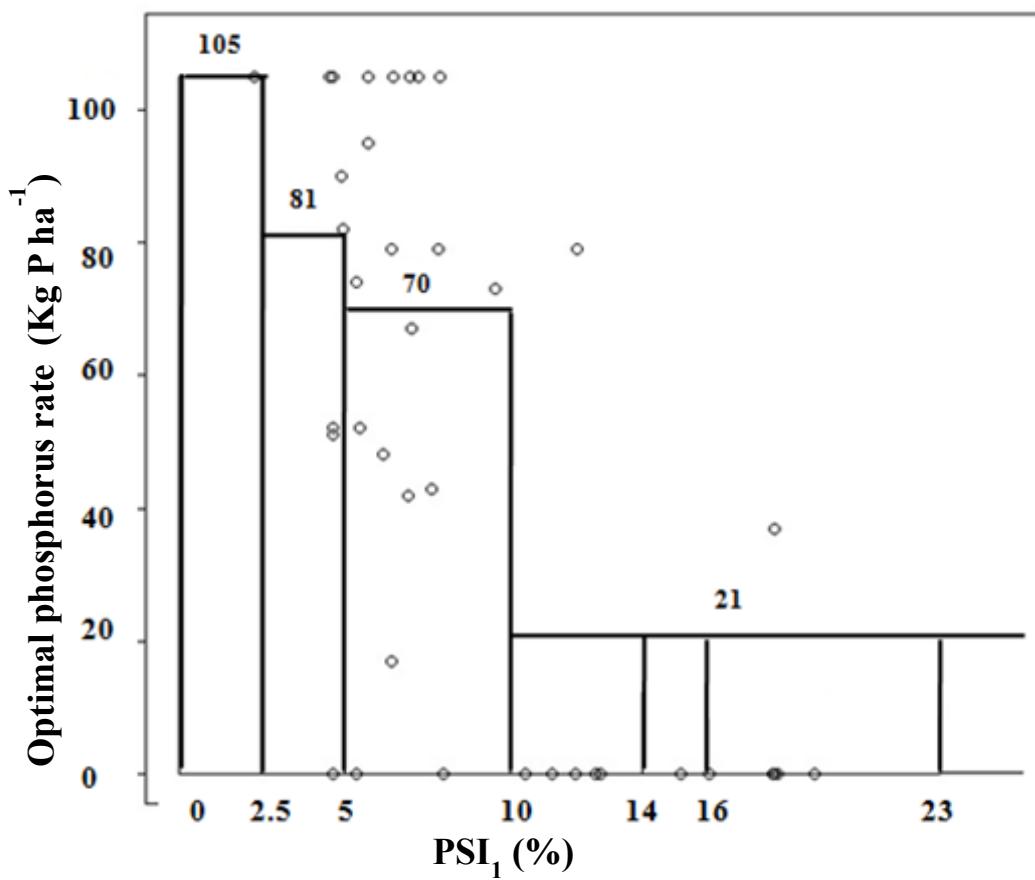


Figure 3.3. Phosphorus recommendation model for potato crops in the Canadian Maritimes.

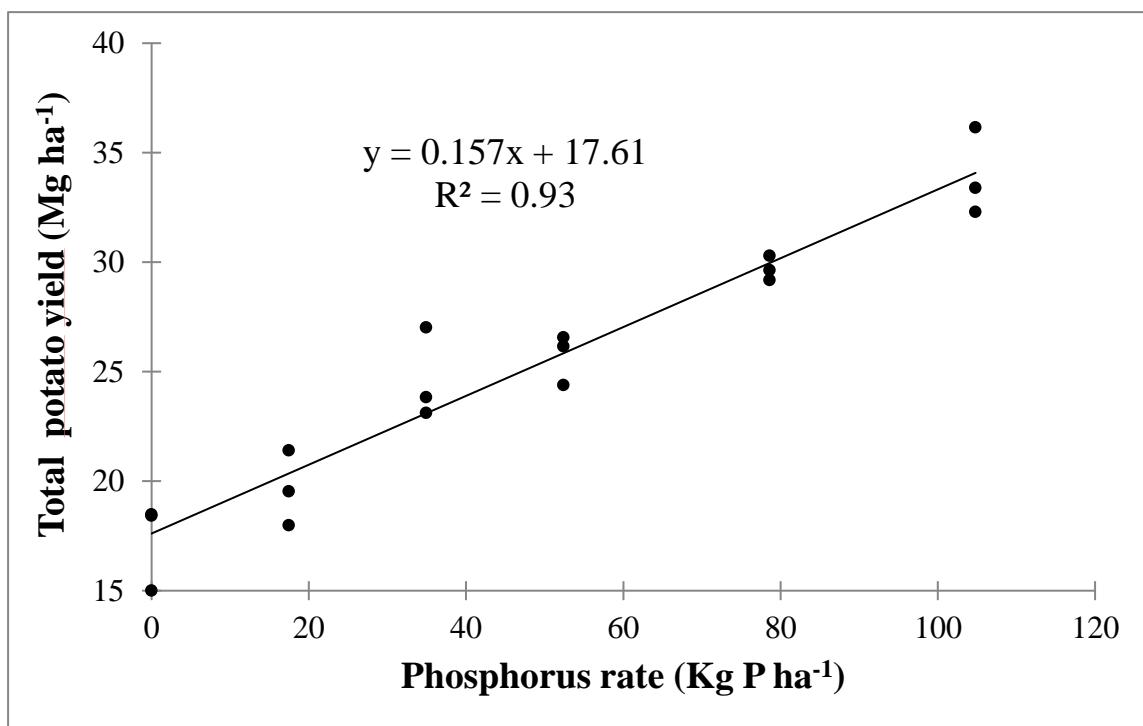


Figure 3.4. Average yield vs. P rates of the site belonging to the very low fertility class (0-2.5% PSI₁).

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4. Chapitre 4: Potato Response to Struvite in Comparison with Conventional Phosphorus Fertilizer in Eastern Canada.

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Key words: *Solanum tuberosum* L., phosphorus, apparent phosphorus recovery, struvite, petiole phosphorus concentration, ion exchange membranes.

Abbreviations: **AEMs**, anion exchange membranes; **APR**, apparent phosphorus recovery; **Cryst.**, struvite manufactured by Ostara Nutrient Recovery Technologies Inc. Crystal Green[®]; **NB**, New Brunswick; **NS**, Nova Scotia; **MY**, marketable potato yield; **PEI**, Prince Edward Island; **PU**, phosphorus uptake; **QC**, Quebec; **TY**, total potato yield; **TSP**, triple superphosphate.

4.1 Résumé

Dans les sols acides hautement fixateurs du phosphore (P), les apports en engrais phosphatés dépassent souvent les besoins des cultures, surtout celles exigeantes en P telles que la pomme de terre (*Solanum tuberosum L.*). Ceci pourrait conduire à l'eutrophisation. L'utilisation d'un engrais P recyclé sous forme de struvite pourrait être efficace. L'objectif de cette étude a été d'évaluer la réponse de la pomme de terre à la fertilisation phosphatée sous forme de triple superphosphate (TSP) ou d'un mélange de TSP avec de la struvite. Les essais ont été réalisés entre 2016 et 2018 dans 12 sites situés à l'Est du Canada. Un total de huit traitements avec cinq doses croissantes de TSP (0, 26, 52, 79, et 105 kg P ha⁻¹) et trois combinaisons de TSP avec la struvite (25%, 50% et 75% struvite) à une dose de 79 kg P ha⁻¹ ont été appliqués. Des indices de disponibilité du P dans le sol (P extrait au Mehlich-3 (P_{M3}), P adsorbé par les membranes d'échanges anioniques (P_{AEMs})) et dans la plante (P des pétioles) ont été mesurés tout au long de la saison de croissance. Dans tous les sites, il y a eu une réponse de la pomme de terre à la fertilisation phosphatée. Les différentes combinaisons de struvite avec le TSP ont donné des rendements de pomme de terre, des concentrations de P dans les pétioles, des teneurs de P_{M3} et de P_{AEMs} comparables à ceux obtenus avec la même dose apportée sous forme de 100% de TSP. Des exceptions ont été notées dans certains sites où une plus grande proportion de struvite (75%) a été associée avec des rendements moins élevés. Nos résultats suggèrent que l'utilisation de la struvite pourrait constituer une alternative durable aux engrais de sources naturelles de P.

4.2 Abstract

In acidic soils with high phosphorus (P) fixing capacity, P application at levels higher than crop requirements is common in crops with high P demands such as potato (*Solanum tuberosum* L.), which could lead to eutrophication. Recycled sources of P fertilizer, such as struvite, can be efficient in meeting crop P demands while minimizing P losses. The objective of this study conducted in four provinces over three years (2016–2018, with 12 sites in total) in Eastern Canada was to assess potato response to P fertilizer as triple superphosphate (TSP) or a mixture of TSP with struvite. Eight P treatments, with five increasing P rates as TSP (0, 26, 52, 79, and 105 kg P ha⁻¹) and three treatments as a mixture of TSP and struvite (25%, 50% and 75% struvite) at a rate of 79 kg P ha⁻¹ were applied. Phosphorus P availability indices measured multiple times over the growing season were composed of Mehlich-3 extracted P (P_{M3}), soil P adsorbed on anion exchange membranes (P_{AEMs}), and petiole P-PO₄ (P_{petiole}) concentrations. There was a potato yield response to P inputs at all sites, with the control treatment showing lower potato yield at all sites. Potato yield, P_{petiole}, P_{M3} and P_{AEMs} were comparable between TSP at 100% and TSP+struvite at the same rate, except at one site where struvite at higher proportion depressed yield and was associated with lower P uptake (PU). Overall, the use of struvite could be a sustainable mean to address long-term globally P scarcity.

4.3 Introduction

Phosphorus (P) is one of the elements essential for plant growth and crop production (Edixhoven et al., 2013). Application of P fertilizer is required to sustain high yields in soils with low available P (Johnston and Richards, 2003). In agriculture, most commercial P fertilizers are derived from phosphate rock (PR) (Van Kauwenbergh et al., 2013; Talboys et al., 2016). According to Edixhoven et al. (2013), nearly 82% of global PR is used for fertilizer production, and Rahman et al. (2011) reported that the consumption of PR is over 1 million tons yearly. However, PR is concentrated in a few countries only (75% in Morocco) (Reijnders, 2014) and is a finite, non-renewable resource. With such high consumption, global reserves of PR are predicted to be depleted within the next century (Cordell et al., 2009; Reijnders, 2014; Reinhard et al., 2017), leading to serious consequences for agricultural production and food security (Cordell et al., 2009).

Potato (*Solanum tuberosum* L.) has a particularly high demand for P. However, its poorly-developed root system results in low P use efficiency when P fertilizer is applied to increase production. In Eastern Canada, potato is mostly grown on coarse-textured acidic soils characterized by a high P-fixing capacity (Hooda et al., 2000; Parent et al., 2003) due to high aluminum (Al) and iron (Fe) contents. Thus, P fertilizer inputs in practice are higher than plant uptake to compensate for P fixation. However, conventional P fertilizers are highly water-soluble and can potentially raise P concentrations in the soil solution and sediment. As a result, P is transported from agricultural soils into adjacent watercourses through runoff, drainage, or erosion, leading to adverse environmental impacts such as surface water eutrophication (Sharpley et al., 2013; Nair, 2014; Li et al., 2019). This is particularly true in Atlantic

Canada, with its high annual precipitation and rolling topography in potato production areas.

The dependence of agriculture productivity on PR and growing eutrophication issues have prompted research initiatives to find sustainable solutions that mitigate P-related impacts on the environment and reduce dependence on industrial fertilizers. One solution is the use of recycled P from waste materials. Struvite recovered during the wastewater treatment process is a potential P source and alternative to PR (Huang et al., 2018). Struvite, or magnesium ammonium phosphate hexa-hydrate ($\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$), is a crystalline compound with a high P and nitrogen (N) content that can be recovered from different types of wastewater (Kataki et al., 2016a) using various processes (Rahman et al., 2014; Li et al., 2019). Rahman et al. (2011) and Cho et al. (2009) have reported that it is possible to remove 93% of P and 40% of N from wastewater through struvite crystallization. In addition to supplying N and magnesium (Mg), struvite is also considered as a P fertilizer source and alternative to natural PR (Münch and Barr, 2001; Shu et al., 2006). Unlike conventional P fertilizers, struvite has low solubility in water (Bhuiyan et al., 2007; Massey et al., 2009; Cabeza et al., 2011). The high solubility of mineral P fertilizers results in high soil solution P concentrations for early crop growth, but this solution P is rapidly adsorbed and fixed on the soil particle surfaces (Barrow and Debnath, 2014), causing less P supply to crops later in the growing season (Veneklaas et al., 2012). However, struvite dissolution is enhanced by organic acids released by crop roots (Ahmed et al., 2015; Talboys et al., 2016), and struvite can be considered as a slow-release P source that better matches crop P needs during the growing season (Ackerman et al., 2013) while being less prone to runoff and leaching losses (de-Bashan and Bashan, 2004; Ackerman et al., 2013). Therefore, struvite manufacturing is a key factor in mitigating eutrophication as it limits P inputs

from wastewater discharge in lakes and rivers (Le Corre et al., 2007). Furthermore, removing P in the form of struvite will reduce the costs associated with water treatment plants, as it will limit the production of sludge during the chemical precipitation of P.

Struvite has been proposed as a potential P source since 1858 (Cerrillo et al., 2014) and could be used to support plant growth in a sustained way (Li et al., 2019). Studies have demonstrated that struvite was better than or as efficient as conventional P fertilizers under different crops such as chickpea (*Cicer arietinum* L.) (Ghosh et al., 1996), ryegrass (*Lolium perenne* L.) (Johnston and Richards, 2003; Plaza et al., 2007), lettuce (*Lactuca sativa* L.) (González-Ponce et al., 2009; Ryu et al., 2012), corn (*Zea mays* L.), tomato (*Solanum lycopersicum*) and wheat (*Triticum* sp.) (Kern et al., 2008; Gell et al., 2011; Uysal et al., 2014). However, the majority of these studies were conducted under greenhouse conditions. To our knowledge, no published research exists assessing potato response to struvite in comparison to conventional P fertilizers.

Under Eastern Canada's cold and wet springs and low soil pH, it is important that crops are provided with readily-soluble starter P fertilizer to ensure a good start. Therefore, the objectives of this study were to evaluate the impact of partially substituting conventional P fertilizer (triple superphosphate [TSP]) with struvite on: (i) the agronomic performance of potato in the major growing areas of Eastern Canada; and (ii) plant- and soil-based P availability indices measured at multiple times during the growing season using potato petiole P concentrations, anion exchange membranes, and Mehlich-3 extracted P.

4.4 Materials and methods

4.4.1. Sites description, experimental design and phosphorus treatments

A total of 12 field trials were conducted during the 2016, 2017, and 2018 growing seasons at four sites across Eastern Canada: at Sainte-Catherine-de-la-Jacques-Cartier in Quebec (QC), and at Agriculture and Agri-Food Canada's Research and Development Centres in Charlottetown, Prince-Edward-Island (PEI), Fredericton, New-Brunswick (NB), and Kentville, Nova-Scotia (NS). The geographical coordinates of the different sites are presented in Table 4.1.

All trials were arranged in a randomized complete block design with four replicates. Each experimental unit consisted of six adjacent rows measuring 8 m in length, with a row spacing of 0.951 m. The potato seeds (Russet Burbank variety) were mechanically planted at a spacing of 0.381 m, and all sites were rainfed.

The struvite granules used in this study are commercially distributed under the trade name of Crystal Green® (5-28-0 + 10% Mg) (Cryst.) and were provided by Ostara Nutrient Recovery Technologies Inc. Cryst. is precipitated from wastewater using the WASSTRIP (Waste Activated Sludge Stripping To Recover Internal Phosphate) (Baur, 2009) and Pearl processes. The WASSTRIP process releases phosphorus and nitrogen from the wastewater stream. During the Pearl process, Mg is added to the nutrient-rich water, and a chemical reaction occurs in which P, N, and Mg bond together and slowly react to form small, white granules with a diameter of approximately 2.4 mm.

Each year, five P treatment rates (0, 26, 52, 79, 105 kg P ha⁻¹, which correspond to 0, 60, 120, 180, 240 kg P₂O⁵ ha⁻¹) of soluble triple superphosphate (TSP), and three P treatment rates having a mixture of TSP and struvite (25%, 50% and 75% Crystal Green®, to supply a total of 79 kg P ha⁻¹) were applied once prior to planting

(Table 4.2). The maximum recommended P rate for potatoes in NB and QC is 92 kg P ha⁻¹ (New Brunswick Department of Agriculture, Aquaculture and Fisheries, 2011) and 87 kg P ha⁻¹ (CRAAQ, 2010), respectively, and most of the sites in PEI fall within the medium to high P fertility status, which would require P recommendations from 59 to 87 kg P ha⁻¹ for Russet Burbank (PEI Department of Agriculture and Fisheries, 2017). Therefore, a P rate of 79 kg P ha⁻¹ would fall within the P sufficiency range for most of the soils cropped to potatoes, and thus struvite substitution was only made for this rate. To increase the likelihood of observing a response to P fertilizer, when possible, soil test reports were consulted in fall to select a field for the following spring, and a site with low P fertility status was favored. Nitrogen application timing varies among growers within the studied sites, with some growers splitting N fertilizer and others applying one single rate with the potato planter. The N (as ammonium nitrate) and potassium (K, as potassium chloride) fertilizers were applied at a rate of 200 kg ha⁻¹. Nitrogen supplied with struvite was not taken into account as it was considered to be slowly-released and thus negligible under a short growing season. All fertilizers were double-banded by hand in spring before planting and were buried 5 cm below the potato seed. No additional fertilizer was applied during the growing season. Planting was carried out in May and/or June, and potatoes were harvested from September to October, depending on the year and site (Table 4.1).

4.4.2. Preplant soil sampling and analyses

Prior to the establishment of the trials and fertilizer application, a composite soil sample of eight cores per block was taken at two depths (0–15 cm, 15–30 cm) using a 2-cm-diameter auger. Samples were air-dried, ground, and passed through a 2-mm sieve. Soil pH was measured using a 1:1 soil/solution ratio (10 g soil: 10 mL water) (Hendershot et al., 2008), and organic matter (g kg⁻¹ OM) was determined by dry

combustion on an Elementar analyzer (Vario Max Elementar Analyzer, Hanau, Germany). Cation exchange capacity (CEC) was estimated based on Mehlich-3 (Mehlich, 1984) extracted nutrients. Mehlich-3 extractable elements (P, K, Ca, Mg, Al, and Fe) were analyzed using plasma emission spectroscopy (ICP) (VARIAN 820-MS, Varian Inc. Scientific Instruments, Mulgrave, Victoria, Australia). Soil textural analysis was determined with the hydrometer method (Sheldrick and Wang, 1993).

4.4.3. Soil characteristics before the trial establishment and fertilizer application

Soils were loamy in texture, with sand and clay contents ranging from 42% to 82% and from 5% to 12%, respectively (Table 4.1). According to the Canadian classification system, the soil series encountered were: Oromocto (OMC125A: 63%), Riverbank (27%) and Oromocto (OMC##8A: 10%) in NB; Somerset (80%) and Woodville (20%) in NS in 2016 and Berwick in 2017; Charlottetown in PEI; and Morin (70%) and Mont-Rolland (30%) in QC. The Somerset, Woodville, Charlottetown and Morin soil series correspond to Haplorthods in the United States soil classification system; the Oromocto series (OMC125A) corresponds to Humaquepts, while the Riverbank and Mont-Rolland series correspond to Dystrochrepts, and the Oromocto (OMC##8A) series corresponds to Aquolls.

Any liming was carried out at any site to increase soil pH. Soil pH ranged from 4.8 to 6.2. In both years, 2016 and 2017, the Charlottetown site was the least acidic site, with a soil pH of 6.2. The other sites had soil pH ranging from 4.8 to 5.7. Most of the pH values were in the ideal pH range for potato crop cultivation (FAO, 2008). Sites located in Quebec were characterized by the lowest soil pH and the highest CEC and OM (Table 4.1). The studied sites had high ($\text{Al}_{\text{M3}} > 1600 \text{ mg kg}^{-1}$) or moderate (1100

$\text{mg kg}^{-1} < \text{Al}_{\text{M3}} < 1600 \text{ mg kg}^{-1}$) P retention capacity according to Tran et al. (1990).

Based on the agronomic and environmental thresholds, defined as the $(\text{P}/\text{Al})_{\text{M3}}$ ratio for the acidic coarse-textured soils of Quebec by Khiari et al. (2000) (8% and 15%, respectively) and of the Canadian Maritimes by Benjannet et al. (2018a;b) (10% and 14%, respectively), the studied sites belonged mostly to the medium P fertility class, and P environmental risk was low or moderate (Table 4.1).

4.4.4. Total P uptake and P recovery calculation

Before vine senescence, a destructive plant sampling was performed by harvesting four plants per plot to determine vine and tuber dry matter biomass and P concentration. After recording the fresh mass of the vines and tubers, a 500 g subsample was chopped for vines or sliced for tubers, weighed and dried at 60°C for 48 h to determine dry matter content. Dried vine and tuber samples were ground to pass through a 0.15 mm screen. Ground samples were dry-ashed, and the residue was brought up to 50 mL volume with 3.6 mol L⁻¹ HCl. Phosphorus concentrations were determined using a VARIAN 820-MS ICP. Total PU in each plot was calculated as the product of their respective dry matters and tissue P concentrations. Phosphorus uptake in vines and tubers was added together to obtain total PU. The apparent phosphorus recovery (APR) was then used to evaluate the plant PU in fertilized soils in comparison to the control (no P applied) as follows:

$$\text{APR (\%)} = \frac{\text{PU fertilized} - \text{PU control}}{\text{P rate}} \times 100 \quad (\text{Eq. 1})$$

4.4.5. Potato yields

Tubers were mechanically harvested on two 6-m-length internal rows in each plot to determine the total yields (TY) and marketable yields (MY). Tubers were

weighed, counted and graded into various size classes to determine TY and MY. Tuber size categories were based on long-type potatoes for French fry processing and included: Culls: < 38 mm; Canada No. 1 small: 38 to 51 mm; Canada No. 1: 51 to 89 mm; Canada No. 1 large: 89 to 114 mm; and Jumbo: > 114 mm. The MY included Canada No. 1 and Canada No. 1 large for NS, NB and QC, while it included Canada No. 1 small, Canada No. 1, and Canada No. 1 large for PEI. Tubers with external defects such as malformation, greening, and soft rot were discarded.

4.4.6. Soil-P availability indices

Anion exchange membranes (AEMs) were used to assess soluble P in situ during the growing season. This allowed the capture of temporal soluble P variations throughout the growing season, as opposed to punctual soil sampling. To facilitate their handling, the AEMs used in this study (type AR204-SZRA, Durpro, Montreal, Quebec, Canada) were cut into strips 6×5.3 cm, yielding a reactive area of 63.6 cm^2 per strip (2 times the strip surface). They were prepared as described by Ziadi et al. (1999): washed with distilled water, shaken for 30 min in a HCl solution (0.5 M) and thoroughly rinsed with distilled water. A solution of sodium chloride (1 M) was used to saturate the anion sites with Cl^- by shaking the strips for 2 hours. The prepared membranes were attached to fishing lines to facilitate their recovery from the soil and kept in distilled water until their installation in the field. Two membranes were inserted vertically into a slit opened with a shovel at a depth of 15 cm between two potato plants in an internal row of each plot, taking care to ensure good contact between membranes and soil. The first set of AEMs was inserted on the planting date. AEMs were subsequently retrieved and replaced every 3 weeks during the growing season. New AEMs were placed in close proximity to the removed AEMs. After being removed,

AEMs were washed with distilled water to remove attached soil particles, extracted in 1 mol L⁻¹ KCl by shaking the tubes for 2 hours on a lateral shaker at 120 oscillations min⁻¹ and then filtered through VWR 410 filters. The PO₄³⁻ (P_{AEMs}) was determined by colorimetry using a Lachat (QuickChem 8000 FIA+ Analyser, Lachat Instrument, Loveland, CO). The results were reported (in µg P 100 cm⁻² d⁻¹) by dividing the concentration by the area of the strip surface and by the period of soil exposure in days and then multiplying by 100 (Ziadi et al., 2011).

To further asses plant available P in the root zone during the growth season, a soil sample composed of six soil cores was taken from each plot at 0 to 15 cm depth on the row (potato hill) near the AEMs locations on each date where AEMs were recovered (a total of five dates). The P was extracted using the Mehlich-3 (P_{M3}) (Mehlich, 1984) method and analyzed using plasma emission spectroscopy (Perkin Elmer AES 4300DV ICP).

4.4.7. Potato petiole sampling and analyses

Petiole sampling for determination of petiole PO₄-P concentrations started 40–50 days after planting (DAP). This period corresponds to the transition from vegetative growth to the tuber initiation stage in the potato growth cycle (ZebARTH and Rosen, 2007). Petioles were sampled weekly on five dates until 80–90 DAP in each of the 3 years of the study. Petioles were taken from plants in the same row where the AEMs were inserted. For each plot, the fourth leaf from the top of the main stem was collected from 30 randomly-selected plants (ZebARTH et al., 2012). Leaflets were removed in the field, and petioles were placed in a cooler for transport to the laboratory. Petiole samples were oven dried at 50°C and ground to pass through a 2 mm screen. The petiole PO₄-P was extracted using 2% acetic acid solution and filtered through Whatman

Grade 5 filters. The petiole PO₄-P (P_{petiole}) concentration in the extract was determined colorimetrically using the Lachat as described above.

4.4.8. Statistical analyses

The statistical analyses were performed using the MIXED procedure of SAS (SAS Institute, 2010). Normality was tested using the Shapiro-Wilk test, and logarithmical transformation was carried out when needed. Yield data was pooled together across sites and year, and year was treated as a random factor. Consideration of year as a random effect allowed the assessment of potato response to different P sources and rates independently of interannual variations. Site, P treatments, and dates were treated as fixed effects. Treatment means were compared using the least mean squares method with the LS-Mean statement in SAS. The effect of the different sources and rates of P fertilization on petiole PO₄-P (P_{petiole}), AEMs PO₄-P(P_{AEMs}) and soil P_{M3} (P_{M3}) was analyzed using the repeated measures analysis of variance (ANOVA) with the MIXED procedure of SAS (SAS Institute, 2010). Pearson's correlation coefficients between P availability indices and MY and total PU were calculated using the CORR procedure in SAS.

4.5 Results

4.5.1. Weather conditions

Weather-related parameters for the studied sites were recorded at the nearest Environment Canada weather station, and mean temperatures ranged from 14°C to 16°C at all sites. Mean temperature over the growing season was slightly warmer than the historical data (14°C to 15°C). The highest monthly air temperatures were recorded in July and ranged between 18.1°C and 21.4°C, which is close to the 30-year climate normals (18.1°C to 19.5°C). Total precipitation during the growing season ranged

between 474.9 and 617.4 mm in 2016, 437.3 and 737.6 mm in 2017, and 608.7 and 618.2 mm in 2018. Precipitation totals were 8.9 to 112.1 mm lower than the 30-year climate normal in both 2016 and 2017, except in QC in 2017, where total rainfall was 74.6 mm higher than the 30-year average. The 2018 growing season was wetter than the 30-year climate normal average by 69 mm in NB, 109 mm in NS, and 35 mm in PEI. Unlike 2017, precipitations at the QC site were 49 mm lower than the normal average.

4.5.2. Total and marketable tuber yields

There was a significant effect of P treatments, sites, and their interactions on potato yield (Table 4.3). Across all sites, the TY varied from 18.5 to 43.3 Mg ha⁻¹, ranging from 28.8 to 41.4 Mg ha⁻¹ in NB, from 34.7 to 41.7 Mg ha⁻¹ in NS, and from 29.0 to 43.3 Mg ha⁻¹ in QC. The lowest TY values were obtained at the PEI site, ranging from 18.5 to 34.5 Mg ha⁻¹ (Fig. 4.1a). When compared with the control treatment, TY tended to increase significantly with increasing P levels of soluble TSP (Fig. 1a). The 0TSP and 26TSP treatments were associated with statistically lower TY than the other treatments at all sites, except in NS, where 26TSP was comparable with other treatments (Fig. 4.1a).

At the NB, NS and QC sites, applying a mixture of conventional fertilizer TSP and struvite at 79 kg P ha⁻¹ rate resulted in statistically comparable yields to those obtained when adding the same P rate as TSP alone (79TSP) (Fig. 4.1.a). The TY obtained with treatments 79Cryst25, 79Cryst50 and 79Cryst75 represented, respectively, 95%, 98%, and 98% of the TY obtained with treatment 79TSP in NB, 95%, 91%, and 95% in NS, and 101%, 102%, and 95% in QC. In PEI, the 79TSP

treatment was associated with higher yield than the 79Cryst50 and 79Cryst75 treatments.

Similarly to TY, a significant effect of treatments, sites and their interaction was observed on MY (Table 4.3). Across all sites, MY ranged from 14.1 to 34.1 Mg ha⁻¹ (Fig. 4.1b). In PEI, the treatments with only 25% TSP and 75% struvite and 50% TSP with 50% struvite were associated with lower MY than 100% TSP applied at equivalent rate (79TSP) (Fig. 4.1.b).

4.5.3. Phosphorus uptake and apparent P recovery

There were significant effects of P treatments, sites, and their interactions on PU (Table 4.3). The PU values ranged from 9.3 to 22.8 kg P ha⁻¹ and tended to increase with the P rate (Fig. 4.2a). The PU values across the three growing seasons ranged from 13.6 to 17.6 kg P ha⁻¹ in NB, from 16.1 to 22.8 kg P ha⁻¹ in NS, and from 11.8 and 17.7 kg P ha⁻¹ in QC (Fig. 4.2a). The 79TSP treatment produced statistically-comparable PU amounts when TSP was mixed with struvite at 75%, 50% or 25% at all sites except in PEI, where 79TSP was associated with lower values than 79Cryst75 (Fig. 4.2a).

The percentage of added P removed by plants as estimated by APR (Eq. 1) has shown that the interaction between P treatment and site was significant (Table 4.3). The APR value in whole plants (vines and tubers) ranged from 4.2% to 15.3% in PEI, from 4.6% to 9.3% in QC, from 0.8% to 5.7% in NB, and from -1.1% to 6.1% in NS (Fig. 4.2.b). In PEI, apparent P recovery was 2.6 and 1.7 fold higher with TSP than 79Cryst50 and 79Cryst75, respectively.

4.5.4. Soluble P content as measured by AEMs

The ANOVA of soil phosphate dynamics as measured by AEMs showed that there was a significant effect of P treatments, sites, sampling dates, as well as the

interaction between sites and sampling dates and P treatments and sampling dates (Table 4.3).

The P_{AEMs} concentrations averaged between 0.12 and 1.44 $\mu\text{g } 100 \text{ cm}^{-2} \text{ day}^{-1}$ across the growing season (Fig. 4.3a). Though the highest nominal P_{AEMs} value at the first sampling date was obtained with the treatment containing 50% struvite (79Cryst50), it was statistically comparable with the values obtained with the 79TSP and 105TSP treatments (Fig. 4.3a). Values declined with time as P was taken up by the potato crop, and at sampling dates 2 and 3, P_{AEMs} values were comparable among treatments. A different trend was observed by the end of the growing season (last sampling date), with a remarkable increase in P_{AEMs} concentrations associated with the 79Cryst50 and 79Cryst75 treatments (Fig. 4.3a).

The P_{AEMs} concentrations varied widely between sites, specifically at the first sampling date, with a clear trend towards lower values in PEI (Fig. 4.3b).

4.5.5. Available P content as measured by Mehlich-3 extraction

Phosphorus availability as assessed by soil P_{M3} content was affected by P treatments, sites, and sampling dates, and by the interaction between sites and sampling dates (Table 4.3). As the Mehlich-3 solution extracts both soluble and reversibly-adsorbed P, averaged values across treatments significantly increased with the increasing P rate, passing from 139 mg P kg^{-1} with 0TSP to 183 mg P kg^{-1} with 105TSP (Fig. 4.4a). Though the highest P_{M3} concentration was obtained at 105 kg P ha^{-1} , it was statistically comparable to that obtained at 79 kg P ha^{-1} (Fig. 4.4a). When sites were compared among themselves per sampling dates, the lowest P_{M3} concentrations were associated with the PEI site, and the highest ones were recorded in QC, followed by NB (Fig. 4.4b).

4.5.6. Petiole phosphorus

The P fertilizer treatments had no significant effect on the petiole P (P_{petiole}) concentration, and comparable values were obtained among the treatments. The sampling date was significant, as well as the interaction between sampling dates and sites and between treatments and sampling dates (Table 4.3; Fig. 4.5). The average P_{petiole} concentrations ranged from 1.0 to 1.6 g P kg⁻¹ in NB, from 2.1 to 2.9 g P kg⁻¹ in NS, from 0.9 to 1.4 g P kg⁻¹ in PEI, and from 0.3 to 0.9 g P kg⁻¹ in the QC site (Fig. 4.5A). Slicing the interaction between sites and sampling dates revealed that higher values tended to be associated with the NS site and lower values with the QC site, whereas NB and PEI tended to be associated with intermediate values (Fig. 4.5A). The interaction between sampling dates and treatments has shown that treatments were comparable across sampling dates except at the first sampling date, where the control was associated with lower values even though it was comparable with the 26TSP treatment (Fig. 4.5B).

4.5.7. Correlation between P availability indices with marketable yield and P uptake

When soil and plant P availability indices as measured with P_{petiole} , P_{AEms} and P_{M3} were correlated with MY and PU, considering overall treatment means across sites and years, it was observed that the best indices resulted from P extracted with Mehlich-3 as it was highly correlated with MY ($r = 0.96$) and total PU ($r = 0.97$) (Table 4.4). Phosphorus extracted with Mehlich-3 was also correlated with P_{petiole} ($r = 0.72$), but P_{petiole} and P_{AEms} were not significantly correlated with MY or PU.

4.6. Discussion

4.6.1. Potato yield as affected by P rates and sources

Total yields at all sites were higher than the potato tuber yields (17.0 to 34 Mg ha⁻¹) obtained in Eastern Canada by ZebARTH et al. (2008) for different potato varieties. The TY obtained in NB and in QC was above that obtained in NB by Snowdon et al. (2013) (30.2 Mg ha⁻¹) and in QC by Cambouris et al. (2016) (16.3 to 38.3 Mg ha⁻¹) for Russet Burbank variety. In PEI, potato yield was in the range of those reported by Nyiraneza et al. (2015) for Shepody variety (20 to 27 Mg ha⁻¹). Overall, TY means were 39.3 Mg ha⁻¹ in the NS and QC sites, 37.7 Mg ha⁻¹ in NB, and 28.2 Mg ha⁻¹ in PEI, which were all above the world 10 yr average yield of 17 Mg ha⁻¹ estimated by FAO (FAO, 2008). A response to P fertilization was observed at all sites.

All sites fell under the low or medium P fertility class based on their P saturation index (PSI_I, P/Al ratio) being below the agronomic critical levels of 8% and 10% established, respectively, for the soils of Quebec (Khiari et al., 2000) and the Canadian Maritimes (Benjannet et al., 2018b) (Table 4.1). According to these authors, when the site has a PSI below the agronomic threshold level, a response to P input is expected. Conversely, when P index is above the critical value, P fertilizer inputs should be limited to crop removal to minimize P losses to the environment. Crop response to P fertilization is expected to increase significantly with additional P inputs when soil P fertility levels are below the established thresholds (Korndörfer et al., 2001; Rosen et al., 2014; Zamuner et al., 2017). Thus, it was not surprising to obtain an increase in potato TY with increasing P rate, which is aligned with previous findings (Bundy et al., 2005; Nyiraneza et al., 2017; Benjannet et al., 2018b). However, the response to P inputs was different across sites, with the control treatment representing 73%, 83%, 67%, and 53% in NB, NS, QC, and PEI, respectively. The Quebec and PEI sites

responded more to P inputs than the NB and NS sites. A more gradual yield increase with P rate was observed in PEI than at other sites (Fig. 4.1a,b). A similar but less pronounced trend was observed in Quebec. However, yield response to P fertilization is not only influenced by soil fertility as indicated by a soil test, but also by many other factors including soil properties, differences in early season rainfall events that impact nitrate leaching, cultivars, seed quality, temperature, rainfall, and crop and soil management practices (Moorehead et al., 1998; Bélanger et al., 2000, 2002; Zvomuya and Rosen, 2001; Nyiraneza et al., 2017), which may explain the variability in TY between sites.

The partial substitution of the highly-soluble P fertilizer as TSP with struvite up to a proportion of 75% did not negatively impact total potato yields at other sites, except for the PEI site, where the use of struvite fertilizer at higher proportion significantly depressed potato TY and MY when compared with 79TSP. Comparable MY results at the NB and NS sites between TSP and struvite mixed with TSP are aligned with previous studies, as presented in the following section. In a greenhouse experiment, Talboys et al. (2016) reported that, when applied at a same rate, struvite produced very similar rates of spring wheat grain yield per plant to those obtained with use of TSP. Gell et al. (2011) tested the fertilizing effect of struvite recovered from urine and black water on corn under field conditions on an acidic ($\text{pH} = 4.5$) sandy loam soil. They concluded that there was no significant difference in corn dry matter yield when compared with TSP applied at the same rate of 87 kg P ha^{-1} . Similar findings were reported by Liu et al. (2011), who tested struvite recovered from swine wastewater on maize in a pot experiment, and by Cabeza et al. (2009) on winter barley (*Hordeum vulgare L.*) grain yields on a P-deficient loamy soil. Crops performed equally under other studies when mineral P fertilizers other than TSP (i.e. monophosphate,

monocalcium phosphate) were compared with struvite (Johnston and Richards, 2003; Plaza et al., 2007).

Struvite at a higher proportion (75%) was associated with lower TY and MY than the equivalent rate as TSP in PEI. A possible partial explanation is the relatively high soil pH in 2016 and 2017 (Table 4.1). Previous studies have demonstrated that the fertility effect of struvite is dependent on soil pH, which affects its solubility and sorption. In a solubility assay, Talboys et al. (2016) tested struvite release under pH conditions varying from 5.5 to 8.0 and demonstrated that the initial struvite P dissolution rate was very negatively correlated to increasing initial pH. Hence, as low pH conditions improve struvite dissolution, this makes it more available and leads to increased P use efficiency (Massey et al., 2009; Gell et al., 2011). However, the effects of soil pH on P release from struvite are controversial in the literature. Other studies on P efficiency from struvite in acidic or neutral pH soils have found struvite to be as effective as conventional P fertilizers (Plaza et al., 2007; Liu et al., 2011). Studies on alkaline soil have produced mixed results. Massey et al. (2009) tested the effectiveness of struvite on a limed soil ($\text{pH} = 7.6$) and found that struvite increased the dry matter production of spring wheat (*Triticum aestivum* L.). The authors concluded that struvite could be used as a P fertilizer on alkaline soils. Ackerman et al. (2013) found that struvite resulted in lower canola yields when compared with conventional P fertilizers in an alkaline soil ($\text{pH} = 7.7$). Another partial explanation for the lower yield associated with the treatments having higher proportion of struvite in PEI may be that P rate at 79 kg P ha^{-1} was under the optimal rate to meet potato P requirements. Based on local P recommendations based on soil P test, PEI would have recommended P rates from 87 up to $174.5 \text{ kg P ha}^{-1}$, depending on soil P levels (PEI Department of Agriculture and Fisheries, 2017). Based on P/Al ratio, Quebec would have recommended a P rate

ranging from 52.4 to 65.5 kg P ha⁻¹ (CRAAQ, 2010), while NB would have recommended a P rate ranging from 65.5 kg P ha⁻¹ to 91.6 kg P ha⁻¹ (New Brunswick Department of Agriculture, Aquaculture and Fisheries, 2011). However, initial P levels and P saturation index were lower and comparable at both PEI and NS sites than at QC and NB, and yet struvite at higher proportion performed equally as well as TSP, and MY was comparable among treatments at NS site. Another factor that might have negatively influenced the efficiency of P released from struvite at higher proportion in PEI could be attributed to lower soil temperature compared to other sites (Fig. 4.6). It was reported that increased temperature was associated with increased yield, root growth and P uptake (McKay and Barber, 1984). Therefore, the optimal P rate may need to be enhanced with struvite P applications, or the proportion of struvite in the mixture should be reduced to compensate for its slow P release under P-limiting and cold conditions. As tuber set can be affected by P fertilization (Rosen and Bierman, 2008), we assessed whether struvite at higher proportions was associated with lower tuber set and observed comparable values between 79TSP treatment with 79Cryst75 at all sites except at the PEI site, where the latter treatment was associated with 8% lower marketable tuber count (data not reported). Overall, yield reflected PU, and the correlation between two variables was high.

4.6.2. Total P uptake as affected by P rates and P sources

As for TY, PU values tended to be lower with the 0TSP and 26TSP treatments than with other treatments at all sites. The increase in PU with increasing rates of P fertilizer has been reported in previous studies (Rosen and Bierman, 2008; Mohr and Tomasiewicz, 2011). The PU value range in this study was comparable with that of previous findings. Average PU ranging from 13 to 20 kg P ha⁻¹ with increasing P rates up to 79 kg P ha⁻¹ were reported in a two-site study conducted in NS on Superior variety

(Nyiraneza et al., 2017). In Western Canada, a 4-year study on Russet Burbank resulted in PU values between 18 and 22 kg P ha⁻¹, with increasing P rates up to 43 kg P ha⁻¹ (Mohr and Tomasiewicz, 2011). Our values were lower than those reported by Rosen and Bierman (2008) in Minnesota, where PU ranged from 37.7 to 49.6 kg P ha⁻¹ for Russet Burbank with increasing P rates up to 74 kg P ha⁻¹. The higher PU in the latter study are proportional to higher yield (from 68.9 to 74.1 Mg ha⁻¹), more so than in the current study. Similarly, the PEI site had the lowest PU values (9.3 to 17.2 kg P ha⁻¹), which could be related to the lower TY obtained. Phosphorus uptake between the 79TSP treatments versus the same rate with different proportions of struvite and TSP were mostly comparable at all sites except in PEI, where the 79Crys75 treatment was associated with lower value than the 79TSP treatment. Talboys et al. (2016) also tested varying ratios of struvite with diammonium phosphate (DAP) in a 36-day pot experiment on spring wheat and buckwheat (*Fagopyrum aesculentum*) and reported comparable PU values between the mixture and DAP alone.

4.6.3. Apparent P recovery (APR) as affected by P rates and P sources

The proportion of total applied P input to total PU by the whole crop is expressed as APR. It is expected that APR would decline with increasing P rate under sites responsive to P application, and this trend was only observed in the PEI and QC sites. Overall, very low APR values were observed in NB and NS in comparison to the PEI and QC sites. When a low response to P fertilizer is observed, PU values obtained with the check treatment are comparable to PU obtained with fertilized plots, which results in low APR values (Fig. 4.2a vs. Fig. 4.2b).

Under acidic soil rich in aluminum and iron oxides, P is fixed within a relatively short period, even when P fertilizer is applied at high rates, which could explain the overall low APR values in this study. Khiari and Parent (2005) found that within

6 weeks of incubation, over 50% of the added P was sequestered as Al-P and Fe-P under acidic soils cropped to potatoes. The 79TSP treatment had comparable APR values in comparison to the 79Crys25, 79Cryst50 and 79Cryst75 treatments (Fig. 4.2.b). This contradicts previous reports by Cabeza et al. (2011) and Vogel et al. (2015) under a greenhouse study where the APR of different P recycling products was significantly lower than that of TSP. Our findings corroborate those of Talboys et al. (2016), where comparable APR values were reported between struvite and DAP. To better understand P dynamics from different sources over the growing season, different soil and plant P availability indices were tested.

4.6.4. Plant and soil P availability index responses to P rates and sources

The effects of treatments on plant and soil availability indices as measured with AEMs, Mehlich-3 and petiole P concentrations tended to mirror the same trend observed on yield and PU, with lower values associated with the 0TSP and 26TSP treatments (Fig. 4.3a; 4.4a; 4.5b). Values of P_{AEMs} tended to decline with the growing season, and there was a trend towards increased values at the end of the growing season with the 79Cryst50, and 79Cryst75 treatments (Fig. 4.3a). Talboys et al. (2016) demonstrated that when struvite is applied in soil in combination with conventional P fertilizer, the P released in the soil from struvite is initially lower. Meanwhile, the readily-soluble fertilizer dissolves quickly in the soil solution, providing early available P for uptake by the crop roots. The struvite dissolution subsequently increases once the ortho-P concentration in the soil solution decreases due to soil P sorption, leaching and/or plant uptake. Additionally, struvite solubility increases with time as the organic acid anions secreted by plant roots increase. This could explain the increase in P_{AEMs} concentrations related to the 79Cryst50 and 79Cryst75 treatments at the last sampling time.

Site response to P availability indices was not consistent and changed depending on the index used, implying that these indices do not measure the same P pool. For instance, higher P_{AEMS} values were observed at the PEI and QC sites, while higher P_{M3} values were observed at the NB and QC sites. Conversely, $P_{petiole}$ concentrations were lower in the PEI and QC sites. The correlation coefficients between P availability indices with PU and MY demonstrated that P extracted with Mehlich-3 solution was the best index. Other P availability indices were not related to MY or PU. Compared to P extracted with Mehlich-3, soluble P extracted with AEMs is highly variable due to low soil area occupied by the ion exchange membranes in comparison to the soil volume taken before Mehlich-3 extraction. Similarly, petiole nutrient concentrations can be affected by many parameters other than P input, such as time of sampling, position of the petiole, time of the day and environmental conditions prior to sampling, as was reported by Vitosh and Silva (1996) and Rosen and Bierman (2008). Critical P petiole to reach maximum yield can also differ among studies and sites. For instance, except for the NS site, petiole P concentration values in this study were below the sufficiency range suggested for potato in Quebec ($3\text{--}5 \text{ g P kg}^{-1}$, CRAAQ 2010) and in Minnesota ($2.2\text{--}4.0 \text{ g P kg}^{-1}$, Rosen et al., 2014). Conversely, petiole P concentration values at all sites except in QC were higher or very close to the critical threshold of 1.0 g P kg^{-1} defined by Westermann and Kleinkopf (1985).

Even if all sites received equal P inputs, as expected, inherent site properties such as soil pH, soil organic matter (OM) and initial P contents could affect potato performance and P availability index responses to P inputs. For instance, soil OM was higher in the NB and QC sites. The Quebec site had higher inherent P contents and lower soil pH. Higher soil OM and higher initial P contents in the QC and NB sites

could explain the higher values of P_{M3} . Higher soil OM contents were reported to reduce P fixation (Yu et al. 2013).

In sum, there was a response to increasing P inputs applied as TSP at all sites. At three out of four sites, a proportion of only 25% of TSP supplemented with struvite was enough to supply potato P requirement, as did a 100% TSP treatment applied at a same rate. In PEI, a high proportion of struvite in the mixture compared to 100% TSP depressed yield and was associated with lower PU, probably because the P rate was not enough to satisfy the potato P requirement or due to lower soil temperature than at the other sites. Our results therefore support the hypothesis that P efficiency from struvite can interact with inherent soil properties (Ackerman et al., 2013) such as soil texture, pH, moisture, and plant P requirements (Li et al., 2019). Even if it has been demonstrated that current P recommendations could be reduced without affecting potato yield (Benjannet et al. 2018b), compared to conventional P fertilizer, struvite at high proportion ($> 50\%$) could potentially depress yield if applied at rate lower than recommended in low P soils. Available phosphorus extracted with Mehlich-3 was the best P availability index and was highly correlated with PU and MY in comparison with $P_{petiole}$ and P_{AEMs} .

4.7 Conclusion

Results obtained from this study indicate that the substitution of conventional, soluble P fertilizer with up to 75% struvite P from Cryst. can be as effective in increasing potato yields as TSP alone. However, under cold conditions and/or P-deficient soils, struvite at higher proportion ($> 50\%$), if applied at lower rate than recommended, could be associated with lower yield than pure conventional P fertilizer. In this case, optimal P to meet crop P requirements may need to be slightly increased to compensate for slow P release from struvite. Further field studies should investigate

the cost associated with mixing TSP with struvite. Overall, this study is a contribution to the few published field trials on the efficacy of struvite as an environmentally-sound P source and adds knowledge on alternative P inputs to sustain high-P-demanding crops such as potato.

4.8. Acknowledgements

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Table 4.1. Soil properties, sites characterizations and potato seeding and harvest time at studied sites in the New Brunswick (NB), Nova Scotia (NS), Prince Edward Island (PEI) and Quebec (QC) experimental sites.

| Year | 2016 | | | | 2017 | | | | 2018 | | | |
|---|---------|------------|------------|-----------------|------------|------------|------------|------------|---------|------------|-----------|-----------------|
| Sites | NB | NS | PEI | QC | NB | NS | PEI | QC | NB | NS | PEI | QC |
| pH | 5.5 | 5.7 | 6.2 | 5.1 | 5.7 | 5.6 | 6.2 | 5.2 | - | 5.5 | 4.8 | 5.3 |
| CEC (Meq 100 g ⁻¹) | 8 | 9 | 7.5 | 11.8 | 11.3 | 11.3 | 6.3 | 15.5 | - | 7.8 | 5.5 | 9.3 |
| Organic matter (%) | 3.1 | 2.7 | 2.9 | 5.2 | 3.6 | 3.3 | 3 | 5.9 | - | 3.9 | 3 | 5.7 |
| Particle size distribution (%) | | | | | | | | | | | | |
| Sand | 47 | 59 | 59 | 79 | 42 | 62 | 54 | 69 | - | 62 | 49 | 82 |
| Clay | 8 | 12 | 7 | 4 | 8 | 11 | 11 | 5 | - | 8 | 11 | 5 |
| Silt | 45 | 29 | 34 | 17 | 49 | 28 | 35 | 26 | - | 30 | 40 | 13 |
| Textural class§ | Loamy | Sandy loam | Sandy loam | Loamy fine sand | Silty loam | Sandy loam | Sandy loam | Sandy loam | - | Sandy loam | Loam | Loamy fine sand |
| Mehlich-3 extractable nutrients (mg kg⁻¹) | | | | | | | | | | | | |
| P | 102 | 73 | 149 | 247 | 155 | 82 | 79 | 467 | - | 66 | 53 | 122 |
| Al | 1703 | 1408 | 1484 | 2080 | 1480 | 1463 | 1186 | 2061 | - | 1745 | 1392 | 2058 |
| Fe | 221 | 350 | 219 | 305 | 259 | 221 | 237 | 238 | - | 157 | 232 | 159 |
| Phosphorus Saturation Indices | | | | | | | | | | | | |
| PSI (P/Al, %) | 6 | 5 | 10 | 12 | 10 | 6 | 7 | 7 | - | 4 | 4 | 6 |
| P retention capacity† | High | Moderate | Moderate | High | Moderate | Moderate | Moderate | High | - | High | Moderate | High |
| Environmental risk class‡ | Low | Low | Moderate | Moderate | Moderate | Low | Moderate | Low | - | Low | Very low | Low |
| Fertility class¶ | Medium | Medium | Medium | Medium | Medium | Medium | Medium | Low | - | Low | Low | Low |
| Coordinates | | | | | | | | | | | | |
| Latitude | 45° N | 45° 07' N | 46° 20' N | 46° 50' N | 45° N | 45° 03' N | 46° 21' N | 46° 50' N | 45° N | 45° 03' N | 46° 20' N | 46° 49' N |
| Longitude | -66° W | 64° 29' W | 63° 10' W | 71° 38' W | -66° W | 64° 29' W | 63° 09' W | 71° 39' W | -66° W | 64° 29' W | 63° 10' W | 71° 40' W |
| Planting and harvesting time | | | | | | | | | | | | |
| Planting date | 26 May | 27 May | 26 May | 24 May | 31 May | 28 May | 30 May | 5 June | 02 May | 08 May | 30 May | 23 May |
| Harvesting date | 13 Oct. | 12 Oct. | 12 Oct. | 04 Oct. | 27 Sept. | 29 May | 16 Oct. | 19 Oct. | 05 Oct. | 10 Oct. | 18 Nov. | 15 Oct. |

CEC: cation exchange capacity; PSI: phosphorus saturation index. §Textural class according to the USDA Natural Resources Conservation Service Soils. Available at: https://www.nrcs.usda.gov/wps/portal/nrcs/detail/soils/survey/?cid=nrcs142p2_054167

† classification according to Tran et al. (1990)

‡ classification according to Khiari et al (2000) and Benjannet et al. (2018). ¶ classification according to Khiari et al (2000) and Benjannet et al. (2018b).

Table 4.2. Phosphorus fertilizer rates and sources.

| Treatment ID | Total P rate (kg ha ⁻¹) | P Source | |
|--------------|--|------------------|---------------------------------------|
| | | TSP (0-46-0) (%) | Struvite (Crystal Green® (5-28-0) (%) |
| 0TSP | 0 | 0 | 0 |
| 26TSP | 26 | 100 | 0 |
| 52TSP | 52 | 100 | 0 |
| 79TSP | 79 | 100 | 0 |
| 105TSP | 105 | 100 | 0 |
| 79Cryst25 | 79 | 75 | 25 |
| 79Cryst50 | 79 | 50 | 50 |
| 79Cryst75 | 79 | 25 | 75 |

TSP: Triple superphosphate

Table 4.3. Analysis of variance of the measured parameters during the 3-year experiment

| P values | Total yield | Marketable yield | P uptake | APR | P_{AEMS} | P_{M3} | P_{petiole} |
|------------------|--------------------|-------------------------|-----------------|------------|-------------------------|-----------------------|----------------------------|
| S | ** | ** | * | * | *** | *** | *** |
| P | *** | *** | *** | n/s | *** | *** | n/s |
| S × P | * | ** | * | * | n/s | n/s | n/s |
| D | n/a | n/a | n/a | n/a | *** | *** | *** |
| S × D | n/a | n/a | n/a | n/a | *** | *** | *** |
| P × D | n/a | n/a | n/a | n/a | * | n/s | * |
| S × P × D | n/a | n/a | n/a | n/a | n/s | n/s | n/s |

S: sites; P: applied P treatment; D: sampling dates.

$$APR \text{ (Apparent P recovery, %)} = \frac{PU_{fertilized} - PU_{control}}{P_{rate}} \times 100 \quad (Eq. 1)$$

P_{AEMS}, P desorbed from anion exchange membranes; P_{M3}, soil P extracted with Mehlich-3 solution; P_{petiole}, petiole P concentration.

n/s: not significant (P > 0.05)

*, **, and ***: significant at the 0.05, 0.01, and 0.001 level, respectively.

n/a: not applicable.

Table 4.4. Pearson correlation coefficients between P availability indices with marketable yield (MY) and total P uptake (PU) using overall treatments means across sites and years (N = 8)

| | MY | PU | P_{M3} | P_{petiole} | P_{AEMS} |
|----------------------------|-----------|-----------|-----------------------|----------------------------|-------------------------|
| MY | - | | | | |
| PU | 0.96*** | - | | | |
| P_{M3} | 0.97*** | 0.97*** | - | | |
| P_{petiole} | 0.62 N/S | 0.69 N/S | 0.72* | - | |
| P_{AEMS} | 0.52 N/S | 0.64 N/S | 0.66 | 0.48 N/S | - |
| | | | N/S | | |

N/S, *, ***: not significant and significant at 0.05 and 0.001 probability level, respectively.

P_{AEMS}, P desorbed from anion exchange membranes; P_{M3}, soil P extracted with Mehlich-3 solution; P_{petiole}, petiole P concentration.

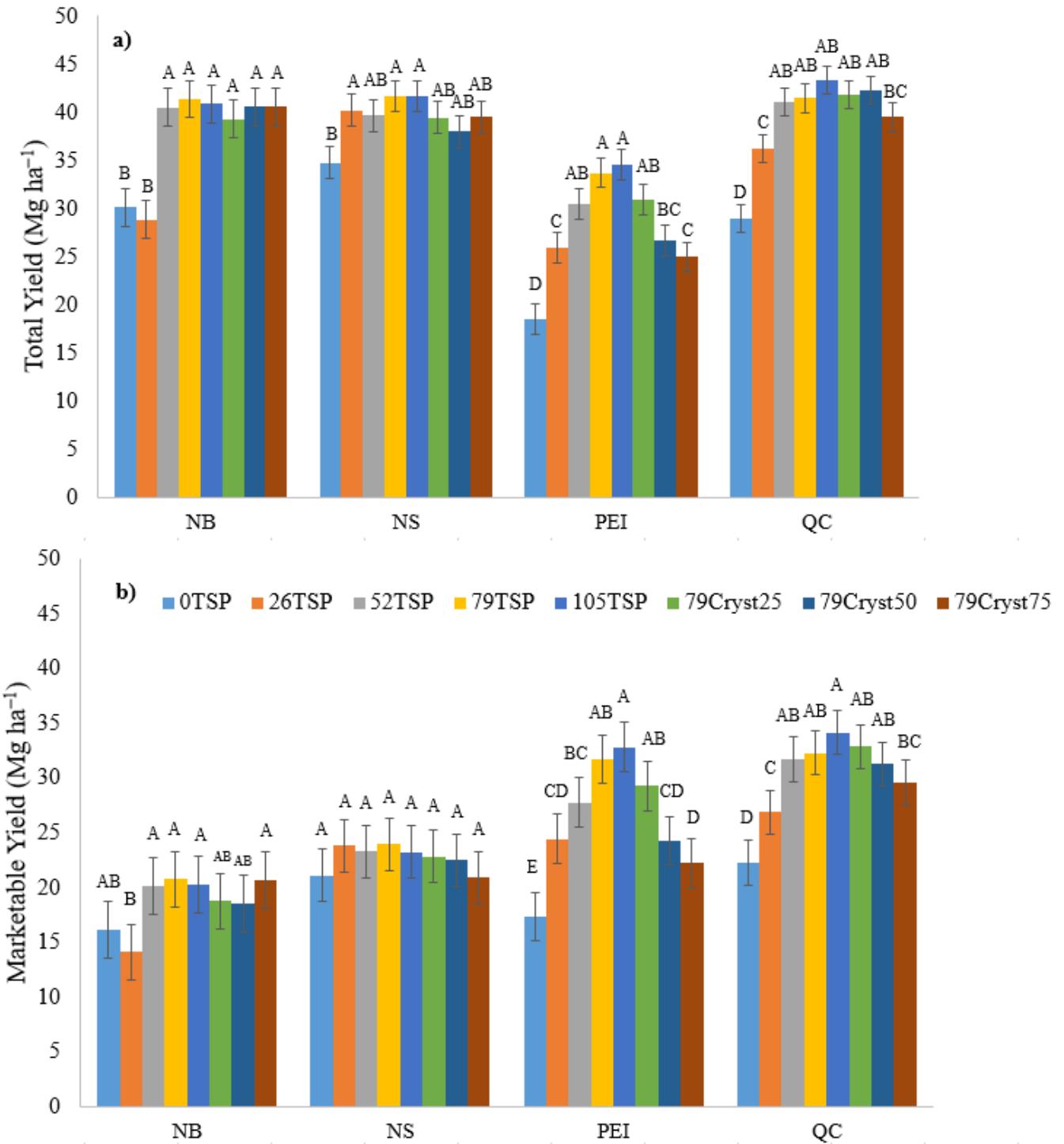


Figure 4.1. The interaction effect between P treatments and sites on the mean total (a) and marketable (b) potato yields at the New Brunswick (NB), Nova Scotia (NS), Prince Edward Island (PEI) and Quebec (QC) sites. Means followed by different letters within a group are statistically different at 0.05 probability level. Cryst25, Cryst50, Cryst75; struvite, trade name Crystal Green mixed with triple superphosphate (TSP) at 25%, 50% and 75% respectively.

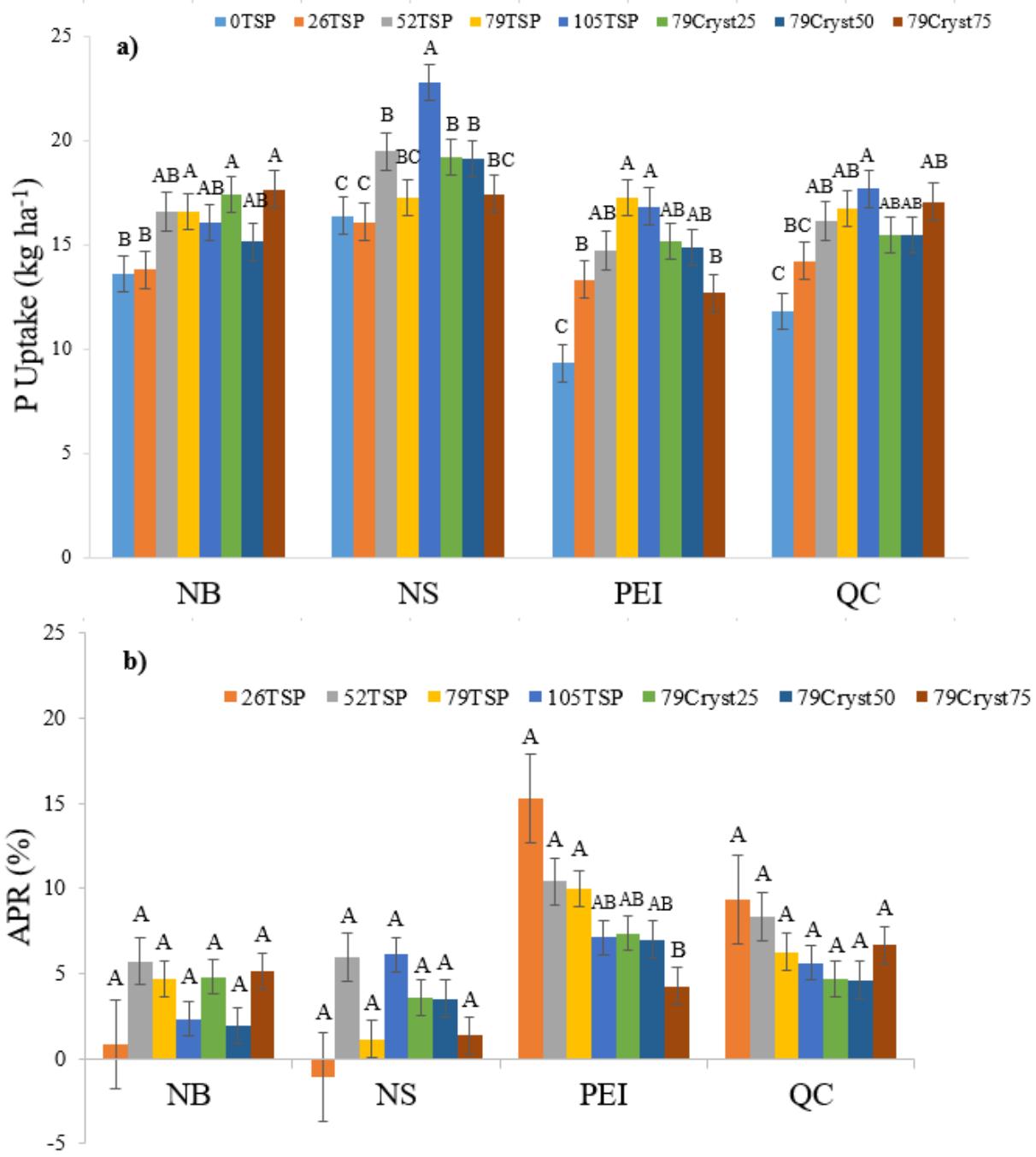


Figure 4.2. The interaction effect between sites and P treatments on (a) P uptake and (b) apparent P recovery (APR) at the New Brunswick (NB), Nova Scotia (NS), Prince Edward Island (PEI) and Quebec (QC) sites. Means followed by different letters within a group are statistically different at 0.05 probability level. Cryst25, Cryst50, Cryst75; struvite, trade name Crystal Green mixed with triple superphosphate (TSP) at 25%, 50% and 75% respectively.

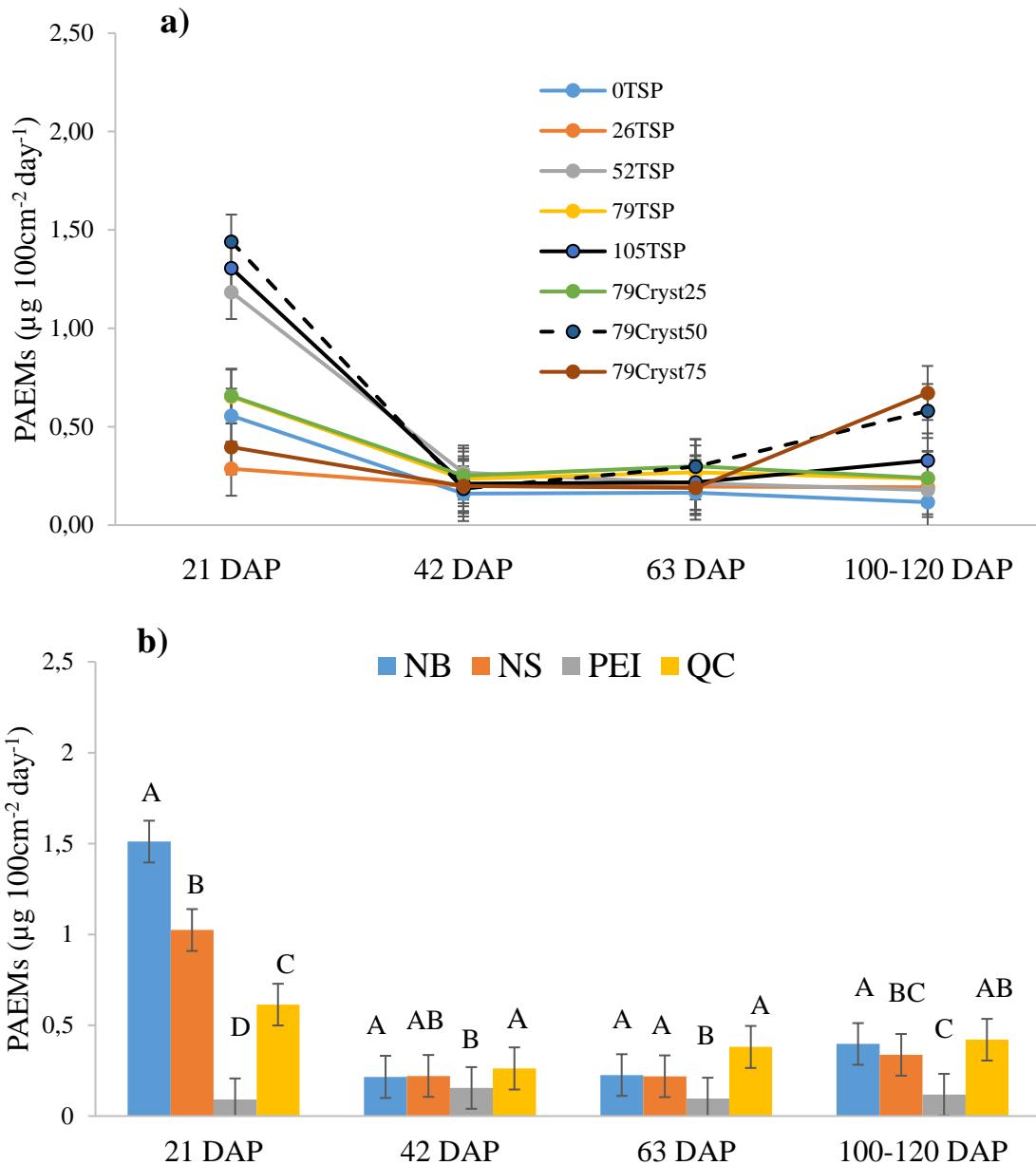


Figure 4.3. The interaction effect between sampling dates and (a) P treatments and (b) sites on the soluble P content measured with anion exchange membranes (PAEMs) at the New Brunswick (NB), Nova Scotia (NS), Prince Edward Island (PEI) and Quebec (QC) sites. Values followed by different letters within a group are statistically different at 0.05 probability level. Cryst25, Cryst50, Cryst75; struvite, trade name Crystal Green mixed with triple superphosphate (TSP) at 25%, 50% and 75% respectively. DAP, days after planting.

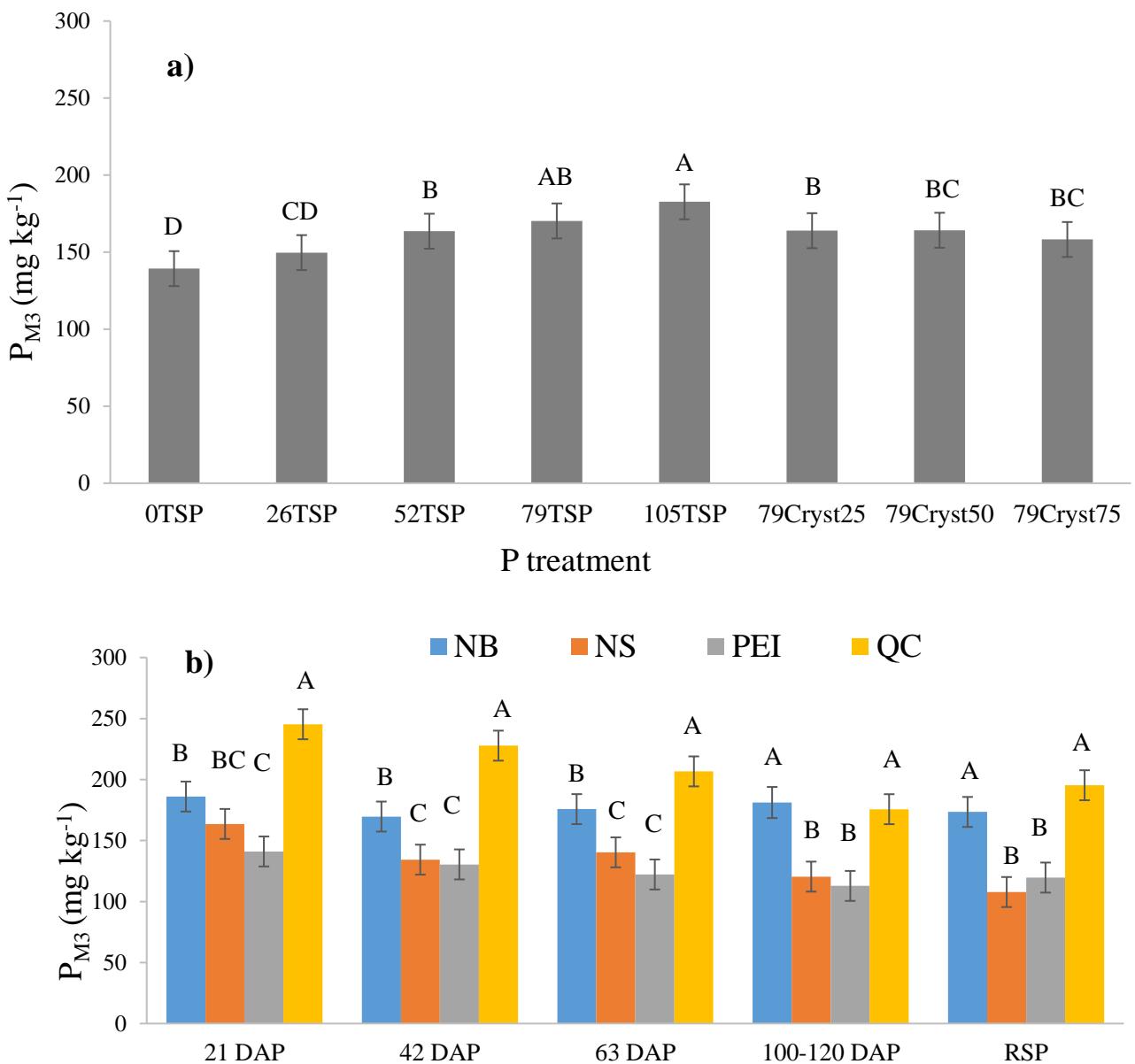


Figure 4.4. The effect of (a) P treatments and of (b) the interaction between sites and sampling dates on the available P content measured as soil P extracted with Mehlich-3 solution (P_{M3}) at the New Brunswick (NB), Nova Scotia (NS), Prince Edward Island (PEI) and Quebec (QC) sites. Values followed by different letters within a group are statistically different at 0.05 probability level. Cryst25, Cryst50, Cryst75, struvite, trade name Crystal Green mixed with triple superphosphate (TSP) at 25%, 50% and 75% respectively. DAP, days after planting; RSP, residual soil P after harvest.

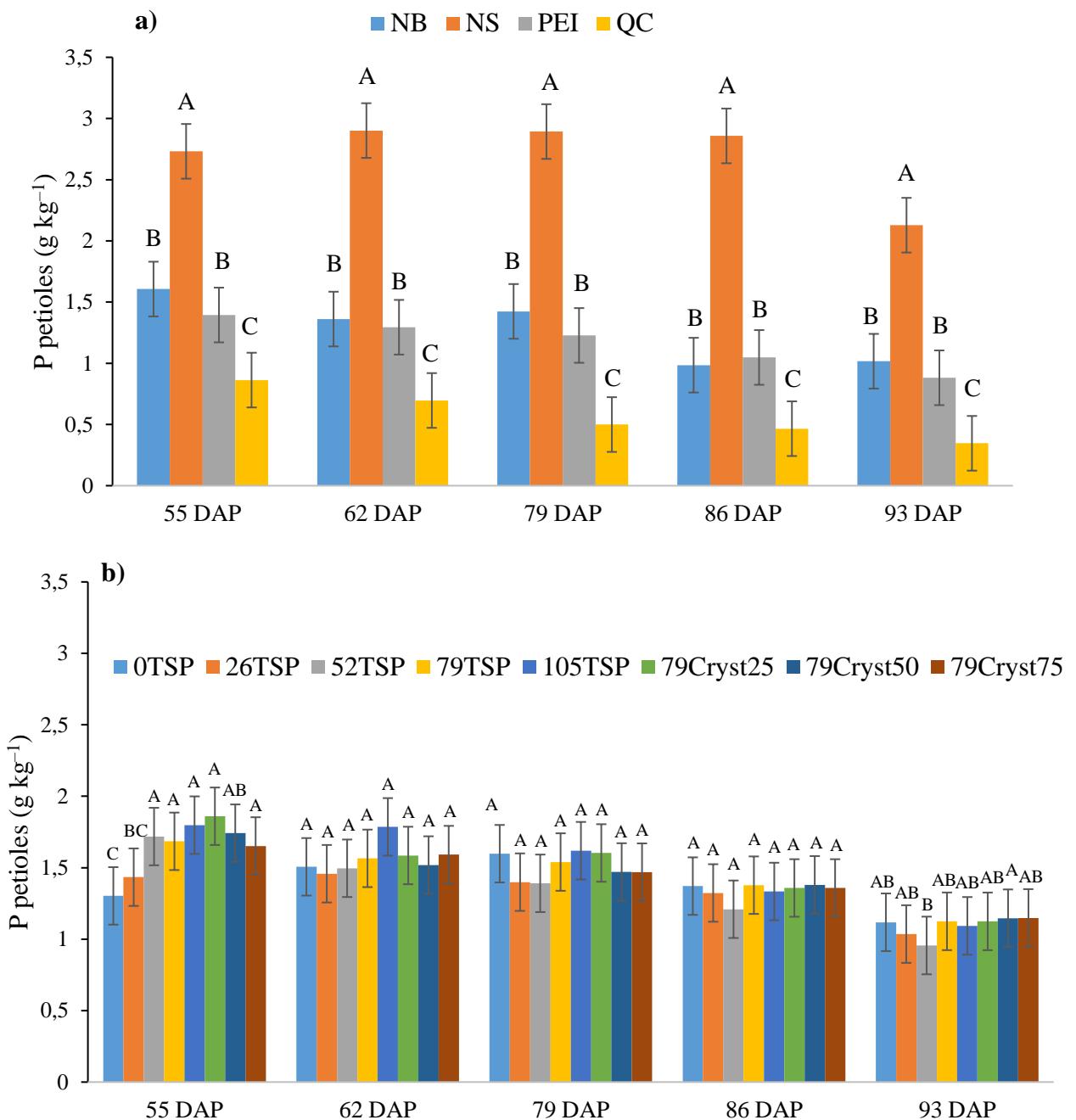


Figure 4.5. The interaction effect between (a) sites and sampling dates and (b) P treatments and sampling dates on the P petiole concentration at the New Brunswick (NB), Nova Scotia (NS), Prince Edward Island (PEI) and Quebec (QC) sites. Values followed by different letters within a group are statistically different at 0.05 probability level. Cryst25, Cryst50, Cryst75, struvite, trade name Crystal Green mixed with triple superphosphate (TSP) at 25%, 50% and 75% respectively. DAP, days after planting.

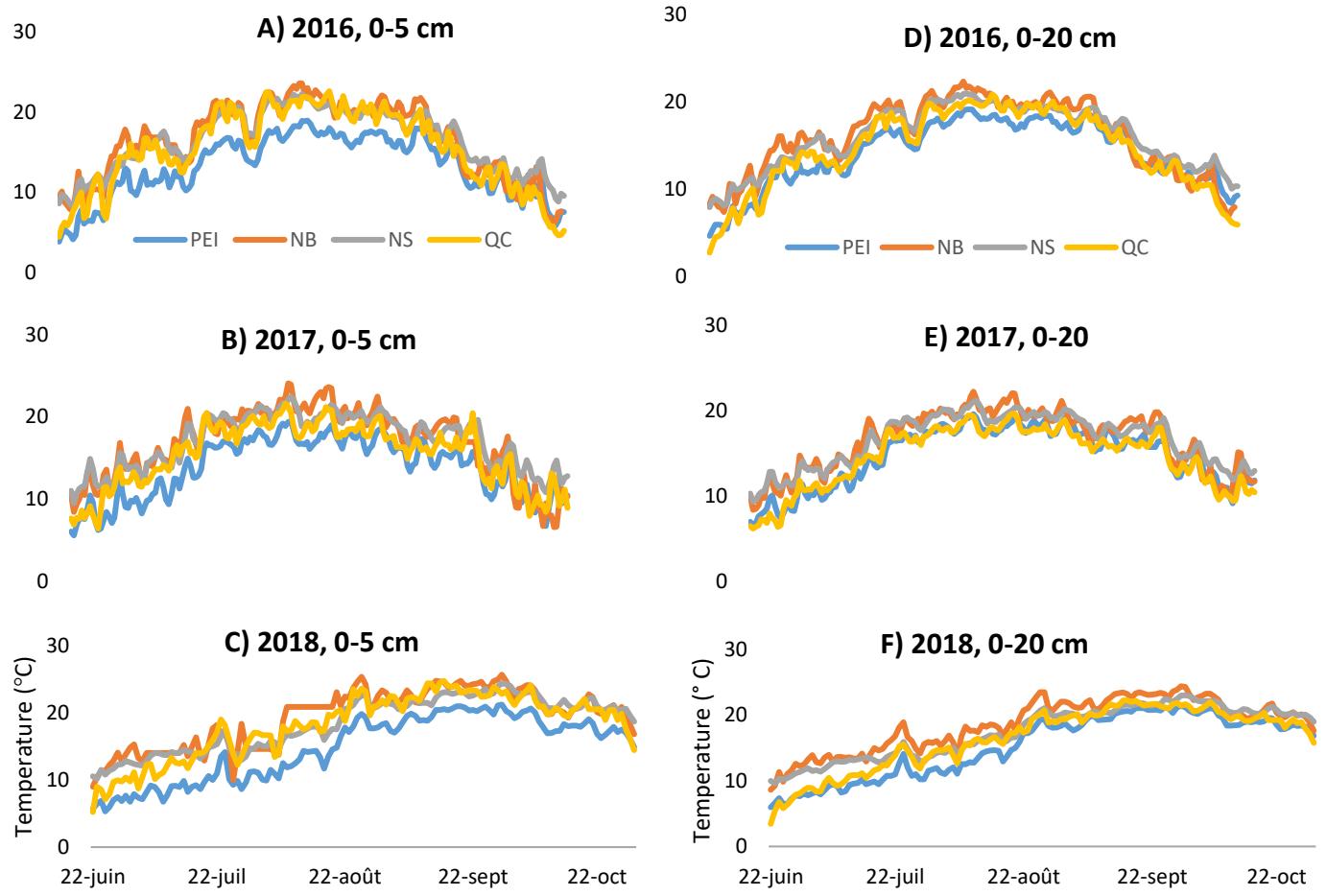


Figure 4.6. Soil temperature pattern at 0–5 cm and 0–20 cm depth at different sites over a 3 yr study period.

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Conclusion générale et recommandations

Cette thèse avait comme objectif général d'élaborer un système de diagnostic et de recommandation phosphatés à la fois agronomique et environnemental. Ce travail comprend trois aspects. Le premier est de nature environnementale et consiste à établir des niveaux d'interprétation pour le risque de saturation des sols en P basés sur l'ISP (indice de saturation en P). Le second est de nature agro-environnementale, puisqu'on utilise le même ISP pour faire le diagnostic et la recommandation du P dans des systèmes de productions de pommes de terre cultivées dans des sols hautement fixateurs en P. Le troisième vise à évaluer les performances agronomiques d'un engrais phosphaté issu du recyclage des biosolides municipaux et pouvant convenir aux sols hautement fixateurs en P.

Dans le premier aspect (chapitre 2), il a été démontré que le risque environnemental de saturation en P des sols agricoles de l'ÎPE est significativement moins important dans les sols fortement acides ($\text{pH}_{\text{eau}} < 5,5$) que dans les sols moins acides ($\text{pH}_{\text{eau}} > 5,5$). En effet, la désorption du P des sols étudiés a été proportionnelle à leur saturation en cet élément. Cette proportionnalité s'est avérée moins importante dans les sols fortement acides que dans les sols moins acides. Pour ces deux groupes de sols, nous avons obtenu deux indices critiques de saturation en P: un ISP_1 de 19,2% pour les sols de $\text{pH}_{\text{eau}} < 5,5$, et un ISP_1 de 14,2% pour les sols de $\text{pH}_{\text{eau}} > 5,5$. Au-delà de ces seuils critiques, les apports en engrais phosphatés doivent se limiter aux besoins de la culture de pomme de terre en phosphore. Un total de six classes de risque environnemental a été déduit pour les sols fortement acides et les sols moyennement acides, respectivement: classe de risque très faible (0–4%) et (0–7%), classe de risque faible (4%–7%) et (7%–11%), classe de risque moyen (7%–14%) et (11%–19%), classe de risque élevé (14%–16%) et (19%–21%), classe de risque très élevé (16%–23%) et (21%–30%) et classe de risque extrêmement élevé (> 23%) et (> 30%). Dans les sols fortement acides, la restriction environnementale est moins prononcée. Les résultats de la répartition spatiale de ces classes ont montré que la classe dominante à l'ÎPE correspond à un l'intervalle de saturation en P de risque moyen pour les sols de $\text{pH}_{\text{eau}} > 5,5$. Bien que les zones dont la saturation en P est considérée très élevée ou

extrêmement élevée représentent moins de 10% de la superficie totale de la province, il est important de comprendre les facteurs ayant contribué à une accumulation excessive du P dans ces sols. Ceci permettrait d'adopter les bonnes pratiques pour une meilleure gestion du P.

Dans le deuxième aspect (chapitre 3), l'étude de la réponse de la pomme de terre à la fertilisation phosphatée dans 42 sites établis dans les provinces maritimes (ÎPE, Nouveau-Brunswick (NB) et Nouvelle-Écosse (NÉ)), a permis d'établir deux modèles. Le premier modèle est agronomique et montre la réponse simultanée de la pomme de terre aux engrains phosphatés et à la saturation des sols en P. Soumis à une partition binaire Cate-Nelson, ce premier modèle a donné un seuil agronomique critique ISP_1 de 10% au-dessous duquel les probabilités de réponse économique de la pomme de terre aux engrains phosphatés sont élevées, et au-dessus duquel on assiste à une stabilité des rendements. À la suite de cet étalonnage, un deuxième modèle de recommandation a montré que les doses de phosphore économiquement rentables ont varié de 0 à 105 kg P ha^{-1} réparties sur les sept classes d'interprétation suivantes: (0–2,5%), (2,5%–5%), (5%–10%), (10%–14%), (14%–16%), (16%–23%) et $ISP_1 > 23\%$. Le modèle de recommandation qu'on suggère au bout de cette étude doit être consolidé par d'autres essais de fertilisation phosphatée pour la culture de pomme de terre dans ces régions. Ces essais devraient être réalisés sur des sols couvrant un large spectre de fertilité en phosphore, surtout dans les classes (0–2,5%) et de $ISP_1 > 23\%$ où on n'avait pas suffisamment de sites pour réaliser l'inférence statistique. Plus on alimente cette grille avec de nouvelles expérimentations de fertilisation phosphatée, plus robuste sera le modèle de recommandation. La taille de la banque de données est d'autant plus probante lorsqu'elle intègre les différents facteurs pouvant influencer la réponse de la culture à l'apport d'engrais. Selon cette même logique, il serait aussi pertinent de penser à uniformiser les recommandations en P dans les provinces maritimes et au Québec. En effet, comme il s'est avéré que les modèles de recommandation sont assez comparables dans les deux régions, un modèle commun qui réunit les provinces de l'Est du Canada est à explorer. Par contre, une attention particulière doit être portée aux spécificités de chaque région. Ainsi, les propriétés des sols, les données climatiques ainsi

que tous les facteurs pouvant influer la réponse de la culture à la fertilisation phosphatée devraient être considérés.

Le troisième aspect (chapitre 4) envisage d'évaluer l'effet de la substitution partielle de l'engrais phosphaté de synthèse hautement soluble dans l'eau, le triple super phosphate (TSP), par un engrais phosphaté issu du recyclage (struvite), le Crystal Green®, dont l'action est plus progressive dans le temps (engrais à libération lente). Les résultats ont montré qu'un mélange d'engrais contenant jusqu'à 75% de Crystal Green® a donné des rendements en pomme de terre et des prélevements de P par la plante comparables à ceux obtenus par l'engrais de synthèse TSP, et ce, dans les sites de QC, du NB et de la NÉ. Par contre, comparé à l'engrais TSP seul, une baisse de P prélevé et de rendements (total et vendable) a été notée dans les sites de l'ÎPE lorsque le TSP a été combiné à l'engrais à libération lente. Le statut du P dans le sol, tel que mesuré par les résines d'échanges anioniques ou par la solution Mehlich-3, a été aussi comparable entre le TSP seul ou quand il est mélangé avec le Crystal Green®, et ce malgré les réponses différentes de la pomme de terre à la fertilisation phosphatée entre les sites et la variabilité de leurs propriétés physico chimiques. Étant donné le nombre très limité d'études qui évaluent les performances des engrains phosphatés produits de recyclage au champ, les résultats de ces essais constituent un apport considérable de connaissances pour les systèmes de fertilisation phosphatée de pomme de terre. En effet, la majorité des recherches qui utilisent cette source de P pour la fertilisation ont été conduites sous conditions contrôlées. Il serait donc important de tester cette génération prometteuse d'engrais phosphatés au champ afin de vérifier leur efficacité sur les rendements et sur la qualité de la récolte de différentes cultures. Ceci permettra, non seulement de valoriser le phosphore présent dans la matière résiduelle fertilisante, mais aussi de familiariser les producteurs avec des engrais phosphatés plus économiques et plus écologiques. Contrairement aux engrais phosphatés conventionnels solubles à l'eau, la struvite est une forme de P à libération lente. Cette libération est étroitement liée aux exsudats racinaires des plantes, mais serait également influencée par les facteurs biotiques et abiotiques du milieu. Quelques-uns de ces facteurs, tels que le pH du sol, ont fait l'objet de certaines études, mais d'autres recherches sur l'impact de ces facteurs sur la dissolution de la struvite seraient requises. Le devenir de la struvite après application au niveau du sol serait aussi à étudier. En effet, comme la struvite

pourrait ne pas être complètement dissoute pendant la saison de culture, il serait nécessaire d'étudier sa dynamique dans le sol après la récolte.

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