Establishing grading indices of available soil potassium on paddy soils in Hubei province, China

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Soil testing is an important diagnostic tool for assessing crop-available soil potassium (K) and hence making appropriate fertilizer recommendation. This study was aimed at correlating grain yield response data to soil-test K extracted with ammonium acetate (NH$_4$OAc), cold nitric acid (HNO$_3$), sodium tetraphenylboron (NaTPB) and boiling HNO$_3$ solution, based on 54 field trials conducted during 2011 to 2015 across 15 counties in Hubei province, China. The specific objectives were to establish abundance and deficiency indices of available soil-K (ASK) for rice (Oryza sativa L.) and make accurate K fertilizer recommendations. Potassium extracted with NaTPB and boiling HNO$_3$ was 1.47 times and 3.61 times higher respectively than that extracted with cold HNO$_3$, while K extracted with cold HNO$_3$ was 1.32 times higher than that extracted with NH$_4$OAc. There were significant logarithmic relationships between crop response and soil-test K. The $R^2$ values for cold HNO$_3$-K and NaTPB-K methods were much higher than for NH$_4$OAc-K method. In order to calibrate the application, the abundance and deficiency indices of ASK categorized by cold HNO$_3$-K in low, medium, high and very high ranges were $<50$ mg kg$^{-1}$, 50 to 100 mg kg$^{-1}$, 100 to 200 mg kg$^{-1}$ and $>200$ mg kg$^{-1}$ respectively, and that defined by NaTPB-K were $<60$ mg kg$^{-1}$, 60 to 150 mg kg$^{-1}$, 150 to 330 mg kg$^{-1}$ and $>330$ mg kg$^{-1}$, respectively. These values could be used to evaluate soil K supplying capacity and make appropriate K fertilizer recommendations for rice.

In agricultural production systems, fertilizer application is required not only to ensure but also to sustain an adequate supply of available nutrients to crops. However, application rates and timing of organic or inorganic fertilizers are often based on an optimal nitrogen supply, but unfortunately, potassium (K) requirement is often neglected. It may lead to an excess or a shortage of K, depending on both crop and soil characteristics. A monitoring of soil K reserves is, therefore, extremely important in order to make accurate fertilizer recommendation.

Soil testing is an important diagnostic tool for assessing crop-available soil K in crop production systems. An estimation of exchangeable soil K by extracting air dried soil sample with neutral ammonium acetate (NH$_4$OAc) is the most widely used soil-test and provides the basis for K fertilizer recommendations. The inadequacy had been clearly demonstrated in illitic and vermiculitic soil. It does not measure plant-available non-exchangeable K (NEK). Adoption of the Mehlich-3 extractant to estimate plant available K is rapidly increasing in the United States. The Mehlich-3 extractant has been adapted by many laboratories as a near-universal extractant. Soil tests based on these two extractants are suggested for soils of the north-central region by the North-Central Extension and Research Committee for Soil Testing and Plant Analysis, USA. Mengel et al. showed that the extraction methods may provide sufficient information for fertilizer recommendations in light textured soils that do not contain 2:1 type clay minerals.

However, the contribution of NEK due to the presence of 2:1 type layer silicates in soils that have the ability to retain K has questioned the validity of using exchangeable K soil extraction methods. In some cases, the non-exchangeable K pool (i.e. K in interlayer sites) can make a considerable contribution (80–100%) to plant available K. The role of this pool becomes particularly important under K-mining conditions when the exchangeable K is low. The significance of the contribution of NEK has probably been underestimated. Nevertheless, various methods have been established to assess slowly or potentially available K in soils, e.g. extraction with...
hydrochloric acid (HCl), boiling in nitric acid (HNO₃), electro-ultrafiltration, exchange resins, sodium tetraphenylboron (NaTPB), and field balances.

It was found that Step-K (loosely bound NEK released by repeated extraction with HNO₃) was a more reliable index of the K-supplying power of Canadian soils than is NH₄OAc-extractable K. However, this approach is not suited for routine soil test purposes because of the extra equipment, bench space, and time required to estimate K. Soil K extraction with NaTPB is characterized by the formation of potassium tetraphenylboron (KBP₄) precipitate, which reduces the concentration of K⁺ in soil solution, thus facilitating the further release of K⁺ from the interlayer sites of soil minerals. The NaTPB method is not only used for characterizing the release of NEK from soil or minerals, but has also been shown to be a good method for determining soil K availability to plants. This method was first proposed by Hanway in 1956 and since then gradually modified by many researchers. The 60-min modified NaTPB method without sodium chloride (0.2 mol L⁻¹ NaTPB) is suitable for evaluating K availability to plants in a variety of soils.

In order to provide theoretical basis for rice production and fertilization by soil testing, 54 field experiments regarding rice yield response to K application were carried out at different locations during 2011–2015. Available K in soils were evaluated using different extractants, i.e. 1.0 mol L⁻¹ neutral NH₄OAc, 2.0 mol L⁻¹ cold HNO₃, 1.0 mol L⁻¹ boiling HNO₃ and 0.2 mol L⁻¹ NaTPB, respectively. The specific objectives were (1) to establish abundance and deficiency indices of ASK for rice, (2) to find the most suitable method for assessing ASK and making accurate K fertilizer recommendations.

### Results

#### Amount of soil K extracted

The concentrations of cold HNO₃-K, NH₄OAc-K, NaTPB-K and boiling HNO₃-K of non-fertilized soil across all site-years ranged from 40.0 to 242.4 mg kg⁻¹, 22.9 to 192.6 mg kg⁻¹, 36.1 to 451.6 mg kg⁻¹ and 109.8 to 1095.3 mg kg⁻¹ respectively, average values being 130.5 mg kg⁻¹, 98.6 mg kg⁻¹, 192.1 mg kg⁻¹ and 471.0 mg kg⁻¹ respectively (Fig. 1). It showed that the mean values of NaTPB-K and boiling HNO₃-K were 1.47 times and 3.61 times higher than of cold HNO₃-K. Similarly, the mean values of cold HNO₃-K was 1.32 times higher than that of NH₄OAc-K.

Table 1 showed relationships between soil-test K extracted with 1.0 mol L⁻¹ NH₄OAc, 2.0 mol L⁻¹ cold HNO₃, 0.2 mol L⁻¹ NaTPB and 1.0 mol L⁻¹ boiling HNO₃ solution. **Refers to significant correlation at P < 0.01.

#### Grain yield and K uptakes response to K application

Regression equations were established to investigate the influences of K fertilizer levels on grain yields and hence calculate the optimum amount of fertilizer required to obtain the highest yield at each site (data did not shown). The wide range of growing conditions considered in this study resulted in grain yields varying from 2825 to 10240 kg ha⁻¹ in non-fertilized plots and from 3775 to 10520 kg ha⁻¹ in optimum K fertilized plots (Fig. 2). Analysis of variance of K effects on yield for
individual sites (not shown) indicated a significant yield increase ($P < 0.05$), on average 10% increase due to K fertilization of the rice site-years. Potassium uptake by aboveground parts ranged from 71.6 to 331.6 kg ha$^{-1}$ in non K fertilized plots and from 122.9 to 343.7 kg ha$^{-1}$ in K fertilized plots. It varied across different sites. Potassium fertilizer application significantly improved K uptakes ($P < 0.05$). On average, K uptake in response to K fertilization was increased by 18.9% significantly ($P < 0.05$).

The distribution of grain yield increase rate due to K fertilization in 54 trials was illustrated in Fig. 3. It showed a significant unimodal distribution, with 5%~10% as the crest. Grain yield of 25.9% of the 54 trials did not respond significantly to K fertilization (increase rate $< 5$%). The percentage of the 54 trials with grain yield increase rate at 5%~10%, 11%~15%, 16%~20%, 21%~25% and $> 25$% were 31.5%, 18.5%, 11.1%, 9.3% and 3.7%, respectively. It indicated that soil K status of the rice trials was uniformly distributed, which was suitable to establish the abundance and deficiency indices of ASK for rice.

**Relationship between soil-test K and crop yield response to K.** Figure 4 showed relationship between relative grain yield (RGY), relative K uptake (RKU) and soil test K extracted with different extractants. The data showed the known general relationship between crop yield response and soil-test values, but also showed high unexplained variability. The $R^2$ values ranged from 0.2155 to 0.3511 for RGY and ranged from 0.2077 to 0.5128 for RKU. There were small differences between soil-test methods. It also indicated high efficiency of cold $\text{HNO}_3$-K and NaTPB-K methods to assess crop K sufficiency and predict crop response to K fertilization in rice.

Table 2 showed abundance and deficiency indices of ASK calculated by the logarithmic models. Abundance and deficiency indices of ASK based on regression equation greatly varied depending on the soil-test method and model used. In four methods, the abundance and deficiency indices values were lowest with $\text{NH}_4\text{OAc}$-K method, intermediate with cold $\text{HNO}_3$-K and NaTPB-K method, and highest with boiling $\text{HNO}_3$-K method. In order to facilitate the application, the adjusted abundance and deficiency indices of ASK for low, medium, high and very high levels of cold $\text{HNO}_3$-K were $< 50$ mg kg$^{-1}$, 50 to 100 mg kg$^{-1}$, 100 to 200 mg kg$^{-1}$ and $> 200$ mg kg$^{-1}$, respectively. The corresponding values defined by NaTPB-K were $< 60$ mg kg$^{-1}$, 60 to 150 mg kg$^{-1}$, 150 to 330 mg kg$^{-1}$ and $> 330$ mg kg$^{-1}$, respectively. According to established abundance and deficiency indices of ASK, average cold $\text{HNO}_3$-K values for low, medium, high and very high levels in this study were 45.7, 83.6, 150.4 and 218.1 mg kg$^{-1}$, respectively. Similarly, average NaTPB-K values for low, medium, high and very high levels were

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**Figure 2.** Rice grain yield and K uptake by aboveground for all trials sites.

**Figure 3.** Frequency distribution of grain yield increase rate of the + K treatment compared to –K treatment for the 54 trials.
50.8, 119.9, 236.6 and 416.4 mg kg\(^{-1}\) respectively, and the average grain yield increase rates were 15.4%, 13.8%, 7.9% and 5.2% respectively. It demonstrated that abundance and deficiency indices of ASK could help evaluate soil K supplying capacity and provide evidence for making accurate K fertilizer recommendations.

Discussion

For soil-test-based K fertilizer recommendations to be accurate, the relationship between the soil K availability index and rice yield must be determined and followed by calibrating the proper K fertilizer rates to the soil nutrient availability index\(^{13}\). This research found that there were significant logarithmic relationships between rice yield response and soil-test K extracted with NH\(_4\)OAc, cold HNO\(_3\), NaTPB and boiling HNO\(_3\). The cold HNO\(_3\)-K and NaTPB-K were relatively more efficient in predicting rice response to K fertilization compared with NH\(_4\)OAc-K. It indicated that cold HNO\(_3\)-K and NaTPB-K methods should be appropriate for rice in Hubei Province, China. However, Slaton et al. correlated relative rice yield with Mehlich-3 and 1 mol L\(^{-1}\) HNO\(_3\) extractable K and suggested Mehlich-3 soil and plant K critical concentrations should be appropriate for U.S. mid-South rice-producing areas\(^{13}\). This showed that the method for evaluating soil available K for rice differed in regions. Adoption of the Mehlich-3 extractant to estimate plant available K, which includes NH\(_4\)OAc in its extracting solution\(^{3}\), is increasing rapidly in the United States. However, Nair et al. showed that NH\(_4\)OAc and Mehlich-3 extractable K lone was a poor indicator of K availability to cardamom in kaolinitic soils, unless it is integrated with soil K buffer power\(^{14}\). It indicated that the NH\(_4\)OAc and Mehlich-3 methods were affected by soil clay mineral composition. Potassium release from soil is dependent on the type and amount of K-bearing minerals\(^{15}\). Fixation and release of NEK affects the abundance and binding strength of exchangeable K, as well as quantity-intensity factors\(^{16}\). There are two ways
The plant availability of NEK depends primarily on the rate at which it can be released as more labile forms compared with that of NH₄OAc-K (Fig. 4). So, respective grading indices of available soil K on paddy soils were proposed using cold HNO₃-K as the method of determining the available K of paddy soil [28]. This paper showed that soil available K extracted with cold HNO₃ could also be appropriate for paddy soils in Hubei Province, China. The abundance and deficiency indices of ASK as defined by NaTPB-K (Table 4) should be used to make K fertilizer recommendations for rice in this region. Traditionally, K availability in soils is studied by its extraction with neutral 1.0 mol L⁻¹ NH₄OAc. However, cereal crops like rice, wheat and ryegrass etc. have strong K absorption capacity [24,25].

### Table 3. Selected properties of the 54 soils included in the trials. Avail., available.

| No. | pH  | Organic matter (g kg⁻¹) | Total N (g kg⁻¹) | Avail. P (mg kg⁻¹) | NH₄OAc K (mg kg⁻¹) | Avail. NH₄OAc-K (mg kg⁻¹) | No. | pH  | Organic matter (g kg⁻¹) | Total N (g kg⁻¹) | Avail. P (mg kg⁻¹) | NH₄OAc K (mg kg⁻¹) |
|-----|-----|-------------------------|-----------------|-------------------|--------------------|-------------------------|-----|-----|-------------------------|-----------------|-------------------|-------------------|
| 1   | 6.08| 38.40                   | 2.11            | 18.9              | 47.1               | 28                      | 7.86| 55.23                | 2.76            | 23.7              | 109.4             |
| 2   | 6.68| 28.15                   | 1.91            | 10.9              | 82.8               | 29                      | 6.23| 23.03                | 1.52            | 17.8              | 170.5             |
| 3   | 6.75| 32.15                   | 1.46            | 12.0              | 44.5               | 30                      | 7.96| 38.50                | 2.22            | 27.2              | 155.2             |
| 4   | 5.57| 23.82                   | 1.25            | 9.6               | 33.1               | 31                      | 6.45| 20.67                | 1.17            | 42.5              | 155.2             |
| 5   | 5.76| 27.18                   | 1.61            | 7.1               | 63.6               | 32                      | 6.60| 44.93                | 2.64            | 21.3              | 134.9             |
| 6   | 6.86| 22.19                   | 1.38            | 7.7               | 58.5               | 33                      | 6.76| 44.37                | 2.36            | 46.2              | 124.7             |
| 7   | 5.64| 30.32                   | 1.54            | 10.6              | 22.9               | 34                      | 5.26| 30.50                | 1.68            | 15.5              | 155.2             |
| 8   | 6.90| 26.83                   | 1.69            | 18.8              | 101.2              | 35                      | 7.99| 14.26                | 1.20            | 12.3              | 104.3             |
| 9   | 6.61| 31.98                   | 1.44            | 10.4              | 61.3               | 36                      | 6.8  | 23.2                | 1.30            | 5.6               | 106.3             |
| 10  | 6.05| 33.06                   | 1.55            | 6.9               | 65.7               | 37                      | 5.8  | 22.5                | 1.40            | 9.4               | 152.8             |
| 11  | 5.92| 35.26                   | 1.89            | 25.9              | 61.3               | 38                      | 5.0  | 20.7                | 1.30            | 12.5              | 79.7              |
| 12  | 5.70| 30.60                   | 2.00            | 7.9               | 34.8               | 39                      | 6.6  | 22.4                | 1.30            | 10.0              | 59.8              |
| 13  | 5.90| 31.07                   | 1.74            | 12.8              | 68.7               | 40                      | 5.3  | 18.0                | 1.00            | 5.0               | 86.3              |
| 14  | 5.63| 30.14                   | 1.94            | 18.9              | 63.6               | 41                      | 4.6  | 23.2                | 0.80            | 5.2               | 86.3              |
| 15  | 6.07| 38.71                   | 2.06            | 11.4              | 170.5              | 42                      | 5.7  | 26.4                | 1.40            | 3.1               | 166.0             |
| 16  | 5.81| 24.61                   | 1.45            | 8.7               | 99.2               | 43                      | 7.1  | 26.7                | 1.50            | 12.5              | 179.3             |
| 17  | 6.68| 10.11                   | 0.61            | 7.5               | 129.8              | 44                      | 7.7  | 30.2                | 1.80            | 3.5               | 93.0              |
| 18  | 5.43| 25.70                   | 1.24            | 43.3              | 84.0               | 45                      | 5.6  | 26.5                | 1.80            | 30.2              | 79.7              |
| 19  | 5.28| 22.42                   | 1.26            | 8.4               | 78.9               | 46                      | 6.0  | 26.9                | 0.61            | 8.1               | 192.6             |
| 20  | 5.00| 19.90                   | 1.25            | 60.2              | 99.2               | 47                      | 5.3  | 27.3                | 0.70            | 17.4              | 159.4             |
| 21  | 7.24| 18.35                   | 1.11            | 10.3              | 53.4               | 48                      | 5.18| 30.12                | 1.54            | 12.73             | 73.1              |
| 22  | 5.73| 21.59                   | 1.26            | 15.1              | 33.1               | 49                      | 4.98| 33.02                | 1.67            | 12.16             | 73.1              |
| 23  | 5.95| 16.88                   | 1.10            | 10.5              | 78.9               | 50                      | 4.97| 32.80                | 1.60            | 14.3              | 79.7              |
| 24  | 5.24| 23.47                   | 1.27            | 35.6              | 70.3               | 51                      | 4.76| 25.6                | 2.00            | 2.32              | 33.2              |
| 25  | 5.68| 22.52                   | 1.17            | 10.9              | 74.5               | 52                      | 6.80| 21.3                | 1.16            | 12.62             | 192.6             |
| 26  | 5.96| 26.48                   | 1.49            | 7.1               | 70.0               | 53                      | 6.20| 30.5                | 1.10            | 12.81             | 132.8             |
| 27  | 5.64| 23.06                   | 1.62            | 20.8              | 25.4               | 54                      | 6.00| 29.9                | 1.31            | 10.68             | 172.7             |

Conclusions

Grain yield and K uptake of rice in Hubei province, China, in response to K fertilization which varied across different sites were increased significantly, on average, by 10.0% and 18.9%, respectively. The grain yield increase rate exhibited a significant unimodal distribution, with 5%~10% as the crest. There were significant logarithmic relationships between crop yield response and soil-test K. The cold HNO₃-K and NaTPB-K were relatively more
efficient in predicting rice response to K fertilization compared with NH₄OAc-K. The abundance and deficiency indices of ASK for low, medium, high and very high ranges as defined by cold HNO₃-K were <50 mg kg⁻¹, 50 to 100 mg kg⁻¹, 100 to 200 mg kg⁻¹ and >200 mg kg⁻¹ respectively, and that defined by NaTPB-K were <60 mg kg⁻¹, 60 to 150 mg kg⁻¹, 150 to 330 mg kg⁻¹ and >330 mg kg⁻¹, respectively. It could be used to evaluate soil K supplying capacity and hence making K fertilizer recommendations for rice in Hubei province, China.

Materials and Methods
Rice grain yield data and soil samples were collected from K response trials conducted during 2011 to 2015 across 15 counties in Hubei province, China where oilseed rape (Brassica napus L.) - rice rotation system predominates (Table 3). A total of random 54 field trials of K application response were conducted in farmers’ fields which belong to the main rice producing areas (Fig. 5). Each trial was comprised of four K fertilizer (granulated potassium chloride, 500 g K kg⁻¹) treatments (K₀, K₁, K₂ and K₃) applied as basal fertilizer. Nitrogen (N) and phosphorus (P) fertilizers were applied across all plots of each trial at rates that were recommended by local Agricultural Technology Promotion Center and Huazhong Agricultural University (Table 4).

Rice was manually transplanted in all the trials. Other management practices such as rice cultivars, seedling rates, transplanting dates and weed control measures were those normally used at each county. At physiological maturity, rice grain yield was determined from an area of 10 m² in each plot through manual harvesting and mechanical threshing and adjusted to the standard moisture content of 14%; Plant samples were collected from 6 ridges per plot and separated into straw and grain. Total K content of plant material was determined after dry ashing method in a muffle furnace at 500 °C for 2 h.

Composite soil samples (12–15 cores) were collected from 0 to 20 cm soil depth before applying K fertilizer. Table 4. Range and average of fertilizer application rate of the 54 trials.

| Treatment | N Range | Ave. | P₂O₅ Range | Ave. | K₂O Range | Ave. |
|-----------|---------|------|-------------|------|-----------|------|
| K₀        | 135–180 | 166 ± 12 | 45–90 | 62 ± 13 | 0 | 0 |
| K₁        | 135–180 | 166 ± 12 | 45–90 | 62 ± 13 | 22–60 | 41 ± 24 |
| K₂        | 135–180 | 166 ± 12 | 45–90 | 62 ± 13 | 45–120 | 85 ± 29 |
| K₃        | 135–180 | 166 ± 12 | 45–90 | 62 ± 13 | 67–180 | 128 ± 34 |

Table 4. Range and average of fertilizer application rate of the 54 trials.
(0.2 mol L⁻¹ NaTPB, 0.01 mol L⁻¹ EDTA) was added. After shaking at 200 r.p.m. for 60 min, 25 mL of quenching solution (0.5 mol L⁻¹ NH₄Cl + 0.14 mol L⁻¹ CuCl₂) was poured into the tubes to stop the extraction of soil K. The tubes were then heated in boiling water for 60 min to dissolve the KBPb precipitate. The supernatant was separated by centrifugation for K determination after dilution. Similarly, boiling HNO₃-K was determined by extracting 2.5 g of soil with 25 mL 1.0 mol L⁻¹ boiling HNO₃ for 10 min and filtering through filter paper. The concentration of K in plant and soil samples was determined using flame photometer.

Relative grain yield (RGY) was calculated using equation (1).

\[
\text{RGY} = \frac{\text{Grain yield of treatment without K fertilizer}}{\text{Grain yield of treatment with K fertilizer}} \times 100
\]

Relative K uptake by crops (RKU) was calculated using equation (2).

\[
\text{RKU} = \frac{\text{K uptake by crops of treatment without K fertilizer}}{\text{K uptake by crops of treatment with K fertilizer}} \times 100
\]

Pairs of matching RGY and corresponding soil-test K values for each site-year are represented by one point in figures. The abundance and deficiency indices of soil-test K for rice were calculated using the logarithmic equation models and the statistical Cate-Nelson method. According to the method of the reference and the actual situation of this study, soil-test K was categorized as low, medium, high and very high based on the corresponding relative yield values which were <85%, 85~90%, 90~95% and >95%, respectively.

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**Author Contributions**

J.L. and X.L. initiated and designed the research. Y.Z. and W.W. performed the experiments. X.L. and Y.Z. analyzed the data and wrote the paper. M.R.K. and R.C. revised and edited the manuscript.

**Additional Information**

**Competing Interests:** The authors declare no competing interests.

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