



Transfer functions for phosphorus and potassium soil tests and implications for the QUEFTS model

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ABSTRACT

Multi-element soil extractions such as Mehlich 3 (M3) have gained popularity in recent years, but comparing outcomes to other soil testing methods is not always straightforward. In this study, extraction mechanisms of M3, Olsen and neutral 1 M ammonium acetate (AA) soil tests were explored and transfer functions were derived between P-Olsen and P-M3 as well as between K-AA and K-M3. Soils from tropical and temperate areas were used to derive these P and K transfer functions and were evaluated separately. The application of these transfer functions for tropical soils was evaluated by using them as input for the Quantitative Evaluation of the Fertility of Tropical Soils (QUEFTS). AA and M3 generally extracted similar amounts of K, but relations between K-AA and K-M3 were different for tropical and temperate soils. For tropical soils, the transfer function did not require additional parameters besides K-M3 to predict K-AA, but for temperate soils inclusion of clay content and pH was needed. This difference between tropical and temperate soils was explained by clay mineralogy. The relation between P-Olsen and P-M3 in tropical soils was found to be dependent on pH, Al-M3, Fe-M3 and Ca-M3. P-Olsen and K-AA values, calculated with their respective transfer functions, were used as input for QUEFTS. The yields predicted with measured P-Olsen and Exch. K were used as benchmark. For 63 out of 81 soil samples, predicted maize yields with transfer functions deviated less than 10% from the benchmark. The largest deviations from the benchmark were found for low P-Olsen and K-AA values, which corresponds to QUEFTS maize yield predictions up to 3000 kg ha⁻¹. We conclude that a M3 extraction results and soil pH can reliably be transferred to, and thus replace P-Olsen and K-AA determinations with the functions developed for tropical soils. The transfer functions can be used to generate input for the QUEFTS model with minor effects on yield predictions, thus expanding its applicability in cases where only M3 extraction results are available.

1. Introduction

Multi-element soil extractions have gained popularity in recent years. Their convenience and lower costs make them more attractive than the use of separate single element extractions (Iatrou et al., 2014). Mehlich-3 (M3) is a multi-element extraction employed in several parts of the world (Wuenschel et al., 2015). The M3 extraction contains a combination of chemicals (CH₃COOH, NH₄NO₃, NH₄F, HNO₃ and EDTA) designed to extract both macro- and micronutrients, among which phosphorus (P) and potassium (K). Single element extraction methods, such as Olsen, Bray, H₂O and CaCl₂, have been developed to quantify available soil P pools (Wuenschel et al., 2015), whereas plant available or exchangeable K and other cations have commonly been

estimated using 1 M ammonium acetate (Barbagelata, 2006).

Soil extraction methods are based on different mechanisms and vary in their extraction efficiency (Wuenschel et al., 2015). Ultimately, soil nutrient test results should relate to bioavailability, i.e. the amount of a nutrient available for plant uptake over a growing season. They are also used as input for decision support tools such as the Quantitative Evaluation of the Fertility of Tropical Soils (QUEFTS) model, which requires P-Olsen and K determined by a 1 M ammonium acetate extraction to estimate the soil's capacity to supply a crop with P and K (Janssen et al., 1990). P-Olsen and Exch. K extraction methods are not always routinely measured however, as other extraction methods such as M3 are more commonly employed in many countries (Wuenschel et al., 2015). Comparing the results of different soil testing methods is often not

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straightforward and requires transfer functions that include specific soil properties to translate the outcome of one soil test into another. Exploring the mechanisms behind soil extraction methods is needed to understand, describe and effectively apply relations between the nutrient pools measured by the different soil extractions. The focus in this study will be on comparing P and K in M3 to P-Olsen and K in ammonium acetate extractions, respectively.

The mechanisms for extracting soil K are similar for the 1 M ammonium acetate (AA) and M3 extraction methods. The extraction solutions differ considerably in pH (2.5 for M3 vs 7.0 for AA) and shaking time (5 min for M3; variable shaking times for AA), but both methods use high concentrations of NH_4^+ (0.25 M for M3 vs 1 M for AA) and a similar solution-to-solid ratio (SSR) of 10 L kg^{-1} to displace exchangeable cations such as K from soil surfaces. For M3, H^+ ions present at the extraction solution pH of 2.5, can displace additional cations. Relations between K-AA and K-M3 have previously been derived in various studies. The transfer functions between K-AA and K-M3 that were reported in literature (Table S1) show that regression slopes vary between 0.54 and 1.54 across studies. In each study, K-M3 was the only variable used to explain K-AA and the regressions showed an average R^2 value of 0.95, indicating K-M3 explains a substantial part of the variation in K-AA. The large variation in regression slopes, however, imply limitations for generic application among different soil taxonomic classes.

For P extraction, Olsen and M3 are based on contrasting mechanisms. The high concentration of bicarbonate in the Olsen extraction, buffered at pH = 8.5, leads to extraction of phosphate through (1) precipitation of Ca as CaCO_3 , thereby releasing Ca-bound phosphates, and (2) displacement of phosphate from the soil surfaces by increased competition with HCO_3^- , CO_3^{2-} and OH^- anions (Olsen et al., 1954).

M3 extracts P through two mechanisms, namely dissolution and complexation reactions. The high acidity of the extract (pH = 2.5) causes dissolution of Ca-P precipitates and of P bound to Al/Fe (hydr)oxides (Penn et al., 2018). At a solution pH of 2.5, the presence of NH_4F promotes the release of P from Al (hydr)oxides through Al-F complex formation. The pH of the M3 extraction solution increases during the procedure, its increase depending on soil pH (Penn et al., 2018). When the solution pH of M3 increases above 2.9 during the extraction procedure, fluoride also complexes Ca, thereby facilitating the release of P from Ca-P precipitates (Penn et al., 2018). Phytates, the largest pool of organic P, are desorbed from Al and Fe oxide surfaces and solubilised through protonation (Penn et al., 2018; Wang et al., 2017). The amount of organic P extracted with M3 can vary strongly from soil to soil (Iatrou et al., 2014; Mallarino, 2003; Pittman et al., 2005). Weak but significant correlations were found between the amount of organic P in a M3 extraction and pH ($R^2 = 0.32$) and C_{org} ($R^2 = 0.16$) (Mallarino, 2003).

Besides a difference in composition and contrasting P extraction mechanisms, the Olsen and M3 methods also differ in shaking time (5 min for M3 vs 30 min for Olsen), although in both methods no chemical equilibrium is reached (Olsen et al., 1954; Penn et al., 2018). An additional difference is the P species that are measured in the extracts. In the M3 extraction, often multiple elements are determined, using inductively coupled plasma – optical emission spectroscopy (ICP-OES) (Penn et al., 2018). As a result, total dissolved P (including organic P) in the extractant is measured. The standard Olsen extraction procedure includes a molybdate blue colorimetric determination of *ortho*-phosphate (ISO 11263, 1994; Olsen et al., 1954). Although certain kinds of organic P molecules can also be determined by colorimetric methods (Baldwin, 1998; Van Moorlehem et al., 2011), it is assumed that P-Olsen represents the inorganic P pool.

The transfer functions that have been reported in literature to predict P-Olsen based on P-M3 alone, show a fivefold variation in regression slopes, which range between 0.14 and 0.70 across studies (Table S2). The average R^2 value across studies is 0.74, but shows more variation compared to the K transfer functions, as R^2 values between 0.45 and 0.94 are reported. These findings indicate that also for P extractions,

transfer functions derived on one soil set may not be applicable to another. Several studies furthermore have shown that inclusion of additional soil properties such as pH, CaCO_3 content, organic matter, Fe and Al can improve relations between P-Olsen and P-M3 (Buondonno et al., 1992; Elrashidi et al., 2003; Iatrou et al., 2014; Schick et al., 2013; Sen Tran et al., 1990). In addition, categorization of soils based on pH and CaCO_3 content has resulted in different transfer functions for each category (Iatrou et al., 2014; Sen Tran et al., 1990) with a higher goodness-of-fit (R^2) compared to models fitted on the entire dataset (Buondonno et al., 1992; Zbiral and Nemeč, 2002; Table S2).

The above review of transfer functions between P-Olsen and P-M3 and between K-AA and K-M3 shows that P and K transfer functions mostly have been derived for soils from temperate regions such as North America and Europe (Table S1 and Table S2). Due to prolonged weathering, soils from tropical climatic regions generally differ from temperate soils in properties such as clay mineralogy and types and amounts of Fe and Al hydroxides (De Campos et al., 2018), which are known to affect P and K availability and may exert a significant influence on relations among soil P and K tests (Buondonno et al., 1992; Sharpley, 1989). As a consequence, transfer functions that have been developed based on temperate soils may not be applicable to soils from tropical areas. The first aim of this study, therefore, was to develop P and K transfer functions for soils from (sub)tropical regions. We compared these with transfer functions for temperate soils. The second aim of this work was to evaluate the application of the transfer functions for tropical soils. To this end, P-Olsen and K-AA values, estimated based on M3 data with the developed transfer functions, were used as input for QUEFTS and compared with yields predicted with measured P-Olsen and Exch. K values as the benchmark.

We hypothesize that K-M3 will be the only parameter needed to explain K-AA, but that the relation between P-Olsen and P-M3 requires additional variables. As the partitioning of soil P is highly pH dependent (Sims and Pierzynski, 2005) and soil pH affects P extraction efficiency of Mehlich 3 (Penn et al., 2018), we expect that soil pH will be an important factor in explaining the relation between P-Olsen and P-M3. Due to their contrasting mechanisms, M3 and Olsen are expected to extract different amounts of P bound as Ca-phosphates and P adsorbed to Al and Fe (hydr)oxides. We therefore hypothesize that Al, Fe and Ca in M3 can describe additional variation in the relation between P-Olsen and P-M3. We expect that organic carbon (C_{org}) can potentially also play a role in the P transfer function as organic P is determined in M3 extractions, but to a limited extent in Olsen. Finally, we expect that the uncertainty associated with predictions of P-Olsen and K-AA using the transfer functions will have an acceptable effect on yield predictions by the QUEFTS model, as C_{org} and pH are the most important determinants of the predicted yields (Janssen et al., 1990).

2. Materials and methods

2.1. Data availability

Three sets of soil samples and soil data were used for deriving the transfer functions. The first set consisted of 90 top soils (0–20 and 0–30 cm) that were sampled in several countries in Sub Saharan Africa, including Burundi, Congo, Ethiopia, Gabon, Kenya and Zambia. Soil properties of these samples were analysed for the purpose of this study (see Section 2.2). The second dataset was a subset of the World Soil Reference Collection (WSRC, 2020) of the International Soil Reference and Information Centre (ISRIC). From the WSRC, 51 soil samples from Ghana, Indonesia, Suriname and Russia were selected that included the required analytical data. The third dataset consisted of soil data from the Wageningen Evaluating Programmes for Analytical Laboratories (WEPAL, 2020). The WEPAL data were generated by several laboratories participating in this evaluation program. Laboratories remained anonymous as WEPAL only provided mean values and standard deviations for each soil parameter. The WEPAL dataset included analytical

data from 81 soils, originating from several African, South East Asian and European soils.

Both the WSRC and WEPAL datasets contained information on P-Olsen, pH-H₂O, organic carbon (C_{org}), K-AA and P, K, Al, Ca and Fe in M3. Additional soil data, such as clay content, CaCO₃ and Fe and Al measured in acid ammonium oxalate (AO), were also part of these datasets.

2.2. Soil analysis

2.2.1. Soil samples

The soil samples from Burundi, Congo, Ethiopia, Gabon, Kenya and Zambia that were obtained, were air-dried and passed through a 2 mm sieve before chemical analysis. Soil pH was measured in a suspension of the soil in water, prepared at a solution-to-solid ratio (SSR) of 2.5 L kg⁻¹ and after shaking for 2 h on a linear shaker at 180 S min⁻¹. For C_{org} determination, soil samples were colloid grinded (50 µm) before analysis (NEN-16179, 2012). C_{org} was then determined spectrophotometrically at 585 nm after chromic acid wet oxidation (Heanes, 1984). To determine P-Olsen, 2.5 g of soil was extracted with 50 mL of a freshly prepared solution of 0.5 M NaHCO₃ (pH = 8.5), followed by shaking in a horizontal shaker at 180 S min⁻¹ for 30 min. After filtration over a Whatman 110 mm filter paper, a subsample was diluted 5 times with 0.15 M HCl and placed in an ultrasonic bath to remove excess CO₂. Afterwards, the inorganic P concentrations were determined with the molybdenum-blue method (Kuo, 1996) and measured by a fully automated segmented flow analyser (SFA). Exchangeable K was extracted by adding 20 mL of a freshly prepared solution of 1 M NH₄ acetate (pH = 7.0) to 2 g of soil and shaking for 2 h in a horizontal shaker at 180 S min⁻¹. After extraction, suspensions were centrifuged for 15 min at 3000 rpm and filtered with a Whatman 110 mm filter paper. A subsample was taken and diluted 5 times with 0.14 M HNO₃ for measurement of K on ICP-OES. The shaking time of 2 h for the K-AA extraction was chosen in correspondence with the extraction time of the percolation method, that was originally used for calibration of QUEFTS (Houba et al., 1995; Van Reeuwijk, 2002). For 18 soil samples, we compared extractable K by using both the percolation method and the batch extraction procedure as described above and no significant differences in the amount of K were observed (data not presented). A M3 extraction solution was freshly prepared as a mixture of 0.2 M CH₃COOH, 0.25 M NH₄NO₃, 0.015 M NH₄F, 0.013 M HNO₃ and 0.001 M EDTA. The pH of the extraction solution was adjusted to 2.5 using concentrated HNO₃. The samples were extracted at a SSR of 10 L kg⁻¹ and shaken for 5 min in a horizontal shaker at 180 S min⁻¹ (Mehlich, 1984). Afterwards, suspensions were passed through a Whatman 110 mm filter paper, diluted 10 times with 0.14 M HNO₃ and analysed for P, K, Al, Ca and Fe on ICP-OES.

2.2.2. WSRC soils

The soil samples in the WSRC dataset were analysed between 2017 and 2019 by the Kellogg Soil Survey Laboratory in Lincoln, Nebraska, USA (USDA, 1996). Analyses were performed on air-dried samples that were passed through a 2 mm sieve. Soil pH was determined in water at a SSR of 1 L kg⁻¹ after an extraction time of 1 h (USDA, 1996; p. 276). C_{org} was calculated as the difference between CaCO₃ and total C of a given soil. Total C was determined in the soil fraction < 180 µm with an elemental analyser (USDA, 1996; p. 464). CaCO₃ was only determined in samples with pH-CaCl₂ > 6.95. After addition of 3 M HCl, samples were placed in a rotating shaker for 10 min at a rate of 140 rpm. The samples were shaken again at the last 10 min of a 1 h interval. Bottle stoppers were pierced with a hypodermic needle connected to a manometer to measure CO₂ development. The amount of carbonate was then calculated as percent CaCO₃ (USDA, 1996; p. 370). P-Olsen was determined in 1 g soil extracted with 20 mL of 0.5 M NaHCO₃ at pH 8.5. After shaking for 30 min at 200 oscillations min⁻¹, samples were centrifuged at 2000 rpm for 10 min. The PO₄ concentration was then determined

spectrophotometrically after adding molybdenum-blue colour reagent to an aliquot of the centrifuged sample (USDA, 1996; p. 336). K-AA was determined by extracting 2.5g of soil with 50 mL of 1 M ammonium acetate at pH 7.0. Extraction was done with a mechanical vacuum extractor and total extraction time was around 13 h. Samples were then shaken manually, and a subsample was submitted for analysis on the atomic absorption spectrophotometer (AAS) (USDA, 1996; p. 230). Nutrients in M3 were measured on the ICP-OES after extracting samples at a SSR of 10 L kg⁻¹, shaking for 5 min at 200 oscillations min⁻¹, centrifugation for 10 min at 2000 rpm and filtration with a Whatman no. 42 filter (USDA, 1996; p. 345). Fe and Al were determined in ammonium oxalate using a mechanical vacuum extractor and measured on ICP-OES. A quantity of 0.5 g soil was extracted with 50 mL of 0.2 M ammonium oxalate solution buffered at pH 3.0 for a total period of around 13 h. Afterwards, samples were shaken manually, diluted 10 times with reverse osmosis water, vortexed and submitted for analysis on the ICP-OES (USDA, 1996; p. 432). Clay content was determined using the pipette method (USDA, 1996; p. 48).

2.2.3. WEPAL soils

The soil samples in the WEPAL dataset were analysed by several labs, with potentially different soil-to-solution ratios (SSR) and extraction times for a given soil parameter as method details were not specified.

2.3. Data selection

The soils in this study were categorised based on their geographical location as “temperate” or “tropical”. These categories are inexhaustive and soil classification based on climatic zone has been criticised for being non-scientific (Hartemink, 2015). For the majority of soils however, soil taxonomic class and dominant clay mineralogy were not known; classification based on these soil properties therefore was not possible. The aim of this study furthermore was to derive functions that are generally applicable rather than being applicable to certain soil taxonomic classes only. In addition, weathering and climate are important controlling factors in the formation of clay minerals (Grim, 1968); commonalities within the categories of temperate and tropical soils are therefore expected. Despite its limitations, classification based on climatic zones was therefore considered most suitable for the purposes of this study.

From the WSRC dataset, only soils that were sampled within a depth of 0–60 cm were included, as layers beyond this depth were not considered relevant for agricultural production. Exact sampling location and sampling depth of the WEPAL soils were unknown and no selection based on this criterion was made.

To derive the K transfer functions for tropical and temperate soils, QUEFTS criteria for soil chemical parameters were used to select the soils that were included in the analysis: C_{org} below 70 g kg⁻¹ and exchangeable K below 30 mmol kg⁻¹ or 1173 mg kg⁻¹ (Janssen et al., 1990). The pH limits were set between 4.0 and 8.0, as most agricultural soils will have pH values within this range. After applying the selection criteria to the 114 samples available for tropical soils, a dataset with 101 samples remained (medians: C_{org} = 15.0 g kg⁻¹, pH = 5.61, K-AA = 2.6 mmol kg⁻¹ or 103 mg kg⁻¹; Figure S1). K-M3 concentrations in the tropical dataset ranged between 19.6 and 710.3 mg kg⁻¹. This dataset included samples from the following countries: Africa (not specified; n = 1), Burundi (n = 23), Congo (n = 1), Gabon (n = 6), Ghana (n = 7), Indonesia (n = 2), Ivory Coast (n = 3), Kenya (n = 26), Mali (n = 1), Philippines (n = 1), Suriname (n = 2), Thailand (n = 2) and Zambia (n = 26). For each soil, the country of origin, sampling year and crop species with the corresponding Exch. K and K-M3 values are presented in Table S3. For temperate soils, a total of 67 out of 86 samples remained after applying the selection criteria: South Africa (n = 1), The Netherlands (n = 41), Russia (n = 17), Spain (n = 1) and Switzerland (n = 7). K-M3 and K-AA concentrations in the temperate dataset ranged between 43 and 706 and 55–661 mg kg⁻¹ respectively.

To derive the P transfer functions for tropical and temperate soils, QUEFTS criteria for soil chemical parameters were used to select the soils that were included in the analysis: C_{org} below 70 mg kg^{-1} and P-Olsen below 30 mg kg^{-1} (Janssen et al., 1990). The pH limits were set between 4.0 and 8.0. After applying the selection criteria to the 119 samples available for tropical soils, a dataset with 92 samples remained (medians: $C_{org} = 14.7 \text{ mg kg}^{-1}$, $\text{pH} = 5.61$, $\text{P-Olsen} = 6.1 \text{ mg kg}^{-1}$; Figure S1). Mehlich 3 concentrations in the tropical dataset ranged between 0.6 and 93.5 (P) , $215\text{--}2114 \text{ (Al)}$, $3\text{--}3283 \text{ (Ca)}$ and $25\text{--}1171 \text{ (Fe)}$ mg kg^{-1} respectively. This dataset included samples from the following countries: Burundi ($n = 23$), Congo ($n = 1$), Gabon ($n = 5$), Ghana ($n = 14$), Indonesia ($n = 2$), Ivory Coast ($n = 2$), Kenya ($n = 24$) and Zambia ($n = 21$). For each soil, the country of origin, sampling year and crop species with the corresponding P-Olsen and P-M3 values are presented in Table S3. For temperate soils, 22 out of 72 samples remained after applying the selection criteria: The Netherlands ($n = 5$), Russia ($n = 13$) and Switzerland ($n = 4$). Mehlich 3 concentrations in the temperate dataset ranged between 3 and 204 (P), 133–1220 (Al), 101–12335 (Ca) and 67–1703 (Fe) mg kg^{-1} respectively.

2.4. Statistical analysis

The P and K transfer functions were developed using R software, version 3.4.4. Results were visualized using the 'ggplot2' package (version 2.2.1). Models were evaluated using R^2 and root mean squared errors (RMSE). Model residuals were checked for normality, homogeneity and independence.

For the P transfer function, all soil parameters including the dependent variable P-Olsen, except pH, were transformed using the natural logarithm to prevent negative predicted values and to normalise input data. No data transformations were applied for the K transfer function. Before running regressions, input parameters were checked for multicollinearity (vif greater than 4), using the vif function (package 'usdm', version 1.1–18). After checking for multicollinearity, model selection was done with stepwise regression (forward and backward) using the stepAIC function from package 'MASS' (version 7.3–50) using the Akaike Information Criterion as selection criterion that compromises between goodness of fit and parsimony (Webster and McBratney, 1989). The residuals of the selected model were inspected visually and checked for normality using the skewness function (package 'e1071', version 1.6–8) and using the Shapiro-Wilk test (function shapiro.test from package 'stats', version 3.4.4). Homogeneity and independence of residuals was visually evaluated by plotting residuals against fitted values and explanatory variables. The RMSE was retrieved using function rmse from package 'ModelMetrics' (version 1.1.0). The contribution of each soil parameter to R^2 was evaluated using function calc.relimp from package 'relaimpo' (version 2.2–3). Regression outliers were identified by checking Cook's distance (D), which is a measurement to identify data points with a high residual value as well as leverage. Data points were further inspected when D was greater than 0.5.

$\ln(\text{P-Olsen})$ predictions were back-transformed using Eq. (1) (Lark and Lapworth, 2012):

$$\text{prediction} = \exp(\ln\text{-prediction} + 0.5 \cdot \text{variance}) \quad (1)$$

To test whether the K transfer function was different for temperate and tropical countries, a regression model was fitted to predict K-AA (mg kg^{-1}) values based on K-M3 (mg kg^{-1}), an origin factor (i.e. temperate or tropical) and interaction of both predictors. The function lstrends from the package 'lstrends' (version 2.30–0) was applied to this regression model in order to test whether origin had an effect on the relation between K-AA and K-M3. Output of the lstrends function consisted of trends (i.e. slopes) per origin as well as the confidence intervals of these trends.

Additional analysis included exploration of the relations between M3 and ammonium oxalate (AO) extraction methods for Fe and Al. The AO

extraction is often used as a proxy to measure the micro-crystalline or short-range-order oxide minerals in soils which are considered the most reactive surfaces for adsorption of anions such as PO_4 (Hiemstra et al., 2010; Schwertmann, 1973). The relation between Fe and Al measured in M3 and AO indicates the efficiency of M3 to extract the reactive Fe and Al (hydr)oxides, which in turn could explain the extraction efficiency of P in M3. The soils that contained information on Fe-AO and Al-AO were used to explore relations between Fe-M3 and Fe-AO ($n = 55$) and between Al-M3 and Al-AO ($n = 55$).

2.5. Application

To evaluate application of the transfer functions, the QUEFTS model was used. The sensitivity of QUEFTS to deviations between measured and predicted P-Olsen and K-AA values was analysed for this purpose. QUEFTS can be used for the evaluation of soil fertility, potential crop yield and fertilizer recommendations. QUEFTS requires four soil chemical parameters as input, being exchangeable K (K-AA; in mmol kg^{-1}), P-Olsen, pH and C_{org} (Janssen et al., 1990; Sattari et al., 2014). These variables are used to calculate the soil supply of N, P and K (step 1), which is then used to calculate the potential uptake of nutrients by a crop (step 2). The interactions among nutrients define the actual uptake of nutrients (step 3), and the final yield estimate (step 4). Soils that were used to derive both the P and K transfer functions ($n = 81$) were used as input for QUEFTS. The most recent QUEFTS version for maize was used (Sattari et al., 2014), with potential yield set to 10 Mg ha^{-1} . QUEFTS was run with both measured and predicted P-Olsen and K-AA values. For pH and C_{org} , measured values were used.

3. Results

3.1. K transfer function

The relation between K-AA and K-M3 in tropical soils could be described with a single linear relationship. The best model that was fitted for tropical soils, violated assumptions however, as model residuals were not normally distributed ($p < 0.001$). Further inspection showed one data point with Cook's Distance greater than 1 (encircled in Fig. 1). The high residual value of this data point could not be explained by soil pH or C_{org} . This sample was consequently removed for further analysis. After rerunning the regression, K-M3 was the only significant variable for predicting K-AA (Fig. 1; Eq. (2)) and residuals showed a normal distribution ($p = 0.567$). The model explained 99% of the variation and RMSE was 11.2 mg kg^{-1} .

$$\text{Tropical soils: K-AA (mg kg}^{-1}\text{)} = 0.59 + 1.09 \cdot \text{K-M3 (mg kg}^{-1}\text{)} \quad (2)$$

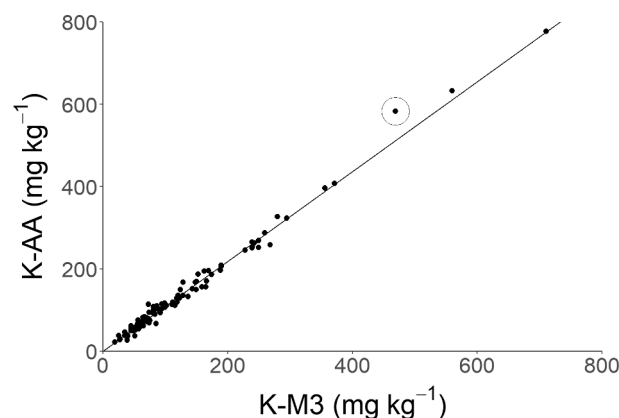


Fig. 1. Relation between K extracted with Mehlich 3 (M3) and ammonium acetate (AA) in tropical soils. The line represents the K transfer function. The circled data point was considered an outlier based on Cook's D and was not included in the regression.

The K transfer function was found to be dependent on the origin of the soil (Fig. 2). In tropical soils, AA extracted significantly more K ($p < 0.001$) than M3 compared to temperate soils. The best model that was fitted for temperate soils selected K-M3, C_{org} and clay content as predictors, but violated assumptions as model residuals were not normally distributed ($p < 0.001$). Further inspection showed two data points with high Cook's D values. The first data point had the highest K-M3, K-AA and C_{org} values, the second data point did not have soil parameters with extreme values. Soil pH could not explain the high residual value of these data points and they were consequently removed from the dataset. Regressions were re-run and the best model that was fitted on temperate soils required K-M3, clay content, C_{org} and pH to explain K-AA. Although residuals were normally distributed ($p = 0.062$), a regression was run without C_{org} as input parameter, as it contributed only 0.3% to R^2 . The best model that was fitted without C_{org} , selected K-M3, clay content and pH as predictors (Eq. (3)). The R^2 of the regression model was 0.996. Residuals showed a normal distribution ($p = 0.498$) and RMSE was 7.70 mg kg^{-1} . K-M3 explained 79% of the R^2 value, clay content 16% and soil pH 6%.

$$\text{Temperate soils: K-AA (mg kg}^{-1}\text{)} = 12.97 + 0.99 \cdot \text{K-M3 (mg kg}^{-1}\text{)} + 0.05 \cdot \text{Clay (g/kg)} - 2.52 \cdot \text{pH} \quad (3)$$

A simpler model without clay content as predictor was tested, as the laboratory procedure to determine soil texture is relatively time-consuming, compared to e.g. soil pH. The model that was fitted based on K-M3 and pH as predictors (Eq. (4)), explained 99.4% of the variation. Residuals showed a normal distribution ($p = 0.731$) and RMSE was 9.44 mg kg^{-1} . K-M3 explained 93% of the R^2 value and soil pH the remaining 7%.

$$\text{Temperate soils: K-AA (mg kg}^{-1}\text{)} = 15.21 + 1.01 \cdot \text{K-M3 (mg kg}^{-1}\text{)} - 2.12 \cdot \text{pH} \quad (4)$$

3.2. P transfer function

In tropical soils, P-Olsen and P-M3 were strongly correlated ($r = 0.87$, $p < 0.001$; Table S4). Both parameters were significantly positively correlated with Ca-M3, whereas P-M3 also showed a significant positive correlation with pH and a significant, negative correlation with C_{org} . For 14 out of 92 samples, Olsen extracted more P than M3. On average, these samples had lower pH values (5.22 vs 5.75 , $p = 0.013$), higher Al-M3 (1565 vs 860 mg kg^{-1} , $p < 0.001$) and higher C_{org} values (36 vs 14 g kg^{-1} , $p < 0.001$) compared to the (larger) subset in which P-Olsen

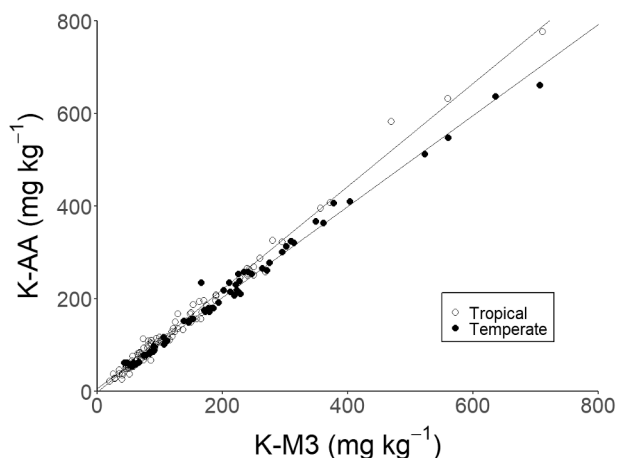


Fig. 2. Relations between K extracted with Mehlich 3 (M3) and ammonium acetate (AA), grouping based on climatic zone: (sub)tropical or temperate. Lines represent linear model fits for both groups.

concentrations were lower than P-M3. No differences in Fe-M3 contents were found ($p = 0.317$).

The best model that was fitted to explain P-Olsen, violated assumptions: model residuals were not normally distributed ($p = 0.006$). Further inspection showed two outliers based on Cook's D, that were characterised by having the lowest Ca-M3 and pH values in the dataset. The sample with the highest residual value (a soil from Indonesia) was removed from the dataset and regressions were rerun. The new dataset contained 90 samples from SSA, as well as one Indonesian sample from the same soil profile as the sample that was removed. Although residuals of the model excluding the first Indonesian soil sample were normally distributed ($p = 0.200$, skewness = -0.35), regressions were also run without the second Indonesian sample, as this could be considered an outlier based on geographical location. The model without both Indonesian samples performed slightly better in terms of R^2 (0.81 vs 0.80), but not RMSE (0.45 vs 0.41). The model predicting P-Olsen in soils from SSA only, used pH, and P-M3, Al-M3, Fe-M3 and Ca-M3 as explanatory variables (Eq. (5)). Residuals were distributed normally ($p = 0.309$, skewness = -0.27), but were positively correlated with $\ln(\text{P-Olsen})$ predictions ($p < 0.001$; Figure S2). Further inspection showed one outlier based on Cook's D, although its value was below 0.5, that was characterised by having the lowest Ca-M3 (2.8 mg kg^{-1}) and pH (4.10) values in the dataset. As the Cook's D value of the outlier was below 0.5 and no clear patterns between residuals and explanatory variables were observed, no attempt was made to further improve the model.

$$\ln(\text{P-Olsen}) = 0.77 \cdot \ln(\text{P-M3}) + 0.62 \cdot \ln(\text{Al-M3}) + 0.13 \cdot \ln(\text{Fe-M3}) + 0.10 \cdot \ln(\text{Ca-M3}) - 0.19 \cdot \text{pH} - 4.31 \quad (5)$$

The P transfer function described 81% of the variation in P-Olsen and RMSE was $0.45 \text{ (ln mg kg}^{-1}\text{)}$. Model predictions are presented in Fig. 3. Relative contribution to R^2 was highest for P-M3 (87%), followed by Al-

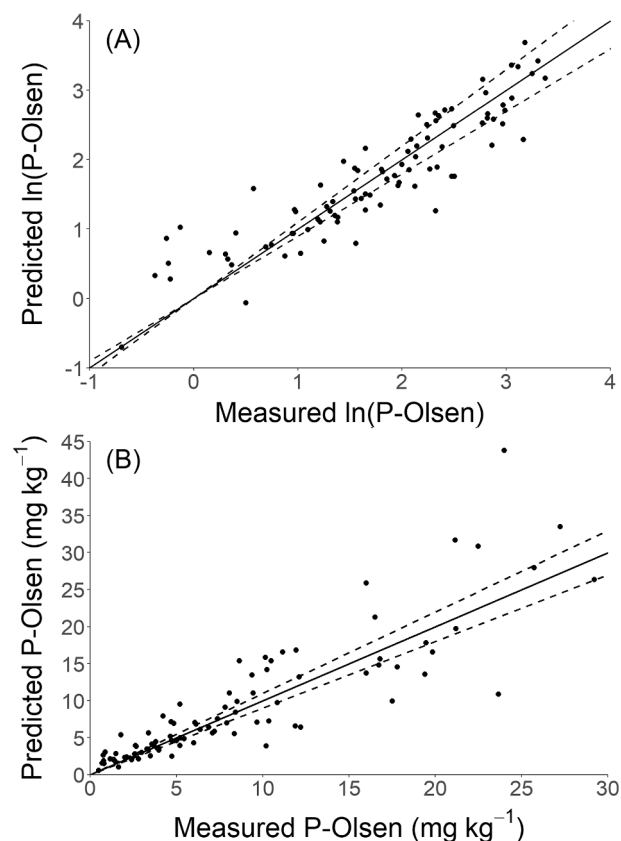


Fig. 3. Measured versus predicted P-Olsen on (A) a natural log scale and (B) after back-transformation. Dashed lines represent 10% deviation from the solid 1:1 line.

M3 (7%), Ca-M3 (2.6%) and pH (2.5%). Fe-M3 contributed <1% to the R^2 .

The dataset with temperate soils was considered too small ($n = 22$) to derive a P transfer function for temperate regions. The low number of remaining samples was partly due to the high P-Olsen values in the available data: more than half of the samples had P-Olsen values above 30 mg kg^{-1} , i.e. the QUEFTS limit for this parameter that was used in our data selection (section 2.3). The remaining 22 soil samples in the temperate dataset were compared to the tropical dataset for discussion purposes (Table S5). Temperate soils had significantly higher P-M3 ($p = 0.001$), Ca-M3 ($p < 0.001$) and Fe-M3 ($p < 0.001$) concentrations compared to tropical soils. Al-M3 concentrations were lower in temperate soils, but the difference with tropical soils was not significant ($p = 0.051$).

3.3. Application

When P-Olsen and K-AA values were predicted using the P and K transfer functions and subsequently used as input for QUEFTS, yield predictions for 63 out of 81 soils deviated less than 10% from the benchmark scenario in which the measured P-Olsen and K-AA values were used as input for QUEFTS (Fig. 4). Yield predictions of 10 observations deviated more than 15% from yield predictions that were based on measured P-Olsen and K-AA input. The corresponding soils were characterized by low P-Olsen and K-AA values: a maximum of 11.9 mg kg^{-1} for P-Olsen and 2.9 mmol kg^{-1} for K-AA, compared to the respective maximum values of 30 mg kg^{-1} and 8.4 mmol kg^{-1} for the complete dataset. Overall, when yield predictions surpassed 3000 kg ha^{-1} , deviations were <10% (Fig. 4). The corresponding soils were characterized by higher average P-Olsen ($11.0 \text{ vs } 7.7 \text{ mg kg}^{-1}$, $p = 0.037$) and K-AA ($4.5 \text{ vs } 2.1 \text{ mmol kg}^{-1}$, $p < 0.001$) values. Average C_{org} values for these samples also tended to be higher ($21.2 \text{ vs } 16.5 \text{ g kg}^{-1}$), but the difference was not significant ($p = 0.080$).

4. Discussion

4.1. Potassium

The AA and M3 methods extracted similar amounts of K (the regression slope was approximately 1.09 for tropical soils), which suggests a corresponding extraction mechanism, most likely $\text{NH}_4^+ \leftrightarrow \text{K}^+$ ion exchange. The differences between the K transfer functions for tropical and temperate soils may be partly methodological. The majority of data on temperate soils was obtained from WEPAL (50 out of 67 samples) for which exact protocols that were used for K-AA analysis are not known. The different extraction times employed in studies described in Table S1

(5, 10 or 30 min) indicate that methods can differ among laboratories. However, when a comparison was made between temperate ($n = 50$) and tropical soils ($n = 9$) within the WEPAL dataset, the ratio of K-AA/K-M3 was significantly higher in tropical soils compared to temperate soils ($p < 0.001$; data not presented). As K-AA measurements for tropical and temperate samples within the WEPAL dataset will have the same variability in extraction methods, it is unlikely that possible differences in extraction time are the main cause of the different K transfer functions obtained for tropical and temperate soils. This difference is more likely related to clay mineralogy, which influences bioavailability and exchange rate of cations (Grim, 1968). Relations among several K extraction methods were indeed found to be different for kaolinite, smectite and mixed clay soils (Sharpley, 1985). Weathering and climate are important factors in the formation of clay minerals; (sub)tropical soils generally contain kaolinite as the dominant clay mineral, whereas in temperate soils, illite or smectite clay minerals are more abundant (Grim, 1968). In tropical soils, AA was relatively more efficient than M3 in extracting K compared to temperate soils. In temperate soils, K is largely selectively bound, particularly to illite clay minerals (Blume et al., 2016), while in tropical soils K is more likely bound to general and less selective cation-exchange sites, such as those of kaolinite and organic matter. The high NH_4^+ concentrations in both extracts can rapidly displace the relatively weakly-bound K from kaolinite and organic matter. NH_4^+ also displaces selectively-bound K from illite clay minerals, but K-release from those sites is characterized by relatively slow exchange kinetics (Sumner and Bolt, 1962). These differences may explain the lower K-extraction efficiency of AA in temperate soils compared to tropical soils.

In contrast to tropical soils, the best model predicting K-AA in temperate soils required clay content and pH in addition to K-M3. It is unclear why clay content was a significant variable in this model. Results of the model without clay content (Eq. (4)) indicate that exclusion of this parameter does not affect precision of the predictions, although accuracy was lower. Soil pH had a negative coefficient in both models (with and without clay content), implying that M3 is relatively more efficient in extracting K than AA when soil pH increases. The acid M3 extract (pH 2.5 vs 7.0 for AA) may release additional K by partial dissolution of illite, smectite or other K-containing minerals that occur in temperate soils. Penn et al. (2018) showed that pH of the M3 solution increased with soil pH during the extraction procedure from 2.5 to ~ 3.1 and dissolution rates of illite and smectite clay minerals decrease across this pH range (Amram and Ganor, 2005; Köhler et al., 2003). We would thus expect that K-M3 extraction efficiency decreases with increasing soil pH, which is in contrast to our findings.

The two soils that were excluded in the final models for temperate soils, were Solonetz and Solonchak soils from Russia, which both are characterised by having high Na^+ concentrations (FAO, 2001a), as was also confirmed by additional data present in the WSRC dataset. The first soil that was removed from the dataset (Solonchak), was most likely identified as an outlier as it had the highest K-AA, K-M3 and C_{org} values (high leverage). The second soil that was removed (Solonetz) did not have soil parameters with extreme values. The ratio of K-AA/K-M3 for this soil was extremely high compared to the other temperate soils (1.41 vs average ratio of 1.03). As M3 and AA most likely have a corresponding mechanism to extract K, i.e. $\text{NH}_4^+ \leftrightarrow \text{K}^+$ ion exchange, the high concentration of Na^+ may have interfered with the K extraction in M3, that has lower NH_4^+ concentrations compared to AA (0.25 M vs 1 M). Although this may indicate limitations for applying the K transfer function to temperate soils in saline soils, in practice these soils are unlikely to be used for intensive agriculture.

The protocol developed for this study is based on an extraction time of 2 h for determination of K-AA, which resembles the extraction time in the percolation method that was originally used to calibrate QUEFTS (Janssen et al., 1990; Van Reeuwijk, 2002). We therefore consider the K transfer function for tropical soils developed in this study more suitable for generating K-AA input for QUEFTS than previous relations described

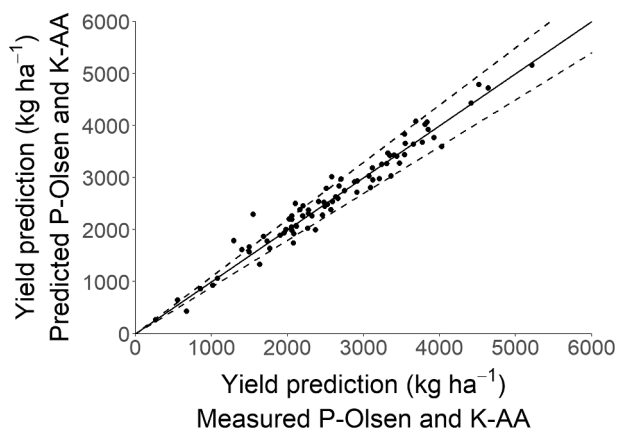


Fig. 4. QUEFTS yield predictions based on measured P-Olsen and K-AA values versus QUEFTS yield predictions based on predicted P-Olsen and K-AA values. Dashed lines represent 10% deviation from the solid 1:1 line.

in literature.

4.2. Phosphorus

The properties in the P transfer function in this study were able to explain 81% of the variance in $\ln(\text{P-Olsen})$ predictions, which is within the range reported in Table S2 (R^2 between 0.45 and 0.94, average 0.74). After back-transformation of $\ln(\text{P-Olsen})$ values, uncertainty increased with P-Olsen levels (Fig. 3B), which is a consequence of fitting regression models to log-transformed variables. However, in terms of soil P status and P fertiliser recommendations, predicting low P-Olsen concentrations accurately is most relevant. Though dependent on other soil properties, the critical P-Olsen concentration below which P is expected to be growth-limiting is around 10 mg kg^{-1} for maize (Bai et al., 2013; Ussiri et al., 1998). The increasing uncertainty with increasing predicted P-Olsen concentrations, especially for values above 10 mg kg^{-1} , is therefore not likely to have an effect on fertiliser recommendations.

Removal of one Indonesian sample from the dataset was needed to derive a model that did not violate the assumption of normality. This soil was classified as an Acrisol and was not used for agricultural purposes at the time of sampling. The results of the final P transfer function similarly showed a sample with a high Cook's D value (although below 0.5) due to very low Ca-M3 concentrations and pH. This soil from Congo was classified as a Ferralsol and was sampled from a rubber plantation. Acrisols and Ferralsols are known for their high P fixation and low nutrient availability (FAO, 2001b). This may indicate that the P transfer function may not predict P-Olsen concentrations well in P fixing soils. A number of other soil samples were also classified or could potentially be classified (based on geographical position) as Acrisols or Ferralsols, however, but were not found to be outliers. A second reason for the Indonesian and Congolese samples being identified as outliers, could therefore be the very low Ca-M3 concentrations (19.6 and 2.8 mg kg^{-1} respectively) or pH (4.16 and 4.10 respectively). This observation may indicate that the P transfer function has a limited applicability to predict P-Olsen in soils with a pH below 4.4 or Ca-M3 concentrations below $\sim 50 \text{ mg kg}^{-1}$.

This study has shown that Al-M3 is relevant for describing the relation between P-Olsen and P-M3 in tropical soils. This presumably is because Al-M3 is a good proxy for the amount of amorphous Al (hydr) oxides as confirmed by the strong similarity between Al measured in AO and M3 ($r = 0.79$, $p < 0.001$; Figure S3). The relation between Al-M3 and Al-AO was curvilinear and similar relations have been reported by Sen Tran et al. (1990) and Sims et al. (2002). The clear effect of Al-M3 was demonstrated by the fact that ratios of P-Olsen/P-M3 above 1 were associated with significantly higher Al-M3 concentrations compared to soils where the ratio was below 1, which was also reported by Buondonno et al. (1992). Thus the Olsen method was apparently more efficient than M3 in extracting P from soils with high amorphous Al content. We hypothesize that in these soils, saturation of the fluoride ion with Al^{3+} during the M3 extraction plays an important role in the reduced extraction efficiency of P associated with Al hydroxides. In the protocol that was used, 20 mL of M3 solution contains 0.3 mmol of F^- , which can complex maximally $0.1 \text{ mmol of Al}^{3+}$ in 2 g soil . This corresponds to around $1350 \text{ mg Al kg}^{-1}$ soil. In the 14 tropical soils in this study where P-Olsen concentrations were higher than P-M3, Al-M3 concentrations were 1565 mg kg^{-1} on average, which indicates that saturation of the fluoride ions may have limited P-M3 extraction in these soils. As the P extraction efficiency of Olsen is not affected by Al concentration, this method can extract more P than M3 from soils with high Al-M3 values. These findings support inclusion of Al-M3 or Al-AO when developing P transfer functions for M3 extractions. In contrast to Al, M3 and AO were not equally efficient in extracting Fe: Fe-M3 was found to be around 10% of Fe-AO (Figure S3; data not presented), which is in line with results reported by Sims et al. (2002). Fe-M3 therefore is not a good proxy for amorphous Fe-oxides, which may explain its limited contribution to the P transfer function.

As expected, soil pH was an important factor in the P transfer function for tropical soils. M3 was relatively more efficient in extracting P compared to Olsen when soil pH increased. Buondonno et al. (1992) argued that M3 is very efficient in dissolving Ca-bound phosphates that are relatively more abundant in alkaline soils (Sims and Pierzynski, 2005) and would consequently lead to higher P-M3 levels when soil pH increases. The positive correlation between P-M3 and Ca-M3 that was found in the tropical soils in this study confirms this hypothesis. We expect that complexation of Ca with fluoride plays an important role in extracting P when soil pH increases. For neutral and alkaline soils, the pH of the M3 solution is expected to increase above 2.9 during the extraction, which will consequently lead to Ca-F complexation and release of P from Ca-P minerals (Buondonno et al., 1992; Penn et al., 2018). Penn et al. (2018) however postulated that an increase in M3 solution pH would lead to a decrease in P-M3 concentrations, as a result of decreased desorption of P from Fe and Al (hydr)oxides. Penn et al. (2018), as well as Wuenscher et al. (2015) reported negative correlations between P-M3 and soil pH and between P-M3 and CaCO_3 , which is in contrast to our findings (Table S4). These studies used temperate soils in their analysis, however. This difference in soil types could indicate that the effect of soil pH on P extraction efficiency of M3 is dependent on soil mineralogy, which supports inclusion of proxies for Al, Fe and Ca reactive surfaces. Given the differences in P, Al, Ca and Fe-M3 values between temperate and tropical soils (Table S5), the applicability of the P transfer function to temperate soils may be limited.

In the tropical soils in this study, C_{org} was not found to be a significant factor in explaining the relationship between P-Olsen and P-M3, despite being correlated to P-M3 ($r = -0.27$, $p = 0.009$; Table S4). Although organic P can be a considerable fraction of total P-M3 concentrations (e.g. Iatrou et al., 2014; Pittman et al., 2005), correlations with C_{org} were weak (Mallarino, 2003). Correlations between the amount of organic P extracted by M3 and soil pH were stronger however (Mallarino, 2003). The work of Iatrou et al. (2014) also showed that the amount of organic P extracted was pH dependent. We therefore hypothesize that C_{org} does not affect P-M3 concentrations directly and that the use of soil pH in the regression made inclusion of C_{org} redundant.

4.3. Application

The use of the P and K transfer functions for predicting QUEFTS input yielded satisfactory results for the majority of soils, as QUEFTS yield predictions deviated $< 10\%$ from yield predictions based on measured P-Olsen and K-AA input. Extracted data from Figure 6b by Sattari et al. (2014), show that QUEFTS yield predictions deviated more than 10% from observed yields for the majority of their fields. The uncertainty introduced by the application of the transfer functions is thus less than the general uncertainty associated with QUEFTS predictions. The effect of the additional uncertainty introduced by the transfer functions on QUEFTS final yield estimate in relation to actual yields, currently is unknown. To gain insights in how the transfer functions affect QUEFTS ability to estimate actual yields, QUEFTS yield predictions based on M3 will have to be validated with field observations in future studies.

Using the transfer functions rather than measured P-Olsen and K-AA inputs had a limited effect on QUEFTS yield predictions. This can partly be explained by the high accuracy of the K-AA predictions using the K transfer function, but is also due to the fact that QUEFTS predictions of N, P and K supply to a crop are also based on soil pH and C_{org} values, besides P-Olsen and K-AA. As a result, only part of the uncertainty associated with P-Olsen and K-AA predictions will result in uncertainty in the final yield predictions. Furthermore, the uncertainty associated with P-Olsen concentrations, especially at higher levels is not propagated within QUEFTS, as yield predictions for soils with higher P-Olsen levels are more likely limited by N or K supply, than by P supply. In contrast, soils low in P-Olsen (and K-AA) were associated with deviations of more than 15% in QUEFTS yield predictions. The prediction uncertainty at low P-Olsen and K-AA values is relatively more important

in QUEFTS yield predictions, as the crop is more likely to be P or K limited. As a result, the largest model deviations occur when predicted yields were below 3000 kg ha⁻¹. Sattari et al. (2014) showed that QUEFTS yield predictions below 3000 kg ha⁻¹ generally have a higher degree of uncertainty than predictions above this level. This suggests that QUEFTS yield predictions below this level should be interpreted with caution, especially when P-Olsen and Exch. K inputs are predicted with the transfer functions developed in this study.

To minimise the uncertainty associated with the use of M3 data as input for QUEFTS, recalibration of QUEFTS P and K supply functions based on M3 soil data is recommended (Sattari et al., 2014). This requires, however, substantial time and capital investments, as NPK fertiliser omission trials need to be executed on a wide range of soils. Until such recalibrated P and K supply functions become available, the P and K transfer functions developed in this study can serve to generate P-Olsen and K-AA inputs for QUEFTS, with introduced uncertainties similar to those of current QUEFTS yield predictions.

5. Conclusions

We conclude that a Mehlich 3 (M3) extraction can be used effectively to predict K extracted by 1 M ammonium acetate (K-AA) in tropical soils, using the K transfer function developed in this study. The P transfer function that was developed in this study to estimate Olsen-P from P extracted by Mehlich 3, is associated with more uncertainty than the K transfer function. However, given that M3 extractions are more commonly used than Olsen soil tests in several parts of the world, the P transfer function can prove useful to estimate P-Olsen values in tropical soils when only M3 data are available. Log-transformation of input variables furthermore ensures that uncertainty in the relevant range up to 10 mg kg⁻¹ P-Olsen is minimised. QUEFTS is a decision support tool that can be used for the evaluation of soil fertility, potential crop yield and fertilizer recommendations. As QUEFTS requires P-Olsen and K-AA as input parameters, its application may be limited when these soil parameters cannot be analysed in local laboratories. We conclude that a M3 extraction and soil pH-H₂O can replace P-Olsen and K-AA determinations for predicting QUEFTS input for tropical soils, by using the P and K transfer functions developed in this study. These functions thus expand the applicability of the QUEFTS model to cases where only M3 extraction results are available.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.geoderma.2021.115458>.

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