POTASSIUM RECALIBRATION FOR CORN IN NORTH DAKOTA AND SAMPLING TIME

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ABSTRACT

Potassium (K) fertilizer recommendations for corn (*Zea mays*, L.) are commonly guided by yield response calibrations to soil K levels; however, sample drying and time of soil sampling have been shown to affect the amount of extractable K. Potassium rate trials were established at 13 locations in southeastern North Dakota during 2015 with objectives to evaluate soil K testing methods and their relationship with corn yield response to K fertilization and to assess temporal soil K variation. Soil K test methods evaluated were NH4OAc on air-dry (DK) and field-moist (MK) soil, sodium tetraphenylboron, and an ion-exchange resin. Sample drying significantly increased NH4OAc-extractable K by a factor of 1.26 on average (range: 0.8-2.4). Both DK and MK were affected by sampling date at 12 sites where soil K highest in spring and lowest in late summer, exhibiting a sinusoidal relationship. The seasonal difference in soil K was great enough to change soil test interpretation classes. Yield responses to K fertilization were observed at only three of seven sites with initial DK below the current 160 ppm DK critical level, and one site responded with DK above the critical level. Quadratic models of relative yield response regressed against soil K showed that DK and MK explained 7% and 19% of yield variability, respectively; however, neither DK nor MK was a strong predictor of yield response. No significant relationship with yield response could be identified for the sodium tetraphenylboron or ion-exchange resin methods. An adequate soil K test for corn in North Dakota remains to be identified.

INTRODUCTION

Fertilizer recommendations are commonly guided by yield response calibrations to soil test levels. The current potassium (K) fertilizer recommendations for corn (*Zea mays*, L.) in North Dakota are based on limited research conducted in the late 1970s and early 1980s; this was a time when soil test K (STK) levels greater than 200 ppm were nearly ubiquitous (Dahnke et al., 1982, 1992) and average state corn yields modestly ranged from 50-90 bushels/acre (USDA-SRS, 1982). The native soil K fertility was sufficient for most crop K requirements with the exception of particularly sandy soils or high K requiring crops like potato (Zubriski and Moraghan, 1983). In 1980, 3% of soil tests in North Dakota indicated medium or lower STK levels (<129 ppm) (Nelson, 1980). Decades of intensive corn and soybean production, particularly in the eastern part of North Dakota, without maintenance K fertilizer applications has resulted in K mining and more low STK values are being reported. In 2010, 17% of soil tests in North Dakota had STK levels below the critical level of 160 ppm (Fixen et al., 2010). Given lower STK values becoming more common, a recent spike in K fertilizer price after decades of price stability, and higher corn yields due to improved genetics and agronomic practices, a reassessment of the soil K test and K fertilizer recommendations in North Dakota has been prompted.

The current North Dakota potassium recommendation for corn is based on the formula: K_2O rate (pounds per acre) = $(1.1660-0.0073 * STK) * YP$, where STK = soil test potassium and YP $=$ yield potential or yield goal (Franzen, 2013). When the nitrogen recommendations for corn in North Dakota were revised, the yield goal concept was abandoned for an economically optimum yield. The K recommendations were concurrently simplified to general recommendations based on soil test level categories with the critical level set to 160 ppm K (Franzen, 2014).

The standard soil K test method for soils in the North Central region of the United States employs a neutral ammonium acetate extraction solution (1.0 M NH4OAc at pH 7.0) on air-dried or 40°C oven-dried soil (Warnacke and Brown, 2012). This method, however, has come under greater scrutiny because soil sample drying (Barbagelata and Mallarino, 2012) and time of soil sampling (Vitko et al., 2009; Franzen, 2011) have been shown to affect STK results. Moreover, yield responses to K fertilizer application may be inconsistent on both low and high STK soils (Rakkar et al., 2015). Recognition of these factors complicates the interpretation of STK results for fertilizer recommendations.

For decades, soil sample drying has been shown to increase exchangeable K (Attoe, 1947; Luebs et al., 1956; Scott and Smith, 1957; Grava et al., 1961). Additionally, the drying of soil samples with high STK or smectitic mineralogy may lead to K fixation and decrease of exchangeable K (Scott et al., 1957; Cook and Hutcheson, 1960; Sucha and Siranova, 1991). These observations have been reinvestigated more recently in the North Central region to reconcile the effect of sample drying on STK from its field-moist condition. The relationship between exchangeable K on air-dry and field-moist soil samples is often not predictable and varies with clay content, clay mineralogy, soil moisture, and STK level (Vitko et al., 2009; Barbagelata and Mallarino, 2012; Rakkar et al., 2015). In Iowa, STK on field-moist soil exhibited a superior predictive relationship to corn yield response to K fertilization than on airdry soil (Barbagelata and Mallarino, 2012). Indeed, the Iowa soil fertility recommendations now include the field-moist soil K test (Mallarino et al., 2013). However, neither the air-dry nor the field-moist soil K tests were shown to be adequate predictors of corn yield response to K fertilization in North Dakota (Rakkar et al., 2015).

Additional methods to assess plant-available K have been investigated when the standard 1.0 M NH4OAc extraction for exchangeable K has been found insufficient. These novel methods often attempt to assess a portion of nonexchangeable K that may become plant-available in addition to solution and exchangeable K. Sodium tetraphenylboron, which has the ability to extract nonexchangeable interlayer-K from 2:1 clays (Cox et al., 1996), was a superior predictor of plant K uptake in wheat compared to NH4OAc, especially in illitic soils (Cox et al., 1999). However, NaBPh₄ had no advantage over NH₄OAc to estimate plant K uptake of corn in montmorillonitic soils due to their diminished capacity to fix K (Schindler et al., 2002). The soils of North Dakota are generally dominated by smectitic mineralogy; however, some soils with mixed mineralogy contain appreciable illite where the NaBPh₄ extraction may be justified. Ionexchange resins have also been explored for their ability to extract nonexchangeable K. Some have found that resins offer a promising relationship with plant K uptake (Rahmatullah and Mengel, 2000); however, others have produced less convincing results (Skogley and Haby, 1981).

Soil K levels are known to vary between years and within a year. Intra-seasonal temporal variation in STK levels has been noted by numerous researchers (Luebs et al., 1956; Childs and Jencks, 1967; Liebhardt and Teel, 1977; Vitko et al., 2009; Rakkar et al., 2015). Over a nine year soil sampling period from 1986 to 1994 in Illinois, Peck and Sullivan (1995) observed a cyclical variation in STK with low levels in late summer and high levels in midwinter, albeit great variability was observed in STK levels throughout that time. They attributed the STK increase over fall and winter to soil moisture content and release of inter-layer K through drying and freezing. Franzen (2011), using the data of Peck and Sullivan (1995), constructed a time-series unobserved components model (*proc UCM* in SAS 9.4) that identified a seasonal variation component in STK whose periodicity paired with the seasonal variation component in soil moisture level. In Mississippi, the temporal variation in STK followed a comparatively similar sinusoidal pattern between years (Oldham et al., 2013, 2015). In laboratory experiments exploring the effect of moisture content on exchangeable K, Luebs et al. (1956) noted a seasonal relationship between exchangeable K and the seasonal change in vapor pressure in the laboratory where soil samples were kept. Identification of a seasonal variation component related to soil moisture in North Dakota may improve the interpretation of STK in relation to its sampling time.

The objective of this study was to revise the K fertilizer recommendation for modern corn production in North Dakota through: (i) evaluation of the NH4OAc extraction method on air-dry and field-moist soil as well as novel soil tests including NaBPh₄ and an ion-exchange resin, (ii) assessment of STK relationship with yield response, and (iii) assessment of STK changes during the growing season. At the time of writing this proceedings paper, the 2016 corn sites had yet to be harvested; therefore, only the 2015 results will be presented.

MATERIALS AND METHODS

Field study

On-farm K rate trials were established in southeastern North Dakota at thirteen locations in 2015. The experiments were organized using a randomized complete block design with four replications and six K rate treatments on corn and one non-fertilized fallow treatment to assess soil K changes without plant uptake. The K treatments were 0, 30, 60, 90 120, and 150 lb K_2O acre⁻¹ applied as fertilizer-grade potassium chloride (0-0-60) granules hand-broadcasted prior to planting. All sites received broadcast application of 100 lb acre⁻¹ pelletized gypsum to reduce potential S deficiencies in the K trials. The experiment unit size was 10 ft by 30 ft. Fertilizer treatments were incorporated shallowly by farmer cooperators, except on no-till sites. The farmer cooperators planted corn and applied herbicides and other inputs on experiment areas when they conducted those activities on the rest of the field. Initial soil samples were collected from nonfertilized check and fallow plots from 0- to 6-inch and 6- to 12-inch depths and thereafter collected biweekly until harvest from the 0- to 6-inch depth only. Six soil cores were taken from each plot.

After physiologic maturity, corn grain was hand-harvested as whole corn ears from one interior 10-foot row, shelled using an Almaco corn sheller (Almaco, Nevada, Iowa), weighed for grain yield, and measured for grain moisture and test weight using a GAC 500 XY moisture tester (Dickey-John Corp., Auburn, Illinois). Grain yield was corrected to 14.5% grain moisture.

Soil and tissue analysis

Soil samples were hand-homogenized and split into two subsamples. One subsample was air-dried following the procedure suggested for the North Central region (Gelderman and Mallarino, 2012), ground to pass through a 2-mm sieve, and analyzed for NH₄OAc-extractable K (Warnacke and Brown, 2012). A 2-g air-dry soil sample was extracted with 20 mL 1M NH4OAc at pH 7.0 and shaken for 5 minutes in Erlenmeyer flasks. The extract was filtered through Whatman No. 2 filter paper (GE Healthcare Bio-Sciences, Pittsburgh, Pennsylvania) and analyzed for extractable K by atomic absorption spectroscopy (AAS). The other subsample kept at field-moisture was stored in a plastic re-closeable bag and refrigerated until analysis. The field-moist sample was prepared following the direct sieving procedure described by Gelderman and Mallarino (2012). Field-moist soil was passed through a 2-mm sieve, and soil water content was immediately after determined by drying a 6 g moist subsample to air-dryness and constant weight. A 2-g air-dry equivalent mass of field-moist soil was measured and extracted with 20 mL 1M NH4OAc at pH 7.0, thereafter, following the same analysis procedure described for the airdry K method.

Resin-extractable K was determined using a mixed-bed, cation- and anion-exchange resin capsule (UNIBEST Inc., Walla Walla, WA). A 30-g air-dry equivalent mass of 2-mm sieved, field-moist soil was measured and incubated with a resin capsule and 30 mL deionized water for 7 days at constant 20 degrees C. After the incubation period, the resin capsule was washed with deionized water to remove attached soil and leached with 50 mL 2M HCl using a manual slowdrip leaching apparatus (UNIBEST Inc., Walla Walla, WA). The resin capsule leachate was analyzed for resin-extractable K using AAS. The sodium tetraphenylboron extraction method (Cox et al., 1999) was used to determine the most reactive and total nonexchangeable K fractions using 5-minute and 7-day extraction times, respectively. The cation exchange capacity was estimated (ECEC) by summation of extractable Ca, Mg, K, Na, and neutralizable soil acidity on air-dry soil (Warnacke and Brown, 2012). Quantitative mineral identification and clay speciation was conducted by Activation Laboratories Ltd. (Ancaster, Ontario, Canada) on a composite field-moist soil sample from each site.

Data analysis

Data were analyzed for individual site years using SAS 9.4 (SAS Institute, Inc.) to determine if K fertilizer responses were significant using *proc glm*. Relative grain yield was calculated by dividing the plot yield by the maximum plot yield at each experiment site. Relative grain yield and STK data were regressed to fit liner and quadratic models for each soil sampling date using *proc glm* and *proc nlin*. Soil K data was analyzed as a randomized complete block design with split-plot in time arrangement using *proc mixed* to determine if changes in soil K were significant and regressed to fit sinusoidal models over time using *proc nlin*; the time modeling did not use *proc ucm* as employed by Franzen (2011) because soil samples in this study were collected during only one sampling season cycle.

RESULTS AND DISCUSSION

Effect of sample drying on soil potassium

The initial soil K levels of 13 K trials in North Dakota revealed a wide range of STK values ranging from 87-444 ppm air-dry soil K (DK) in late May (Table 1). The correlation between DK and field-moist soil K (MK) was strong across sites and sampling times $(r^2=0.89, p<0.01,$ n=868). The ratio of DK/MK regressed against MK showed that DK was usually greater than MK, and the relative amount of K released upon drying was greater for low K soils (Figure 1). The obverse scenario of regressing the MK/DK ratio against DK does not exhibit any recognizable relationship (data not shown), indicating that MK is the independent factor in the sample drying effect. The average DK/MK ratio was 1.26 (range: 0.8-2.4). Soil moisture was positively correlated with the sample drying effect (i.e., DK/MK), but the relationship was weak

 $(r^2=0.10, p<0.01, n=868)$. Within the DK/MK relationship, distinct trends for individual soils or groups of soils could be identified within the general trend indicating that other soil or site characteristics influenced STK upon drying. Such DK/MK trends could be visually grouped by taxonomic soil texture family in fine to fine-silty soil textures (Leonard N, Fairmount 1, and Prosper) and sandy to coarse- and fine-loamy soil textures for the remaining sites.

Effect of sampling time

Soil test K measured throughout the growing season from late May to early September revealed that STK levels did change from spring to fall. The effect of sampling time on both DK and MK was statistically significant ($p<0.05$) at 12 of 13 sites, the Dwight site omitted (data not shown). Generally, STK was highest in late May or early June and reaching its lowest value in late summer before slightly increasing in fall, agreeing with the observations of Peck and Sullivan (1995) in Illinois. Regression analysis showed that the relationship between STK and sampling time could be modelled by a sinusoidal function. Sinusoidal relationships of MK and sampling time were established for 11 of 13 sites where individual sites had p-values<0.1 and r^2 values ranged from 0.74 – 0.99. The seasonal difference in MK from spring to fall ranged from 44-88 ppm MK, which is great enough to change STK interpretation classes just depending on a spring versus fall soil sampling time. The 11 sinusoidal sites were summarized into one function that indicated the periodicity of the sinusoidal relationship was consistent across low and high MK sites (Figure 2). The seasonal decrease in STK was influenced by plant K uptake and seasonal soil moisture status. The study region received limited rainfall in late summer and early fall of 2015, which exacerbated the effect of K fixation on reducing STK later in the growing season. The sinusoidal relationship between DK and sampling time was only significant $(p<0.1)$ at six of 13 sites. Due to the sample drying effect, there was greater variability in DK, which obscured the relationship between DK and sampling time. Although this analysis only encompasses data from 2015, it provides an enticing avenue to resolve the role of sampling time when interpreting STK information. Analysis of 2016 STK data will indicate if the relationship can be replicated between years.

Yield response to K fertilization

The wide range of STK in the K trials encompassed the current 160 ppm DK critical level, which should lend itself useful prediction of a yield response to K fertilization. In contrast, corn yield increased to K fertilization at only three of seven sites with DK below the 160 ppm critical level (Table 2). Moreover, one site with DK above the 160 ppm critical level (Fairmount 1) had a yield decrease at the high 150 lb K_2O /acre rate (Table 2). The established critical level for the DK test predicted K response less than half of the time.

Quadratic models of relative yield regressed against DK and MK from soil samples collected in late May (Figure 3) and early September (Figure 4) showed that MK was a superior predictor of yield response to K fertilization over DK as observed by Barbagelata and Mallarino (2012). The time of year at sampling did not affect the relationship between relative yield and STK. The DK models were not statistically significant, which was probably due to additional variability in DK caused by the sample drying effect. The MK model coefficients of determination (r^2 =0.19 and 0.22) show that MK was not a strong predictor of yield response for K recommendations, and much unexplained variability still exists. Furthermore, percent K saturation of the estimated cation exchange capacity (ECEC) had no significant linear $(r^2=0.14,$ $p<0.28$) or quadratic (r^2 =0.25, $p<0.37$) relationship with relative yield. These poor relationships

between NH4OAc-extractable K tests (i.e., exchangeable K) and relative yield response suggest that other factors contribute to plant-available K supply. Relative yield information from Leonard N, Leonard S, and Valley City were excluded from regression analysis because of non-K nutrient deficiencies or poor plant population.

Sodium tetraphenylboron-extractable K (TBK) for the 5-minute and 168-hour extractions was considerably higher than DK at all sites, indicating that NaBPh₄ was able to extract nonexchangeable K forms (Table 1). Nevertheless, the 5-minute TBK, which contains the most readily available portion of nonexchangeable K, had no significant linear $(r^2=0.12, p<0.33)$ or quadratic (r^2 =0.12, p<0.63) relationship with relative yield. Furthermore, the 168-hour TBK had no significant linear $(r^2=0.20, p<0.19)$ or quadratic $(r^2=0.27, p<0.34)$ relationship with relative yield. The NaBPh4 extraction with its ability to extract a portion of nonexchangeable K did not provide a better estimate of plant-available K than $NH₄OAc-extractable K$. Yet, the NaBPh₄ extraction may provide a useful estimation of nonexchangeable K as a tool in future K management (Carey et al., 2011). Much like the NaBPh₄ extractions, resin-extractable K had no significant linear (r^2 =0.24, p<0.15) or quadratic (r^2 =0.24, p<0.38) relationship with relative yield. Resin-extractable K was positively correlated with MK $(r^2=0.58, p<0.01, n=13)$ and percent K saturation (r^2 =0.32, p<0.04, n=13), but negatively correlated with estimated cation exchange capacity (r^2 =0.52, p<0.01, n=13). This suggests that other cations competed for exchange sites on the resin in high ECEC soils, which would reduce its ability to extract nonexchangeable K forms in these soils. Both of the alternative K extraction methods investigated were not better predictors of yield response than NH4OAc-extractable K; however, much opportunity still remains in the search for a better soil K test.

Inconsistent yield responses to K fertilizer were also observed on low and high STK soils by Rakkar et al. (2015). A post-hoc mineralogical analysis of these sites indicated that a number of non-responsive, low STK sites had substantial amounts of K-feldspar and/or illite (Franzen, 2015). Potassium-feldspars may serve as a large source of plant-available K if plant demand is high (Martin and Sparks, 1983; Sadusky et al., 1987; Parker et al., 1989), which may explain the lack of demand on high K-feldspar soils. Furthermore, the potassium kinetics of vermiculite and illite are much slower than kaolinite and montmorillonite and may take hours to reach equilibrium with the soil solution (Jardine and Sparks, 1984). Therefore, STK of soils with illitic mineralogy may be grossly underestimated by a 5-minute extraction with NH4OAc.

Mineralogical analysis of soils from the 2015 K trials showed that some sites also contained appreciable amounts of K-feldspar, muscovite, and/or illite (Table 3). All soils contained 5-10% K-feldspar, and responsive and non-responsive sites were distributed throughout that K-feldspar range. The illite content of the clay fraction was more variable between responsive and nonresponsive sites, and its role in K availability warrants further analysis in this study. Potassium release from these mineral K or nonexchangeable K forms may provide enough K to satisfy plant K requirements in low STK, non-responsive soils; however, the kinetics of K dynamics for such soils in North Dakota remains to be elucidated.

SUMMARY

The standard soil K test in the North Central region employs a 1.0 M neutral ammonium acetate extraction on air-dry or oven dry soil; however, the method has come under scrutiny because sample drying and time of soil sampling have been shown to affect the amount of extractable K. Soil sample drying was shown to significantly increase NH4OAc-extractable K from its field-moist condition by a factor of 1.26 on average. The ratio of DK/MK varied with MK level, but other soil factors contributed to the degree of K release or fixation upon drying. The time of soil sampling did affect soil test K levels during the growing season. Soil K was highest in spring and reached its lowest values in late summer. The degree of soil K change was great enough to change soil test level interpretation classes. At most sites, the change in soil K could be described by a sinusoidal function over time during the growing season. Variations in seasonal soil moisture will likely challenge the robustness of the sinusoidal model.

The current soil test critical level of 160 ppm DK only predicted corn grain yield response to K fertilizer less than half of the time. Relative yield response could only be significantly related to MK; yet, the relationship was not adequate for confident K fertilizer recommendations. The DK test was not significantly related to yield response, likely due to increased variability from the sample drying effect. Sodium tetraphenylboron and resin extraction methods, which have the ability to assess a plant-available portion of nonexchangeable K, also had no significant relationship to yield response to K fertilization. An adequate determination procedure for plantavailable K in North Dakota soils for the guidance of K fertilizer recommendations remains to be identified.

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TABLES AND FIGURES

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Site	Site	DK ⁺	MK	TBK	TBK	RK	K sat.	ECEC			
	abbr.			5 min	168h						
				ppm			$\%$	meq $/100$ g			
Absaraka	AB	108	102	235	713	17.2	2.2	15.5			
Arthur	AR	90	84	282	1255	8.3	1.0	33.8			
Barney	B	160	199	384	2456	22.1	1.6	31.5			
Casino	C	87	103	284	1206	13.0	2.1	20.1			
Dwight	D	109	101	249	1293	12.3	1.4	28.4			
Fairmount 1	F1	169	143	497	2607	7.8	1.8	31.7			
Fairmount 2	F2	100	82	250	1157	7.2	0.9	36.3			
Leonard N_{+}^{+}	LN	444	428	1146	3475	79.5	4.1	29.1			
Leonard S	LS	198	201	257	709	87.1	4.6	9.5			
Milnor	M	134	129	284	1160	13.8	1.4	38.2			
Prosper	\mathbf{P}	163	143	548	2637	13.7	2.1	28.8			
Valley City	$\overline{\mathsf{V}}$	203	235	551	2195	25.3	2.8	17.1			
Walcott	W	111	135	223	707	21.7	2.4	11.5			

Table 1. Soil potassium levels using different extraction methods for 13 potassium rate trials sampled in late May 2015.

† DK and MK, NH4OAc-extractable K on air-dry and field-moist soil; TBK, tetraphenylboronextractable K; RK, resin-extractable K; K sat., K saturation of ECEC; ECEC, estimated cation exchange capacity.

‡ Leonard N was first sampled in early June.

Table 2. Corn grain yield response to K fertilization.

† Within rows, treatment means followed by the same letter are not significantly different. ‡ LSD alpha-value changed to 0.1 significance level.

		Whole soil	$<$ 2 _µ m-fraction				
Site	Quartz			Plagioclase K-feldspar Muscovite/illite	Smectite		Illite Kaolinite
AB	42.0	28.4	9.9	1.8	84	14	$\overline{2}$
AR	37.9	27.5	9.5	3.0	85	12	3
B	36.5	18.3	6.3	3.8	79	16	5
\mathcal{C}	41.6	22.7	6.4	2.6	85	12	3
D	45.8	21.9	6.0	2.3	82	15	3
F1	38.5	15.4	5.6	3.0	87	10	3
F2	38.2	18.3	7.4	1.9	79	14	7
LN	33.4	21.6	6.9	6.6	70	25	5
LS	52.4	17.8	5.5	\triangle IDL \dagger	52	41	7
M	39.7	17.6	8.6	3.4	74	20	6
P	34.0	17.4	9.2	3.6	83	14	3
V	36.5	18.2	5.6	1.7	65	30	5
W	39.4	20.7	6.2	1.8	47	48	5

Table 3. Soil mineral composition for K-bearing and other relevant minerals.

† <IDL, below instrument detection limit

Figure 1. Relationship between the ratio of air-dry soil K (DK) and field-moist soil K (MK) regressed against the field-moist soil K (MK) value.

Figure 2. Relationship between field-moist soil K (MK) and sampling time (fortnight of year, FOY) fit to a sinusoidal regression model (MK = $111.3 + 28.65 * sin(0.6431 * FOY + 1.1791)$, r² $= 0.21$, p<0.01) for 11 of 13 combined potassium rate trials from 2015.

Figure 3. Relationship between soil test potassium (STK) from soil samples collected in late May as air-dry soil K (DK) and field-moist soil K (MK) and the relative grain yield (RY) of check plots to the highest plot yield in potassium rate trials from 2015.

Figure 4. Relationship between soil test potassium (STK) from soil samples collected in early September as air-dry soil K (DK) and field-moist soil K (MK) and the relative grain yield (RY) of check plots to the highest plot yield in potassium rate trials from 2015.

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