Published April 29, 2019

Soil Fertility & Plant Nutrition

Potassium Requirements for Corn in North Dakota: Influence of Clay Mineralogy

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Due to initially high soil test K values, K soil test correlation and calibration for corn in North Dakota has previously not been intensely investigated. Potassium fertilizer rate experiments were conducted on 25 sites from 2014 to 2016. The previously published soil test K critical value of 150 mg kg^{-1} predicted crop response correctly at 16 of the sites. Alternative soil test methods, including a resin-based extraction at two timings, sodium tetraphenylboron extractions at two timings, and 1 mol L^{-1} NH_4OAc extraction using moist soil were conducted; however, the currently used 1 mol L^{-1} ammonium acetate extraction using dry soil was most predictive. Mineral analysis of soil from all sites was determined for potassium feldspar content of whole soil, and clay species, particularly smectite, illite, and kaolinite, were determined on the clay fraction. Cluster analysis revealed that a smectite/illite ratio of 3.5 separated the sites into two unique K response data sets. Sites with a smectite/illite ratio >3.5 had a K critical level of ~200 mg kg^{-1}, whereas sites with a smectite/illite ratio <3.5 had a K critical level of ~130 mg kg^{-1}. For soils with K soil tests between 130 and 200 mg kg^{-1}, consideration of clay chemistry improves the predictability of crop yield response with K fertilization.

Abbreviations: AAS, atomic absorption spectroscopy; CEC, cation exchange capacity; DK, ammonium acetate–extractable K from air-dry soil; MK, ammonium acetate–extractable K from field-moist soil.

Core Ideas

- **Use of dry soil K soil test was most predictive of corn response in North Dakota.**
- **Consideration of clay chemistry increased the prediction of yield response by the K soil test.**
- **A smectite/illite ratio of 3.5 separated the sites into one requiring a higher critical K soil test value and one with a lower critical K value.**

Historical application of K fertilizer in North Dakota has been minimal and infrequent because soil K levels were generally high (i.e., >150 mg K kg^{-1}) through the 1990s. In 1980, 3% of soil samples tested in North Dakota indicated medium or lower exchangeable K levels (<130 mg K kg^{-1}) (Nelson, 1979). Grain K export has far exceeded fertilizer K added to North Dakota soils for decades (Cassel et al., 1973; Dahnke et al., 1986; IPNI, 2014). In eastern North Dakota, the recent cropping system change to corn (*Zea mays* L.)- and soybean [*Glycine max* (L.) Merr.]-intensive crop rotations from wheat (*Triticum aestivum* L.)-dominated rotations has exacerbated grain K export (IPNI, 2014). Increased grain K export without maintenance K fertilizer application has resulted in accelerated soil K removal, and 16% of soil tests were below the critical level in a 2015 soil test survey (IPNI, 2016).

Although the total K content of soils is relatively high compared with other nutrient elements (Sparks and Huang, 1985), the amount of soil K readily available to plants is much lower. For edaphic purposes, soil K has been classified into four groups based on their relative availability to plants, in order of decreasing availability: solution K, exchangeable K, nonexchangeable K, and structural K (Sparks and Huang, 1985). Many K recommendations for crops in the United States are based on soil solution K and a portion of exchangeable K determined with an extraction solution (Warncke and Brown, 2012). Some K recommendations are based on

Soil Sci. Soc. Am. J. 83:429–436
doi:10.2136/sssaj2018.10.0376
Received 9 Oct. 2018.
Accepted 21 Dec. 2018.
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the "balance" of K to other cations, although this approach has been rejected by most in the soil science community (Kopittke and Menzies, 2007). However, there is evidence that soil minerals (particularly K-feldspars, K-micas, and 2:1 clays) are also important in the K nutrition of plants. Whereas soil fertility textbooks often indicate that nonexchangeable K or interlayer K release from 2:1 minerals and mineral K release from K-feldspars contributes little K to plants in a given growing season, requiring many years to render plant-available K (Tisdale et al., 1985), laboratory studies and subsequent field research have indicated that soil solution equilibrium with interlayer K and mineral K release from K-feldspars can be reached in hours or a few days (Sparks and Huang, 1985). The contribution of nonexchangeable K or interlayer K to crop yield has been noted in several studies (Brar et al., 2016; McLean and Watson, 1985; Swarup and Chhillar, 1986; Zhang et al., 2011).

The standard soil K test method for soils in the north central region of the United States uses a neutral ammonium acetate extraction solution (1.0 M NH_4OAc at pH 7.0) on soil that has been air-dried or oven-dried at temperatures <40°C (Warncke and Brown, 2012). This method, however, has come under scrutiny because soil sample drying prior to laboratory analysis (Barbagelata and Mallarino, 2013) and date of soil sampling (Franzen, 2011; Vitko et al., 2009) have been shown to affect soil test K results. The relationship between exchangeable K from dried and field-moist soil samples is often not predictable and varies with clay content, clay mineralogy, soil moisture, and soil test K level (Barbagelata and Mallarino, 2013; Martins et al., 2015; Vitko et al., 2009). In Iowa, NH_4OAC-extractable K from field-moist soil exhibited a superior predictive relationship to corn yield response to K fertilization than using oven-dry soil (Barbagelata and Mallarino, 2013).

In soils where nonexchangeable K is a significant source of plant available K (e.g., illitic or vermiculitic soils), NH_4OAc may provide an inadequate assessment of plant-available K (Cox et al., 1999; Rouse and Bertramson, 1950). Sodium tetraphenylboron ($NaBPh_4$) extraction, which has the ability to remove nonexchangeable K from 2:1 phyllosilicates (Cox et al., 1996), was identified as a superior predictor of wheat K uptake over NH_4OAc in illitic soils from Indiana (Cox et al., 1999). Ion-exchange resins, which have a cation exchange capacity (CEC) that is too high to serve as a sink for solution K to facilitate nonexchangeable K release (Helmke and Sparks, 1996), have been explored for their ability to mimic plant K uptake in being dependent on solution K activity and rate of diffusion (Schaff and Skogley, 1982; Skogley and Schaff, 1985). Ion-exchange resins have been calibrated for crops in Brazil on low exchangeable K, 1:1 clay soils (Cantarella et al., 1998); however, calibration data are lacking for resins on soils containing 2:1 clays, whose K exchange kinetics are much slower than 1:1 clays (Jardine and Sparks, 1984; Nilawonk et al., 2008).

The objective of this study was to evaluate K fertilizer recommendations for corn production in North Dakota through (i) evaluation of various laboratory soil testing methods and (ii) assessment of soil test K relationship with corn yield response to K fertilization to establish soil test K critical levels.

MATERIALS AND METHODS

Between 2014 and 2016, on-farm fertilizer K rate experiments in corn were conducted in two phases, with initial trials in 2014 and expanded trials in 2015 to 2016, at 25 nonirrigated locations in southeastern North Dakota (Table 1). The experiments were organized as a randomized complete block design with four replications and six K rate treatments. Experiment unit size was 3.0 m by 9.1 m with a 1.5-m alleyway between each replication. The K treatments were 0, 28, 56, 84, 112, and 140 kg K ha^{-1} applied as fertilizer-grade KCl (500 g K kg^{-1}) granules hand-broadcasted prior to planting; site Milnor 2014 received K applications of 0, 56, 112, 168, 224, and 280 kg K ha^{-1}. To reduce non-K nutrient deficiencies, each location received uniform broadcast N as Limus-treated urea (450 g N kg^{-1}) [Limus rate was 2.84 L 907 kg^{-1}; active ingredients are 0.183 g L^{-1} N-(n-butyl) thiophosphoric triamide and 0.061 g L^{-1} N-(n-propryl) thiophosphoric triamide] (BASF Corporation, Research Triangle Park, NC) and P as fertilizer-grade monoammonium phosphate (110 g N kg^{-1}, 230 g P kg^{-1}) following North Dakota corn fertility guidelines (Franzen, 2014) based on preplant soil tests (Table 2) as well as 112 kg ha^{-1} pelletized gypsum (180 g S kg^{-1}). Fertilizer treatments were shallowly incorporated (5–8 cm) by farmer-cooperators in the spring except on no-till sites. Farmer-cooperators chose corn hybrid, planted corn, and applied herbicides on experiment alareas when they conducted those activities on the rest of the field (Table 3). Soil samples were collected from nonfertilized check plots from 0- to 15-cm soil depth; six soil cores were obtained from each experimental unit.

After physiologic maturity, corn grain was hand-harvested as whole corn ears from one interior 9.1-m row, shelled, and measured for grain moisture and test weight. Grain yield was corrected to 145 g kg^{-1} grain moisture.

Soil samples were hand-homogenized and split into two subsamples. One subsample was air-dried following the procedure recommended for the North Central Region of the United States (Gelderman and Mallarino, 2012), ground to pass through a 2-mm sieve, and analyzed for NH_4OAc-extractable K (Warncke and Brown, 2012). A 2-g air-dry soil sample was extracted with 20 mL 1 M NH_4OAc at pH 7.0 and shaken for 5 min in Erlenmeyer flasks. The extract was filtered through Whatman No. 2 filter paper (GE Healthcare Bio-Sciences, Pittsburgh, PA) and analyzed for extractable K by atomic absorption spectroscopy (AAS) (Model 200A; Buck Scientific, Norwalk, CT). The other subsample was kept at field-moisture, stored in a plastic recloseable bag, and refrigerated at 4°C 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 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 1 mol L^{-1} NH_4OAc at pH 7.0 following

Table 1. Location and soil characterization of experiment sites.

Site	Year	Latitude	Longitude	Series	Soil classification†
Arthur	2014	47.063°	97.134°	Glyndon	coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Buffalo	2014	46.920°	97.422°	Lankin	fine-loamy, mixed, superactive, frigid Pachic Hapludolls
Fairmount	2014	45.971°	96.619°	Gardena	coarse-silty, mixed, superactive, frigid Pachic Hapludolls
Gardner	2014	47.166°	97.051°	Galchutt	fine, smectitic, frigid Vertic Argialbolls
Milnor	2014	46.276°	97.467°	Emden	coarse-loamy, mixed, superactive, frigid Pachic Hapludolls
Page	2014	47.161°	97.367°	Swenoda	coarse-loamy, mixed, superactive, frigid Pachic Hapludolls
Walcott E	2014	46.495°	96.885°	Wheatville	coarse-loamy over clayey, mixed over smectitic, superactive, frigid Aeric Calciaquolls
Walcott W	2014	46.588°	97.047°	Hecla	sandy, mixed, frigid Oxyaquic Hapludolls
Wyndmere	2014	46.261°	97.064°	Glyndon	coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Arthur	2015	47.111°	97.281°	Glyndon	coarse-silty mixed, superactive, frigid Aeric Calciaquolls
Barney	2015	46.241°	96.991°	Glyndon	coarse-silty mixed, superactive, frigid Aeric Calciaquolls
Casino	2015	45.951°	96.776°	Mantador	coarse-loamy, mixed, superactive, frigid Aquic Pachic Hapludolls
Dwight	2015	46.307°	96.819°	Gardena	coarse-silty, mixed, superactive, frigid Pachic Hapludolls
Fairmount 1	2015	46.009°	96.630°	Doran	fine, smectitic, frigid Aquertic Argiudoll
Fairmount 2	2015	45.979°	96.697°	Gilby	fine-loamy, mixed, superactive Aeric Calciaquolls
Leonard	2015	46.701°	97.266°	Kindred	fine-silty, mixed, superactive, frigid Typic Endoaquolls
Milnor	2015	46.272°	97.478°	Arveson	coarse-loamy, mixed, superactive, frigid Typic Calciaquolls
Prosper	2015	47.128°	97.114°	Kindred	fine-silty, mixed, superactive, frigid Typic Endoaquolls
Valley City	2015	46.834°	97.918°	Embden	coarse-loamy, mixed, superactive, frigid Pachic Hapludolls
Walcott	2015	46.509°	97.012°	Garborg	sandy, mixed, frigid Typic Endoaquolls
Absaraka	2016	46.979°	97.422°	Glyndon	coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Colfax	2016	46.399°	96.925°	Mantador	coarse-loamy, mixed, superactive, frigid Aquic Pachic Hapludolls
Gardner	2016	47.166°	97.051°	Galchutt	fine, smectitic, frigid Vertic Argialbolls
Lisbon	2016	46.464°	97.177°	Garborg	sandy, mixed, frigid Typic Endoaquolls
Valley City	2016	46.891°	97.917°	Swenoda	coarse-loamy, mixed, superactive, frigid Pachic Hapludolls

† United States Soil Taxonomy (Soil Survey Staff, 1999, 2014).

Table 2. Preplant soil test analysis for experiment sites.

		Soil test parameter†							
Site	Year	NO_3–N	P	K	pH	EC	OM	Clay	CEC
		kg ha^{-1}	—— mg kg^{-1} ——			dS m^{-1}	—— g kg^{-1} ——		cmol$_c$ kg^{-1}
Arthur	2014	15	10	170	7.6	0.26	31	145	23.1
Buffalo	2014	18	12	115	5.9	0.19	21	108	12.9
Fairmount	2014	23	10	140	7.4	0.30	27	155	19.9
Gardner	2014	10	13	110	7.9	0.09	22	113	12.5
Milnor	2014	9	18	110	7.6	0.43	22	73	14.1
Page	2014	20	12	200	6.2	0.48	24	100	14.9
Walcott E	2014	6	3	105	5.8	0.45	23	115	12.1
Walcott W	2014	10	16	80	5.8	0.10	15	45	10.6
Wyndmere	2014	20	8	100	7.9	0.27	23	115	15.6
Arthur	2015	36	7	125	8.1	0.34	27	200	27.2
Barney	2015	201	14	170	7.8	1.35	32	250	23.6
Casino	2015	67	6	120	8.1	0.34	27	180	12.5
Dwight	2015	52	10	110	8.0	0.57	23	250	23.1
Fairmount 1	2015	112	21	188	7.9	0.50	40	320	22.5
Fairmount 2	2015	90	9	118	8.0	0.87	30	260	29.4
Leonard	2015	334	16	380	5.8	0.39	55	450	19.9
Milnor	2015	62	7	117	8.1	0.26	39	150	29.4
Prosper	2015	66	9	205	8.0	0.57	42	450	17.4
Valley City	2015	76	8	201	6.9	0.25	25	260	12.4
Walcott	2015	49	32	108	5.4	0.10	13	120	10.0
Absaraka	2016	69	16	162	7.5	0.90	41	200	29.4
Colfax	2016	119	4	54	8.3	0.33	27	100	31.0
Gardner	2016	40	13	60	6.7	0.24	22	240	16.5
Lisbon	2016	41	6	78	7.9	0.27	17	100	21.9
Valley City	2016	77	22	226	6.4	0.19	22	120	25.6

† CEC, cation exchange capacity by summation; EC, 1:1 soil/water electrical conductivity; K, NH_4OAc-extractable K; NO_3–N, KCl extractable NO_3–N (0–60 cm); OM, organic matter; P, Olsen bicarbonate P; pH, 1:1 soil/water pH.

Table 3. Agronomic information for experiment sites.

Site	Year	Previous crop	Tillage†	Plant date	Hybrid‡	Population§
						plants ha^{-1}
Arthur	2014	soybean	CT	5/18	PS 11–91	74,300
Buffalo	2014	soybean	CT	5/15	D 36–30	86,100
Fairmount	2014	soybean	CT	5/23	GC 95–30	81,800
Gardner	2014	soybean	CT	5/18	NT 5B782	68,900
Milnor	2014	soybean	CT	5/17	P 9917	77,500
Page	2014	soybean	CT	5/25	R 2A550	74,300
Walcott E	2014	soybean	CT	5/30	D 36–30	78,900
Walcott W	2014	soybean	CT	5/23	D 39–07	77,500
Wyndmere	2014	soybean	CT	5/27	D 43–10	86,100
Arthur	2015	soybean	CT	4/25	D 36–30	68,100
Barney	2015	dry bean	ST	4/18	C 3899	73,200
Casino	2015	soybean	CT	5/1	R 4B953	74,100
Dwight	2015	soybean	CT	4/18	C 3899	70,200
Fairmount 1	2015	corn	CT	4/26	GC 95–33	68,000
Fairmount 2	2015	soybean	CT	5/2	P 9917RR	77,100
Leonard N	2015	soybean	CT	6/1	P 9366	78,400
Milnor	2015	soybean	CT	4/26	NS 9505	83,600
Prosper	2015	soybean	CT	4/28	P 9284R	73,600
Valley City	2015	wheat	NT	4/29	NK N17P-3110	41,700
Walcott	2015	soybean	CT	4/29	PF 76S92	79,400
Absaraka	2016	wheat	CT	5/3	PF 92G84	76,900
Colfax	2016	corn	CT	4/22	D 48–12	82,400
Gardner	2016	soybean	CT	4/30	NT 5B782	55,500
Lisbon	2016	soybean	CT	4/23	P 9284R-NM61	74,800
Valley City	2016	wheat	NT	5/5	NK N17P-3000	50,210

† CT, conventional till; CTnw, conventional till but not worked; NT, no-till; ST, strip-till.

‡ C, Cropland; D, Dekalb; GC, Gold Country; NS, Nuseed; NT, NuTech; P, Pioneer; PF, Peterson Farms; PS, ProSeed; R, REA.

§ Plant population at harvest.

the same analysis procedure described for the air-dry method (Warncke and Brown, 2012).

In 2015 to 2016, the laboratory study was expanded to include additional soil test methods. Sodium tetraphenylboron extraction, following the procedure of Cox et al. (1999), was used to determine the most reactive and total nonexchangeable K fractions using 5-min and 168-h extraction times, respectively. A 0.5-g air-dry soil sample was weighed into a 50-mL Folin-Wu digestion tube with 3 mL extraction solution (0.2 M $NaBPh_4$ + 1.7 M NaCl + 0.01 M EDTA). After the extraction period (5 min or 168 h), 25 mL of quenching solution (0.5 M NH_4Cl + 0.11 M $CuCl_2$) was added to arrest K extraction. Digestion tubes were placed in the digestion block at 150°C until precipitate dissolved completely (30–60 min). The suspension was diluted to 50 mL with deionized water, mixed, and left to settle. A 20-mL aliquot of the supernatant was transferred to 50-mL flasks, centrifuged, and acidified with three drops of 6 M HCl to prevent Cu^{2+} precipitation. The extract was diluted (1:10) with deionized water and analyzed for $NaBPh_4$ extractable K by AAS.

Resin- extractable K was determined using a mixed-bed, cation- and anion-exchange resin capsule (UNIBEST Inc., Walla Walla, WA) based on the design of Skogley (1992). 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 168 h at a constant temperature of 20°C. After the incubation period, the resin capsule was washed with deionized water to remove attached soil and leached with 50 mL 2 M HCl using a slow-drip leaching apparatus (UNIBEST Inc.). The resin capsule leachate was analyzed for resin-extractable K using AAS.

In 2014, CEC was determined by the sodium-ammonium saturation-displacement method (U.S. Salinity Laboratory Staff, 1954). In 2015 to 2016, estimated CEC was determined by summation of extractable cations (Ca, Mg, K, Na) and neutralizable soil acidity (Warncke and Brown, 2012). Semiquantitative mineral identification and clay speciation was conducted using the Rietveld method (Rietveld, 1969) by Activation Laboratories Ltd. (Ancaster, Ontario, Canada) on a composite field-moist soil sample from each site; their diagnostic procedure did not distinguish between smectite species (Borchardt, 1989). Soil clay content was determined by hydrometer method (Gee and Or, 2002).

The statistical software SAS version 9.4 was used for analysis (SAS Institute, 2013). Corn yield response to K fertilizer treatments was analyzed with PROC GLM ($p < 0.05$). Relative grain yield was calculated by dividing the average yield of the 0 kg K_2O ha^{-1} treatment by the maximum average treatment yield at each experiment site. Relative grain yield of the unfertilized treatment and extractable K by various soil test K methods were regressed to fit linear-plateau, quadratic-plateau, quadratic, and exponential models using PROC NLIN.

Mineralogical analyses were related to soil test K and relative grain yield using PROC PRINCOMP to identify principal components between yield response and mineralogical variables. Experiment sites were partitioned by the smectite/illite ratio of their clay-sized fraction using PROC CLUSTER and Ward's minimum variance method (Ward, 1963), and clusters were verified with PROC GLM; this identified two smectite/illite ratio clusters. Because the variance of mineralogical variables may be unequal, variables were standardized to a Z-score using PROC STANDARD prior to cluster analysis. For each site cluster, relative grain yield and NH_4OAc extractable K from air-dry soil were regressed to fit linear-plateau and quadratic-plateau models using PROC NLIN.

RESULTS AND DISCUSSION

Potassium fertilizer recommendations for corn in North Dakota prior to 2014 were based on the soil test K critical level of 150 mg kg^{-1} (Franzen, 2014). A yield increase to K fertilization is more likely using these recommendations for soils testing <150 mg kg^{-1}. Above the critical level, a yield increase was not likely. In the 2012 and 2016 experiments, yield responses were correctly predicted by the 150 mg kg^{-1} critical level at 16 of 25 sites (Table 4). For responsive sites, the K fertilizer rate required to achieve maximum yield ranged between 84 and 140 kg K ha^{-1}. Corn grain yield increase was correctly predicted for 11 sites <150 mg kg^{-1}. Furthermore, five sites with soil test K <150 mg kg^{-1} did not have yield increases to K fertilization,

Table 4. Corn yield response to K fertilization and soil test K prediction accuracy of experiment sites.

Site	Year	Soil test K	Was a yield increase predicted by soil test K?†	Yield increase from K fertilization	Maximum yield§	K rate to achieve maximum yield
		mg kg^{-1}		——— kg ha^{-1} ———		kg K_2O ha^{-1}
Arthur	2014	170	no	1690	10,400	84
Buffalo	2014	115	yes	1190	9660	84
Fairmount	2014	140	yes	1250	11,160	112
Gardner	2014	110	yes	2010	10,700	84
Milnor	2014	110	yes	ns‡	12,480	–
Page	2014	200	no	ns	9780	–
Walcott E	2014	105	yes	ns	7710	–
Walcott W	2014	80	yes	ns	8660	–
Wyndmere	2014	100	yes	ns	9660	–
Arthur	2015	125	yes	1100	10,660	84
Barney	2015	170	no	ns	13,740	–
Casino	2015	120	yes	2070	14,100	84
Dwight	2015	110	yes	ns	13,300	–
Fairmount 1	2015	188	no	1130	13,170	84
Fairmount 2	2015	118	yes	1760	13,100	112
Leonard	2015	380	no	ns	10,600	–
Milnor	2015	117	yes	2010	12,170	140
Prosper	2015	205	no	ns	12,100	–
Valley City	2015	201	no	ns	7530	–
Walcott	2015	108	yes	940	12,360	112
Absaraka	2016	162	no	1400	13,170	112
Colfax	2016	54	yes	1568	15,430	112
Gardner	2016	60	yes	1630	12,100	112
Lisbon	2016	78	yes	3070	13,230	84
Valley City	2016	226	no	ns	10,960	–

† Soil test K critical level of 150 mg kg^{-1}, dry soil with 1 mol L^{-1} NH_4OAc-extractable K.

‡ No significant difference among treatment means ($P < 0.05$).

§ Maximum yield was greatest yield by treatment within the site.

which was contrary to the prediction for low soil test K. Six sites with soil test K above 150 mg kg^{-1} did not respond to K, which is in agreement with the original K soil test recommendations. However, three sites with K soil test >150 mg kg^{-1} had yield increases with K fertilizer, which was contrary to the prediction for high soil test K.

Overall, from 2014 to 2016, corn yield responses at eight of 25 sites were contrary to prediction. In 2014, the relationship between relative yield response to K fertilization and soil test K was not significant, indicating a poor ability to predicted yield response to K fertilization (Fig. 1). This prompted more extensive investigation of other soil test methods and consideration of soil mineralogy for K recommendations for corn.

Soil Test Methods

Several alternative soil test methods were investigated to determine if there were methods superior to the 1 mol L^{-1} ammonium acetate extraction for predicting yield response to K fertilization (Table 5). Of the six soil test K methods evaluated, NH_4OAc-extractable K from air-dry soil (DK) was the soil test showing the highest correlation using linear-plateau or quadratic-plateau models (Table 6). Other soil test K methods with significant regression relationships to relative yield response were NH_4OAc-extractable K from field-moist soil (MK) and K saturation, although relationships defined by these methods had lower R^2 values than DK. The K saturation method, with the CEC determined by summation of extractable cations, has been found unsatisfactory in a review by Kopittke and Menzies (2007). Other problems with the summation method are that North Dakota soils commonly have soil EC values >0.2 (Table 2), and Ca and Mg ions can be solubilized from the free lime present in soils with pH >7 (Warncke and Brown, 2012). Soil test K methods with the ability to extract some portion of non-exchangeable K, interlayer K, or mineral K (i.e., TBK, RK) could

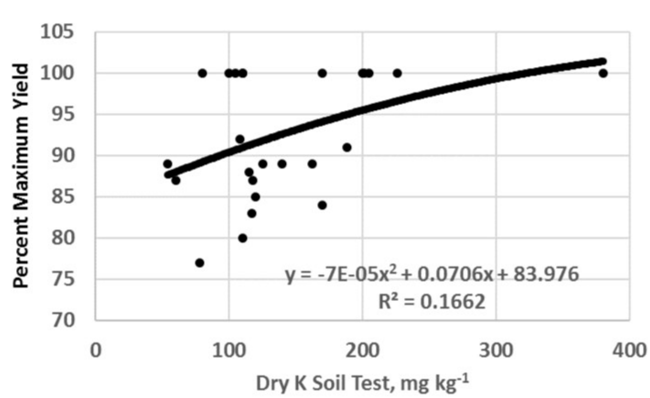

Figure 1. Response of corn to soil test K values without consideration of soil clay chemistry.

Table 5. Soil test K analysis of 2015–2016 experimental sites using various soil test methods and relative yield response of unfertilized treatment.

Site, year	Relative yield†	DK‡	MK	TBK 5 min	TBK 168 h	RK	K sat.	ECEC
	%	mg kg^{-1}					%	cmol$_c$ kg^{-1}
Arthur, 2015	89.7	90	84	282	1260	8.3	1.0	33.8
Barney, 2015	100	160	199	384	2460	22.1	1.6	31.5
Casino, 2015	85.3	87	103	284	1210	13.0	2.1	20.1
Dwight, 2015	100	109	101	249	1290	12.3	1.4	28.4
Fairmount 1, 2015	91.4	169	143	497	2610	7.8	1.8	31.7
Fairmount 2, 2015	86.6	100	82	250	1160	7.2	0.9	36.3
Leonard, 2015	100	444	428	1150	3480	79.5	4.1	29.1
Milnor, 2015	83.5	134	129	284	1160	13.8	1.4	38.2
Prosper, 2015	100	163	143	548	2640	13.7	2.1	28.8
Valley City, 2015	100	203	235	551	2200	25.3	2.8	17.1
Walcott, 2015	92.4	111	135	223	707	21.7	2.4	11.5
Absaraka, 2016	89.4	163	115	366	1900	14.0	1.4	30.6
Colfax, 2016	89.8	84	56	166	797	12.0	0.7	30.5
Gardner, 2016	86.5	84	55	225	1140	5.8	1.3	16.8
Lisbon, 2016	76.8	69	45	160	689	12.0	0.8	23.0
Valley City, 2016	100	201	171	504	2520	13.7	2.2	24.1

† Relative yield, relative grain yield of unfertilized treatment to maximum yield.

‡ DK, NH_4OAc extractable K from air-dry soil; ECEC, estimated cation exchange capacity via summation of extractable cations; K sat., K saturation of estimated cation exchange capacity; MK, NH_4OAc extractable K from field-moist soil; RK, resin extractable K; TBK, tetraphenylboron K.

not be related to relative yield. The coefficients of determination for DK models still indicate high residual variation, and about one-third of site yield responses were not accurately predicted using the 150 mg kg^{-1} soil test K critical level.

Consideration of Clay Mineralogy

Soil mineralogical analysis indicated that K-feldspar content was similar across sites (Table 7); however, the smectite/illite ratios of the clay fraction were different. Principal component analysis indicated that smectite and illite were the mineral components most strongly [$r = 0.33$ (sig $p < 0.05$) and 0.25 (sig $p < 0.05$), respectively] associated with relative yield (data not shown). Subsequently, Ward's minimum variance cluster analysis was used to determine if experimental sites could be partitioned by their smectite/illite ratio. Cluster analysis indicated that the experimental sites could be separated into two groups: smectite/illite ratio >3.5 and smectite/illite ratio <3.5.

Separating experimental sites into two smectite/illite ratio groups improved regression analysis of relative yield response and soil test K (Table 8). The regression models shown in Table 8 were the best-fit models (linear vs. quadratic) for the smectite/illite ratio groups. The two smectite/illite ratio groups had different soil test K critical levels. Consideration of clay chemistry is probably related to crop contributions of K from nonexchangeable K, which is not a significant source of K extracted by the DK method (Brar et al., 2016; Cox et al., 1999; Zhang et al., 2011). However, the investigations of soil extraction methods that might include extraction of nonexchangeable K (tetraphenylboron K and resin K) were ineffective in improving the soil test relationship to yield response, whereas the use of the soil clay fraction smectite/illite ratio was very effective.

Regression models in Table 6 suggest that the soil test K critical concentration should range from 93 to 115 mg kg^{-1}, depending on

Table 6. Regression models for relative yield of unfertilized corn plots and various soil test K methods for 2015–2016.

Model†	Equation‡	X§<	Maximum	R^2	$P > F$
Linear-plateau					
DK	31.5 + 0.664X	93	93	0.49	0.017
MK	32.6 + 0.998X	61	93	0.47	0.022
TBK 5 min	74.2 + 0.0636X	333	95	0.33	0.090
TBK 168 h	81.9 + 0.00704X	2028	96	0.30	0.117
RK	84.2 + 0.528X	–	no maximum	0.16	0.140
K sat.	74.1 + 12.7X	1.56	94	0.42	0.037
Quadratic-plateau					
DK	$-5.2 + 1.72X - 0.0075X^2$	115	94	0.52	0.012
MK	$-34.8 + 3.67X - 0.264X^2$	69	93	0.47	0.022
TBK 5 min	$60.5 + 0.178X - 0.00023X^2$	389	95	0.36	0.068
TBK 168 h	$77.3 + 0.0148X - 0.00000293X^2$	2522	96	0.31	0.109
RK	no model fit	–	–	–	–
K sat.	$62.1 + 35.9X - 10.2X^2$	1.77	94	0.39	0.052

† DK, NH_4OAc extractable K from air-dry and field-moist soil; ECEC, estimated cation exchange capacity via summation of extractable cations; K sat., K saturation of estimated cation exchange capacity; MK, NH_4OAc extractable K from field-moist soil; RK, resin-extractable K; TBK, tetraphenylboron K.

‡ Equation applies only for X values less than the value at which the segmented model joins (i.e., critical level corresponding to 100% maximum predicted yield by model shown).

§ X = soil test K (mg kg^{-1} for DK, MK, TBK 5 min, TBK 168 h, RK; % for K sat.).

Table 7. Soil mineral composition of K-bearing minerals of the 25 study locations. Whole soil refers to all mineral matter in the soil sample. The <2 mm fraction is the clay particle size fraction in the sample.

Site	Year	Whole soil		<2 mm fraction			
		K-feldspar	Muscovite-illite	Smectite	Illite	Kaolinite	Smectite/illite ratio
		g kg^{-1}					
Arthur	2014	49	17	860	110	<IDL†	7.8
Buffalo	2014	71	22	860	110	<IDL	7.8
Fairmount	2014	54	49	830	140	<IDL	5.9
Gardner	2014	53	39	750	200	<IDL	3.8
Milnor	2014	49	7	280	570	120	0.5
Walcott E	2014	61	38	720	220	<IDL	3.3
Walcott W	2014	73	23	510	400	70	1.3
Wyndmere	2014	61	36	720	220	<IDL	3.3
Absaraka	2015	99	18	840	140	20	6.0
Arthur	2015	95	30	850	120	30	7.1
Barney	2015	63	38	790	160	50	4.9
Casino	2015	64	26	850	120	30	7.1
Dwight	2015	60	23	820	150	30	5.5
Fairmount 1	2015	56	30	870	100	30	8.7
Fairmount 2	2015	74	19	790	140	70	5.6
Leonard	2015	69	66	700	250	50	2.8
Milnor	2015	86	34	740	200	60	3.7
Prosper	2015	92	36	830	140	30	5.9
Valley City	2015	56	17	650	300	50	2.2
Walcott	2015	62	18	470	480	50	1.0
Absaraka	2016	56	38	700	250	50	2.8
Colfax	2016	53	<IDL	770	160	70	4.8
Gardner	2016	61	28	770	190	40	4.1
Lisbon	2016	50	19	720	220	60	3.3
Valley City	2016	55	<IDL	810	160	30	5.1

† Below instrument detection limit.

whether the linear-plateau or quadratic-plateau model was used. The maximum of the quadratic model for sites with a smectite/illite ratio of <3.5 is 126 mg kg^{-1} soil test K, and the intersection of the linear regression equation for the sites with a smectite/illite ratio >3.5 with 100% of maximum yield is 200 mg kg^{-1} soil test K. This indicates that the critical soil test K level for sites with a smectite/illite ratio <3.5 should be ~130 mg kg^{-1}, and sites with a smectite/illite ratio >3.5 should be ~200 mg kg^{-1}.

Using these soil test K critical concentrations, yield response prediction for sites with smectite/illite ratios >3.5 was correct for 14 of 16 sites. For sites with smectite/illite ratios <3.5, yield response prediction was correct for five of seven sites. Overall, the two-soil test K critical levels improved yield response prediction for 19 of 25 sites. Other studies have recognized the contribution of soil clays to soil K availability to plants; however, there has been little work to define how the contribution could be used within a fertilizer recommendation strategy (Bhonsle et al., 1992; Sharpley, 1989). These separate soil test K critical levels identify additional criteria for predicting corn yield response to K fertilization.

CONCLUSIONS

Potassium fertilizer rate experiments in corn were conducted from 2014 to 2016 in southeastern North Dakota where the transition from wheat-dominated cropping systems to corn–soybean cropping systems has resulted in a rapid decline in soil test K levels over the past 30 years. The previously published soil test K critical level in North Dakota of 150 mg kg^{-1} only predicted 16 of 25 site responses correctly. Alternative soil test methods were explored, but none was superior to the standard NH_4OAC extraction on dry soil.

After mineral analysis for K-bearing minerals, cluster analysis indicated that experimental sites should be separated in groups with smectite/illite ratios greater or less than 3.5. The soil test K critical level for soils with smectite/illite ratio <3.5 was 128 mg kg^{-1}. The soil test K critical level for soils with smectite/illite ratio >3.5 was ~200 mg kg^{-1}. Using these soil test K critical levels for each clay mineralogical group, response prediction was increased from 16 of 25 sites to 19 of 25 sites. For soils with soil test K between 128 and 200 mg kg^{-1}, these criteria will improve yield response prediction that would have been considered nonresponsive based on soil test K alone. Therefore, clay mineralogy should be considered for improved soil test prediction of corn yield response to K fertilization in North Dakota. A clay mineralogy map of North Dakota has been produced based on a 144-soil sample survey that includes soil samples from at least two major soil groups within each county in the state (Franzen and Bu, 2018). The state clay mineralogy map will serve as a guide to

Table 8. Regression models for relative yield of unfertilized corn plots and soil test K for separated sites with smectite/illite ratio greater or less than 3.5.

Smectite/illite ratio	n	Equation	R^2	$P > F$
<3.5	16	$15.44 + 1.36X - 0.0054X^2$	0.52	0.001
>3.5	9	$75.272 + 0.1131X$	0.37	0.01

the probable soil smectite/illite ratio expected in a field without expensive analysis by individual farmers.

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