EVALUATION OF SOIL POTASSIUM TEST TO IMPROVE FERTILIZER RECOMMENDATIONS FOR CORN

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The Supervisory Committee certifies that this ***disquisition*** complies with North Dakota State University's regulations and meets the accepted standards for the degree of

MASTER OF SCIENCE

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ABSTRACT

A study was conducted at thirteen locations in North Dakota and Minnesota in 2013 and 2014 with the objectives of determining difference between the soil potassium (K) results based upon air-dried (K_{Dry}) and field-moist (K_{Moist}) soil samples during the corn growing season and to evaluate corn response to applied K-fertilizer. Overall, K_{Dry} tests showed higher K levels in the soil test results compared to K_{Moist} but the pattern of deviation was dependent upon various soil properties such as initial soil K level. Temporal variation of soil K levels indicated a need to consider time of soil sampling while making fertilizer recommendations. Potassium application significantly increased corn yields at only five out of 11 sites with soil K levels below critical K soil test levels (<150 ppm). Therefore, development of an improved soil testing strategy is required to improve the predictability of corn response to applied K fertilizer in this region.

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LIST OF ABBREVIATIONS

K	potassium
K_{Moist}	plant-available-K test of field-moist soil samples.
K_{Dry}	plant-available-K test of air-dried soil samples
ANOVA	analysis of variance
GDD	growing degree days

GENERAL INTRODUCTION

The soils of Red River Valley region of North Dakota (ND) and Minnesota (MN) are among the most fertile soils of the world. According to ND soil fertility summary report of 1991-2001, more than 98 percent of tested fields had soil K levels in high and very high K categories (Cihacek et al., 2009). The median K level for ND soils was 236 ppm in 2010, which is above the critical level of 150 ppm (Fixen, 2010). However, in recent years, the increasing agricultural area under soybean and corn has resulted in higher K removal from the soils and soil K levels were reported to be decreasing in eastern ND (Franzen, 2014). Therefore, evaluation of soil K-fertility status of this region is important to maintain optimum nutrient levels to ensure high crop yields.

For plant available K analysis, soil samples are air-dried prior to chemical analysis. However, drying soil samples can lead to over- or under-estimation of actual soil K levels (Haby et al., 1988; Barbagelata and Mallarino, 2012). Recently, Iowa State University has re-introduced the methodology of using of field-moist soil samples for soil K analysis to formulate the fertilizer recommendations. Better correlation of field-moist soil K analysis with corn yield in Iowa has made it an area of interest for North Dakota and Minnesota corn growers. Farmers of this region are becoming more interested in corn production due to higher profits compared to other crops. Hence, it is important to review the 40 year-old K recommendations for corn to ensure beneficial outcomes to farmers. Therefore, a research study was conducted to determine the corn response to different K-fertilizer rates and to assess the variation of soil K test levels between air-dried (K_{Dry}) and field moist (K_{Moist}) soil samples during the growing season.

In my thesis, I have presented the findings of field K-experiments conducted in 2013 and 2014. In 'literature review' chapter, I have extensively reviewed the literature on (i) comparison of soil potassium test based upon air-dried and field moist samples, (ii) effect of time of sampling on soil K test results and (iii) corn response to applied K fertilizer rates. Chapter entitled ‘Is air-drying of soil samples an appropriate step in determining plant available potassium for corn?’ is a paper submitted to Journal of Plant Nutrition which includes findings from field trials during 2013 growing season. In following chapter ‘evaluation of soil potassium test for recalibration of corn response curves, I have presented my 2014 data. I have summarized my two-year research results in ‘general summary and conclusion’.

LITERATURE REVIEW

Comparison of soil potassium test based upon air-dried or field moist samples

The reliability of soil test results for fertilizer recommendation is based upon the correctness of soil sampling techniques and analytical procedures (Sabbe and Marx, 1987). Therefore, soil sample preparation should involve minimal chemical and mechanical disturbance to represent the field conditions. However, due to inconveniences in handling and analyzing field-moist soil samples, air-drying of soil samples is a common pre-treatment utilized to conduct most soil analysis (Gelderman and Mallarino, 1998). Several researchers have documented that the drying of soil samples alters surface acidity, affecting the solubility of various nutrients (Bartlett and James, 1980; Eric and Hoskins, 2011), while others have recognized that the collapse of clay structure due to change in the oxidation state of iron during drying within clay minerals may lead to release or entrapment of certain cations, particularly potassium (K) (Khaled and Stucki, 1991).

Whether or not to dry soil samples prior to soil K analysis has been controversial since the early 1900's. Since then, various researchers have analyzed field-moist (K_{Moist}) samples and dry (K_{Dry}) soil samples to understand the extent of variation of soil test K levels due to sample moisture and better correlate soil K analysis with crop response. Barbagelata and Mallarino (2012) reported 1.92 times higher K values in K_{Dry} than K_{Moist}, while Haby et al. (1988) found decreases in K values when soil samples were dried before analyzing plant available K. These conflicting results indicate that increases and decreases in soil K are both possible among different soils. Efforts have been made to identify the factors responsible for release or fixation of K upon drying (Steenkamp, 1928; Attoe, 1947; Luebs et al, 1956; Hanway and Scott, 1959;

Burns and Barber, 1961; Grava et al., 1961; Barbagelata and Mallarino, 2012; Schneider et al, 2013).

Potassium ions (K^+) possess an appropriate size to become trapped in the interlattice spaces within clay minerals. Dehydration of the K^+ ion due to soil drying can result in redistribution of interlayer cations. Clay layers are not often uniform in interlayer distances, and ions such as Fe^{3+} may form ‘wedges’ that restrict normal flow of K^+ and other ions in and out of the interlayer space. Since calcium has a higher affinity for negative charged clays, it may compete with K^+ for wedge zones and K^+ can be released into the soil solution upon drying (Sparks and Huang, 1985). The chemistry of clay present in the soil has a great influence on soil K levels. Clay mineral types show different behavior in release and fixation of K when exposed to dry conditions. George (1947) while working with electro-dialyzed illite clay and acid-washed Wyoming bentonite reported that illite and expanding type of clays fixed K upon drying. Weathered micas were found to show characteristic of fixing potassium under dry as well as moist soil conditions whereas montmorillonite (a smectitic, expanding clay type) fixed K only under dry conditions (Rich, 1968). On the other hand, Fine (1940) reported release of K from bentonite clay when exposed to freezing conditions (a drying process).

McLean and Watson (1985) stated that initial soil K level is an important factor in determining an increase or decrease of K levels in air-dried soil K analysis. The effect of initial soil K level is evident in Cook and Hutcheson’s (1960) research study where they identified that Kentucky soils gave higher soil K levels upon drying if soil-K levels were below 0.5 meq $100g^{-1}$, and lower K values if soils had K level of above 0.5meq $100g^{-1}$. Similarly, Haby et al. (1988) found that soil drying leads to over-estimation of K if soil K levels were below 420 mg kg^{-1} and under-estimation of K if soil K level were above 500 mg kg^{-1} in dry-land agricultural soils of the Northern Great Plains.

Burns and Barber (1961) reported that soil texture and the relative level of exchangeable K are the primary factors for controlling the extent of K variation between K_{Dry} and K_{Moist} results. Sandy soils showed little change, while silt loam showed appreciable change in the results of K_{Dry} and K_{Moist} (Hanway, 1962). Others have identified cation exchange capacity, clay mineralogy, soil sample moisture content, soil organic matter, total base content, and Ca^{+2} plus Mg^{+2} to K^{+} ratio as additional factors responsible for release or fixation of K upon drying (Matthews and Sherrell, 1960; Barbagelata and Mallarino, 2012).

Soil samples collected from different depths of the same location have shown variable K_{Dry} and K_{Moist} results (Hanway and Scott, 1957). Air-dried sub-surface soils showed an increase of 118% while surface soils showed an increase of 14.3% of K level compared to field-moist sample K (Large, 1969). The extent of K-release depends upon the weathering state of clay minerals (Steenkamp, 1928). Since soil's surface layers are more prone to weathering, they have less potential to release K than sub-surface soils (Thomas and Hipp, 1968).

After consideration of the relationship between K_{Dry}, K_{Moist} and associated crop yield response to K, the Iowa State University Soil and Plant Analysis Laboratory started using field moist soil K analysis in the mid-1960s. It was later discontinued in 1988 due to the inconvenience of moist soil sample handling and processing. Iowa State used a factor of 1.25 based upon relation of K_{Dry}/K_{Moist} results in their experiments (Mallarino, 2005), while Bates et al., (1969) proposed treating soils with an organic compound (dextrose) to overcome the effect of sample drying. However, use of dextrose was not helpful in controlling the fixation of K experienced in many soils upon drying (Bates et al., 1969). Also, Mallarino et al. (2003)

observed that K_{Dry}/K_{Moist} ratio was dependent upon the soil drying temperature, soil drainage class and many unknown factors. The use of a factor-1.25 was not sufficient to overcome the sample drying effect.

Many researchers have tried to evaluate the variation of dry and moist K soil tests in the past several decades. However, due to the extreme complexity of soil K dynamics, it is difficult to propose a universal solution for the soil drying effect to all soil types. The extent of over or underestimation of soil K test upon drying and factors responsible for release or fixation are specific to soil and other unexplained factors. In the context of existing unpredictability of soil K tests, it would be advisable to review the existing methodology of soil sample drying before K analysis in North Dakota and to find the factors effecting the variation of air-dried soil K and field- moist K analysis.

Effect of sample timing on soil K test results

Potassium management is often difficult due to the inconsistency of soil K test results (Mallarino, 2011). Extensive research has been conducted to improve the soil K test calibration to predict the yield response. However, current ability of the soil K test to estimate the plant-available K in many soils has resulted in an effort to improve soil K test methodology (Romheld and Kirby, 2010). One of the problems in K management is temporal variation of soil K; however little consideration has been given to the time of sampling. A number of studies have revealed the occurrence of huge variability in soil test results and nutrient availability with space and time (Cain et al, 1999; Anonymous, 2012; Franzen, 2012).

Peterson and Krueger (1980) reported that soil K levels were variable within and between the crop growing seasons in an 8-year experimental period. A cyclic trend of variation had been reported for soil K, with highest K level in April and May and then gradual decrease up to

September, followed again by increase in winter season (Lockman and Molloy, 1984). In an experiment conducted over five different locations in Wisconsin, large differences were observed between the soil K levels of fall and spring that affected the soil test interpretation category and the fertilizer recommendations (Vitko et al., 2010). Similarly, Mallarino et al. (2011) found large variability of soil K results in a three-year experiment in Iowa. They observed that trend of increase or decrease in K levels was not consistent and seasonal K fluctuations were concluded to be site specific.

Various factors have been identified for seasonal variation in soil K test results. Under - estimating the importance of short term equilibrium between exchangeable K and non-exchangeable K is found to be one of the major factors for high temporal variations of soil K levels (Mallarino, 2011). Current measurement methods for estimating plant available potassium involve measurement of exchangeable and soil solution K. However, the exchangeable K-ion is liable to transform into non-exchangeable K or *vice–versa* depending upon the chemical equilibrium (Bray and DeTurk, 1938), such transformations can lead to increase or decrease of soil-K test levels over the time.

Soil moisture has a profound influence on the release and fixation of soil K. An exponential increase of soil K was observed by lowering the soil moisture level below 10 percent (Luebs et al., 1956). Therefore, interpretation of soil K under different moisture regimes is likely to show variable soil K results. Further, North Dakota often attain sub-zero temperatures in winter months (NDAWN, http://ndawn.ndsu.nodak.edu/). Freezing and thawing can affect the soil physical, chemical and biological properties (Oztas and Fayetorbay, 2003; Henry, 2007). In a Saskatchewan study, freeze and thaw cycles decreased exchangeable K levels (Hinman, 1970).

Likewise, Fine et al. (1940) observed increase and decrease of soil exchangeable K due to freezing depending upon initial soil K levels.

In addition to the soil moisture effect, plant uptake during the growing season can greatly reduce the soil-K content (Murrell, 2011). At the end of the growing season, release of K from crop residues with rainfall, or removal of the residue may have major roles in K recycling. Potassium is present in plant tissue in an inorganic form and can be leached from tissues following senescence into the soil (Tukey, 1966). Rosolem et al. (2005) found that crop residues of millets can recycle up to 3 to 8 kg ha^{-1} K per ton of residue if the residue remains in the soil. Mallarino et al. (2011) mentioned a sharp decrease of K content in corn and soybean residues from physiological maturity to harvesting due to leaching of K. These results indicate that soil test K results are likely to show variable results during the growing season depending upon the plant K-uptake and leaching pattern, in addition to soil moisture trends.

In some of the Corn Belt areas, crop advisers have reported that farmers are requesting a shift to spring, rather than fall soil sampling (Murrell, 2009). As there is considerable variability between K results of samples taken in fall as compared to spring, fertilizer recommendations may need to vary accordingly (Childs and Jencks, 1967; Liebhardt and Teel, 1977; James and Wells, 1990). Since fluctuations in soil K tests are soil and environment dependent, it would be important to inspect the temporal variation of soil K levels of North Dakota to further improve K-fertilizer recommendations.

Corn response to applied K fertilizer rates

Potassium is an essential plant nutrient required in large quantities by corn (McLean and Watson, 1985). Potassium is not a component of biochemical structure but it plays a vital role in water uptake, translocation of assimilates, enzymatic activities and improving the quality of grains and other plant products (Havlin et al., 2005). Therefore, yield losses can be expected in potassium limited soils (Barber, 1959; Dessele, 1967, Pettigrew, 2008). Application of K on high-K testing soils can lead to no or negative yield responses (Mallarino et al., 1991; Wortmann et al., 2009).

For fertilizer recommendations in North Dakota, soil test levels are calibrated with yield response and grouped into five categories. These five soil classes and corresponding probability of getting yield response are very low (>80%), low (50-80%), medium (20-50%), high (10-20%) and very high (<10%) (Franzen, 2010). However, there have been studies where even high K soils have shown positive corn yield response to fertilizer application while others with very low K fertility status have not (Kuchenbuch and Buczko, 2011). Other experiments showed minimal benefits from K application (Hanway, 1962; Bruns et al, 2006). Barbagelata and Mallarino (2012) reported increases in corn yield with added K on 41% of their experimental sites; however, there was a great deal of unexplained variability. High temporal and spatial variability of available soil K and soil test K values was reported as factor for unexpected yield responses from twenty experimental sites in Iowa (Clover and Mallarino, 2013). There was no grain yield response to K application for two consequent years in a study conducted by Heckman et al. (1992) over loamy sand soil while yield increased linearly in third year of their experiment. These studies illustrate that corn response may not be solely related to soil K analysis, but to additional unconsidered factors. This is further affirmed by Kuchenbuch and Buczko (2011) where they reported that soil test values and fertilizer application alone were insufficient to predict the corn yields and they recommended the use of soil physical properties, soil moisture dynamics and plant use efficiency to make appropriate fertilizer recommendations.

Nutrient utilization efficiency of a crop is the net effect of existing soil physical, chemical and biological conditions. Nutrient acquisition by the plants is effected by the ionic form of the nutrient present in soil as well as ability of roots to uptake that ion (Brouder and Volenec, 2008). Different plant species show different K uptake and utilization efficiencies which lead to variable K responses (Schenk and Barber, 1980; Rengel et al., 2008). In addition, certain plant species have shown the ability to utilize even the non-exchangeable form of K from the minerals by releasing organic acids as root exudates (Zorb et al., 2013).

Further, prevailing rainfall conditions have a great impact upon the maintenance of moisture level which is responsible for different trends of K availability to plants. High rainfall can lead to significant potassium leaching in coarse textured soils on the one hand, while resultant increase in soil moisture content can increase the diffusion rate of K into the plant roots (Havlin et al., 2005). Another theory of high uptake of K by plants after intercepting high rainfall is that during low rainfall years, roots extend to deeper layers where K level is generally low. But in high rainfall years, roots concentrate in upper layers where they intercept more K (Barber, 1959). Apart from climatic and plant factors, existing soil conditions such as pH, cation exchange capacity (LaBarge and Lindsey, 2012) and clay content is known to effect the nutrient availability to plants (Blake et al., 1999).

Most of the North Dakota soils have potential to supply K to fulfill the needs of the crops. Since corn requires a large quantity of K during the growing season, it is likely to respond to K-fertilizer application (Norum and Weiser, 1957). The fertilizer recommendation for corn was established about 40 years ago (Franzen, 2014). Therefore, it is essential to refine the strategies to ensure economic returns to farmers from corn. In the context of recent findings, it would be necessary to recalibrate the corn response to the applied K-fertilizers in North Dakota soils.

IS AIR-DRYING OF SOIL SAMPLES AN APPROPRIATE STEP IN DETERMINING PLANT AVAILABLE POTASSIUM FOR CORN?[1]

Abstract

Potassium (K) fertilizer recommendations are mainly based on air -dried soil samples which can lead to over- or under-estimation of plant available soil K. Three on-farm trials were conducted in North Dakota and Minnesota to determine the variation of soil test-K between air-dried (K_{Dry}) and field moist (K_{Moist}) soil samples. The differences between K_{Dry} and K_{Moist} decreased as soil K increased, but increased linearly with increasing soil moisture. Soil drying influenced the plant available soil K-test value, producing higher K values compared to the moist soil K. It is unclear based on these initial experiments which method might produce a more predictable K critical value to aid in directing K application for corn in this region.

Introduction

Soil testing plays a key role in formulating fertilizer recommendations. Most of the commercial soil testing labs and universities in the USA, with the exception of Iowa (Mallarino et al., 2013) include air drying of soil sample as a standard soil preparation step. Recommendations are based on extracting solutions, such as 1-*N* ammonium acetate or Mehlich-3, added to the dried and ground soil sample (Erich and Hoskins, 2011; Nathan and NCERA-13 Soil Testing and Plant Analysis Committee, 2011). There is concern that air-drying soil samples for plant available soil potassium (K) status prediction may lead to over- or under-estimation of plant available K (Attoe, 1947; Luebs et al, 1956; Burns and Barber, 1961; Barbagelata and

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[1] The material in this chapter was co-authored by Manbir Rakkar, David Franzen and Amitava Chatterjee. Manbir Rakkar had primary responsibility for collecting samples in the field and lab analysis. Manbir Rakkar drafted and revised all versions of this chapter. David Franzen and Amitava Chatterjee served as proofreader and checked the math in the statistical analysis conducted by Manbir Rakkar.

Mallarino, 2012). When soil-K levels are high, clays may trap K inside interlattice spaces upon drying. When soil-K levels are low, soil drying may cause clay edges to scroll outwards, and K may be released from the interlayers, that results in a sort of chemical K equilibrium controlled by inherent soil characteristics (McLean and Watson,1985). Variation between K_{Moist} and K_{Dry} has been reported by many studies. Barbagelata and Mallarino (2012) reported 1.92 times higher K values in K_{Dry} than K_{Moist} while Haby et al. (1988) found decreases in K values when soil samples were dried. Burns and Barber (1961) reported that soil texture and the relative level of exchangeable K were the primary factors controlling the extent of K variation between dry and moist samples. Others have identified cation exchange capacity, clay mineralogy, initial soil sample moisture content, soil organic matter, total base content, and Ca^{+2} plus Mg^{+2} to K ratio as additional factors responsible for release or fixation of K upon drying (Barbagelata and Mallarino, 2012).

Plant available soil test K levels are not only subject to moisture content through the analysis procedure, but also from the timing of obtaining the soil sample. In a long-term Illinois temporal variability study (Franzen, 2011) soil test K levels were lowest in August/September, when the soil was driest, and highest in December/January when the soil was wettest. Because soil wetness occurred in some years at normally dry season, but K levels persisted in the seasonal variation despites peaks of moisture, the seasonality is probably not only soil moisture driven, but may also include K leaching from crop residues, seasonal variability in moisture (high moisture in winters and comparatively low moisture towards the end of growing season when soils are driest), freezing and thawing, and microbial activity (Murrell, 2011). Temporal variations of soil-K levels may need to be considered when making fertilizer recommendation, and certainly when looking for trends in field K levels over years.

Potassium fertilization effects on corn yield and plant tissue K concentration needs to be revisited for modern corn varieties and cultural management. Current corn K recommendations in North Dakota are based on the categories for soil K analysis that are very low (<40 ppm), low (41-80 ppm), medium (81-120 ppm), high (121-160 ppm) and very high (>161 ppm) (Franzen, 2010). Some studies have shown little to no increase in grain K concentration with increasing K application rates (Clover et al., 2013). However, regardless of soil test K level or grain yield response, K fertilization nearly always increases plant tissue K concentration during vegetative stage (Clover et al., 2013; Mallarino et al., 2009). Corn yield increases have been recorded with soil test-K values were <135 ppm in Ontario (Vyn et al., 2001), while Ebelhar et al. (2000) found 168 ppm as the critical level in Illinois for obtaining a yield increase.

Most of the USA including North Dakota was reported to have a negative potassium budget (Fixen et al., 2010), which means that K removed from the soil is greater than the amount returned as amendments. Although some of these soils currently have a K surplus, many others do not. One reason that in many regions K is not being applied at rates required for soil replacement is that the soil test values may not reflect critical levels of corn response. The objectives of this study are: 1) to record the corn yield and corn plant K uptake response to K fertilization, 2) examining the differences in soil test K levels between field-moist and air-dried soil samples, 3) record the temporal variation of soil test K through a corn growing season.

Material and methods

Description of experimental sites and treatments

Three on-farm trials were conducted at (1) Gardner (N 47^{O} 9’586, W 97^{o} 02’830”) and (2) Valley City (N 46^{O}53’407” and W 97^{O}55’033”) in North Dakota, and (3) Ada (N 47^{O} 19’53” and W 96^{O} 23’33”) in Minnesota. The soils are classified at Gardner as Gardena fine sandy loams:

coarse silty, mixed, superactive, Pachic Hapludols; Valley City as Barnes loams Fine-loamy, mixed, superactive, frigid calcic Hapludolls; Ada as a Ulen soil sandy, mixed, frigid Aeric Calciaquolls (Soil Survey Staff, 2013). Annual average temperature and precipitation of the growing season in 2013 and past 32 years are presented in Table 1.

The experimental design of the trials was a randomized block with six K application rates in the form of KCl (0-0-60), control (K0), 33.60 (K1), 67.1 (K2), 100.8 (K3), 134.4 (K4), K5-168.0 (K5) K_2O kg ha^{-1} and four replications. Nitrogen and phosphorus were applied to the entire site according to soil test recommendations (Franzen, 2010). Corn variety Pioneer 4086 was planted at a population density of 87500 plants ha^{-1} with row spacing of 0.55 m at Ada and 0.76 m at Gardner and Valley City. Each experimental unit (plot) was 9.14 m long by 3.34 m wide with 6 rows per plot at Ada and Valley City and 4 rows per plot at Gardner.

Soil sampling and analysis

Three initial composite soil samples per plot were collected from 0-15 cm and 15-30 cm before planting and analyzed for plant available K using the 1-*M* ammonium acetate method, with both the moist and dry soil methods. Texture, electrical conductivity, soil pH, organic matter, and bulk density were analyzed (Elliot et al, 1999; Thomas, 1996; Combs and Nathan, 1998; and Blake and Hartge, 1986).

Soil and plant samples were collected during the growing season at corn growth stages described in Table 2. Soil samples (0-15 cm depth) were collected from each plot and stored in zip-lock plastic bags to retain moisture. Each soil sample was thoroughly mixed and divided into three sub-samples. One intact sub-sample was analyzed for field moist soil-K (K_{Moist}) and the other sub-sample for dry soil-K (K_{Dry}) level according to procedure recommended by Warncke and Brown (1998) with some modification. For K_{Dry}, 1g of sample with 20 mL of neutral 1M

NH_4OAc and for K_{Moist}, 2g of dry equivalent was used with 40 ml of neutral 1M NH_4OAc maintaining the ratio of 1:20 (soil to extracting solution). The soil and extractant was shaken for 5 min and filtered through Whatman No. 2 filter paper. Soil K concentration was determined on the filtrate using a Buck Scientific Atomic Absorption Spectrometer - Model 200A (Norwalk, CT, USA) using 766.5 nm of wavelength. The third subsample was used for gravimetric moisture content determination by first weighing moist, then reweighing after oven-drying at 105 ^{o}C for at least 24 hours.

Plant sampling and analysis

Plant samples were collected at the same times as soil sample collection (Table 2). The entire above ground plant was taken from an exterior row (V4), the uppermost mature leaves within the same rows were obtained at V8, V12, VT and ear leaf at R1 and tasseling, were obtained, transported in a cooler to the drier, dried , ground and analyzed for K concentration. At harvest stover and grains were analyzed separately for K concentration (Clemson University, 2013). After drying in an oven at (55 ^{o}C) for 4-5 days until the weight was stable, the samples were ground in a Wiley Mill (Swedesboro, NJ, USA) using 2 mm screens. About 0.5 to 1g ground plant material was ashed in an electric muffle furnace using a gradually increased temperature up to 500 ^{o}C for 2 hours followed by a constant temperature of 500 ^{o}C for 4 hours. The ash was then acid-treated with 5 ml of 6 N HCl and then the sample was dried over a hot plate at 285 ^{o}C. The sample was re-dissolved with 10 mL of 1N HCl and transferred to 50 mL volumetric flasks. The K concentration was analyzed using the same atomic adsorption spectrometer as was used for soil K analysis. To determine yield, a 2 m row was harvested from two-middle rows and grain yield was estimated as Mg ha^{-1}.

Data analysis

Analysis of variance for all soil and plant parameters was conducted using SAS Enterprise Guide 4.3. Means of main effects were compared using Fisher's least significant difference (LSD). Pearson correlation coefficients were used to evaluate the relationship among the parameters at a 95% significance level.

Results

Initial soil samples

The initial soil test results of all three sites are presented in Table 3. Ada and Gardner were coarser in texture compared to the Barnes soil at Valley City. The Ulen soil at Ada had a higher pH and EC compared to the other sites. Initial soil K levels are presented in Table 4. On average, percent variation between K_{Dry} and K_{Moist} was 36%. The differences between K_{Moist} and K_{Dry} were greater at Ada and Gardner compared to Valley City. The difference between K_{Dry} and K_{Moist} in initial samples at Gardner was similar to that of Ada, with a 53.8% difference at the 0-15 cm depth (Table 4). The percentage change was greater in K_{Moist} for soil samples collected from 15-30 cm depth as compared to 0-15 cm soil.

Differences in extracted soil K between air-dried and field-moist samples

Soil K extracted from air-dried samples and field-moist samples showed significant relation at 95 % confidence level. Ratio of K_{Dry} and K_{moist} showed a quadratic relation with increasing K_{Moist} level (Fig 2) while moisture was not significantly related to the amount of variation in K_{Dry} and K_{moist} during the corn growing season

Temporal variations

Soil samples collected during the growing season showed variation in plant available K values at all sites (Fig 3(a)). Corresponding changes of moisture at particular GDDs are shown in

Figure 3(b). Temporal changes of soil K was not significantly related to moisture (%) of soil sample at Gardner (R^2=0.41) and Valley City (R^2=0.51). At Ada, changes in plant available K during the growing season was significantly related to changes in moisture (%) of soil samples (R^2=0.82, p –value <0.0001).Change in K_{Dry} and K_{Moist} of Ada site during the growing season are presented in Fig. 4. There was about 80% greater K_{Dry} compared to K_{Moist} levels at V4 (350 GDD), but only 20% at VE (168 GDD). Amount of variation between K_{Dry} and K_{Moist} remained similar after V12 (GDD 949) at Ada and throughout the season at Gardner and Valley City.

Corn response to potassium fertilization

There was an increase in plant K concentration with an increase in K rate at Ada (Fig. 5). There was no difference in K level of corn vegetation due to K rate at any sampling date at Valley City site while Gardner site showed significant difference only at V12 (1180 GDD).

Corn grain yield showed a significant increase in yield and had quadratic relation with K-rates at Ada site with correlation coefficient (R^2=0.99) and Gardner (R^2 =0.97) (Table 6). But other site which initially had a high soil–K showed an increase in yield but it was not statistically significant.

Discussion

Variation among extracted-K from air-dried and field-moist soil samples

The texture of soils at Ada and Gardner was coarser than those at Valley City. These sites showed more difference between K_{Moist} and K_{Dry} compared to the Valley City site. At all sites, soil test K tended to decrease as the season progressed and drier conditions were present from early July to harvest. The percent difference between K_{Dry} and K_{Moist} was also less with drier conditions. There was also a trend of decreasing difference between K_{Dry} and K_{Moist} as soil K increased (Table 5). For example, at Ada, the ratio of K_{Dry} and K_{Moist} decreased from 2.91 to 0.54 between minimum and maximum value of K_{Dry} and K_{Moist} tests.

Temporal variations

Potassium levels decreased during the cropping season as the soil dried and K was taken up by the growing corn crop (Fig 3(a)). Approximately 95% of the corn K uptake is absorbed within 54 days from planting (Welch and Flannery, 1985). So, the amount of variation between K_{Dry} and K_{Moist} was similar after V12 at Ada (Fig. 4). Some studies have showed that soil moisture influences the differences between K_{Dry} and K_{Moist} (Barbagelata and Mallarino, 2012); however, moisture had little effect on variation in these experiments. Analysis of variance showed non-significant relation of moisture content of soil to percentage change in K_{Dry} and K_{Moist} level.

Corn response to potassium fertilization

An increase in corn K concentration in vegetative tissue is often recorded in fertilizer K rate experiments (Clover and Mallarino, 2013). An increase in corn plant K concentration was observed at Ada, but not at Gardner or Valley City. But the corn grain yield showed high correlation with the K-fertilization. Percentage increase in yield was highest at Ada site while it was minimal at Valley City. Such response was corroborated with the finding that probability of getting yield response for soil-K interpretation classes of low and medium category is 50-80% and 20-50%, respectively (Franzen, 2010). Soils with optimum levels of K provide the required amount of K to plants even if no additional nutrient is applied.

Barbagelata and Mallarino (2012) found field moist soil samples to be a better predictor of corn yield rather than dried soil sample analysis. Although dried soil samples allow convenience in sample handling, and the laboratory procedure, it leads to a difference in alteration in nutrient extractability (Erich and Hoskins, 2011). On the other hand, field moist samples keep moisture intact and are more likely to represent field conditions.

Summary and conclusions

The extent of variation between K_{Dry} and K_{Moist} can be explained by initial soil K levels of sites. Differences between K_{Dry} and K_{Moist} changes as the soil K level decrease during the growing season. Potassium application increased corn yield at the two sites having very low and medium soil K status while vegetative K concentration was significantly increased at the site that had lowest K level. More site data is required to construct the corn response curves to applied K fertilizers based upon K_{Moist} test results to change the existing K recommendation protocol.

Table 1. Average monthly temperature (^{o}C) and precipitation (cm) with 32-year of average recorded at three experimental sites during growing season (NDAWN).

	Average Temperature (^{o}C)						**Average Precipitation (cm)**					
	Ada		**Gardner**		**Valley City**		**Ada**		**Gardner**		**Valley City**	
Month	**Past 32 years**	**2013**	**Past 32 years**	**2013**	**Past 32 years**	**2013**	**Past 32 years**	**2013**	**Past 32 years**	**2013**	**Past 32 years**	**2013**
May	13.4	13.4	14.0	14.1	14.1	13.9	6.85	11.1	6.72	14.1	6.51	10.5
June	18.6	18.5	19.7	19.0	19.9	18.7	6.90	6.14	8.39	19.9	7.92	19.3
July	20.2	21.4	22.0	22.0	21.4	21.6	6.27	1.30	6.52	2.65	6.91	2.01
Aug	20.0	20.0	21.7	20.7	21.0	20.2	5.36	4.00	6.27	1.22	5.98	5.05
Sept	16.4	14.6	18.0	14.8	17.6	14.4	6.19	10.6	5.76	10.6	5.86	9.25

Table 2. Corn stage corresponding to growing degree days and days after sowing.

Ada			Gardner			Valley City		
Days after sowing	**GDD**	**stage**	**Days after sowing**	**GDD**	**stage**	**Days after sowing**	**GDD**	**stage**
1	0	V0	1	0	V0	1	0	V0
14	168	VE	13	249	VE	23	266	VE
30	350	V4	34	470	V4	35	492	V4
45	663	V8	45	681	V8	46	772	V8
59	949	V12	54	981	V12	55	1000	V12
71	1180	VT	70	1338	VT	70	1275	VT
85	1360	R1	84	1497	R1	84	1474	R1
98	1580	R2	96	1720	R2	97	1705	R2
139	2235	R6	137	2451	R6	138	2417	R6

Table 3. Basic soil properties of initial soil samples collected before planting from three experimental sites.

Site	Soil Series	Texture	pH	EC	Organic Matter	Bulk Density
				µS cm^{-1}	(%)	(g cm^{-3})
Ada	Ulen	Loamy sand	8.60	131	4.07	1.59
Gardner	Gardena	Sandy loam	6.69	94.0	3.33	1.49
Valley City	Barnes	Loam	6.09	42.0	3.66	1.44

Table 4. Standard air-dried soil K (mg kg^{-1}) of initial soil samples at Ada, Gardner and Valley City and percent difference in available K (mg kg^{-1}) of field-moist compared to standard air- dried soil K.

Site	Depth	K_{Dry}	K_{Moist}	Change in K_{Moist}
	cm	-------mg kg^{-1}-----		%
Ada	0-15	47.1	22.8	-51.6
	15-30	21.7	22.5	3.74
Gardner	0-15	89.5	41.3	-53.8
	15-30	54.3	18.7	-65.5
Valley City	0-15	115	103.11	-10.1
	15-30	79.0	50.1	-36.6

Table 5. Summary table of K levels of K_{Dry} and K_{Moist} tests of three sites during the growing season.

	Ada			**Gardner**			**Valley City**		
	K_{Dry}	**K_{Moist}**	**K_{Dry} / K_{Moist}**	**K_{Dry}**	**K_{Moist}**	**K_{Dry} / K_{Moist}**	**K_{Dry}**	**K_{Moist}**	**K_{Dry} /K_{Moist}**
					-------------------------mg K kg^{-1}------------------------------				
Mean	42.1	31.9	1.39	75.2	62.3	1.37	156	161	1.12
Max.	121	104	2.91	200	199	3.70	383	398	2.75
Min.	17.3	11.5	0.54	31.4	20.4	0.52	50.9	29.9	0.35

Table 6. Corn grain yield for each site as affected by different potassium application rates.

	Location		
K_20 (Kg ha^{-1})	**Ada**	**Gardner**	**Valley City**
	----------------- Mg ha^{-1}	-----------------	
0	6.53 c*	4.84c	9.18 a
33.6	8.14 bc	5.33bc	9.60 a
67.3	9.16 ab	6.09 ab	9.26 a
101	9.93 ab	6.15 ab	9.66 a
135	10.3 ab	6.52 a	9.60 a
168	10.6 a	6.77 a	10.2 a
LSD (α=0.05)	2.06	1.03	ns

*Values followed by the same letter in each column are not significantly different from each other

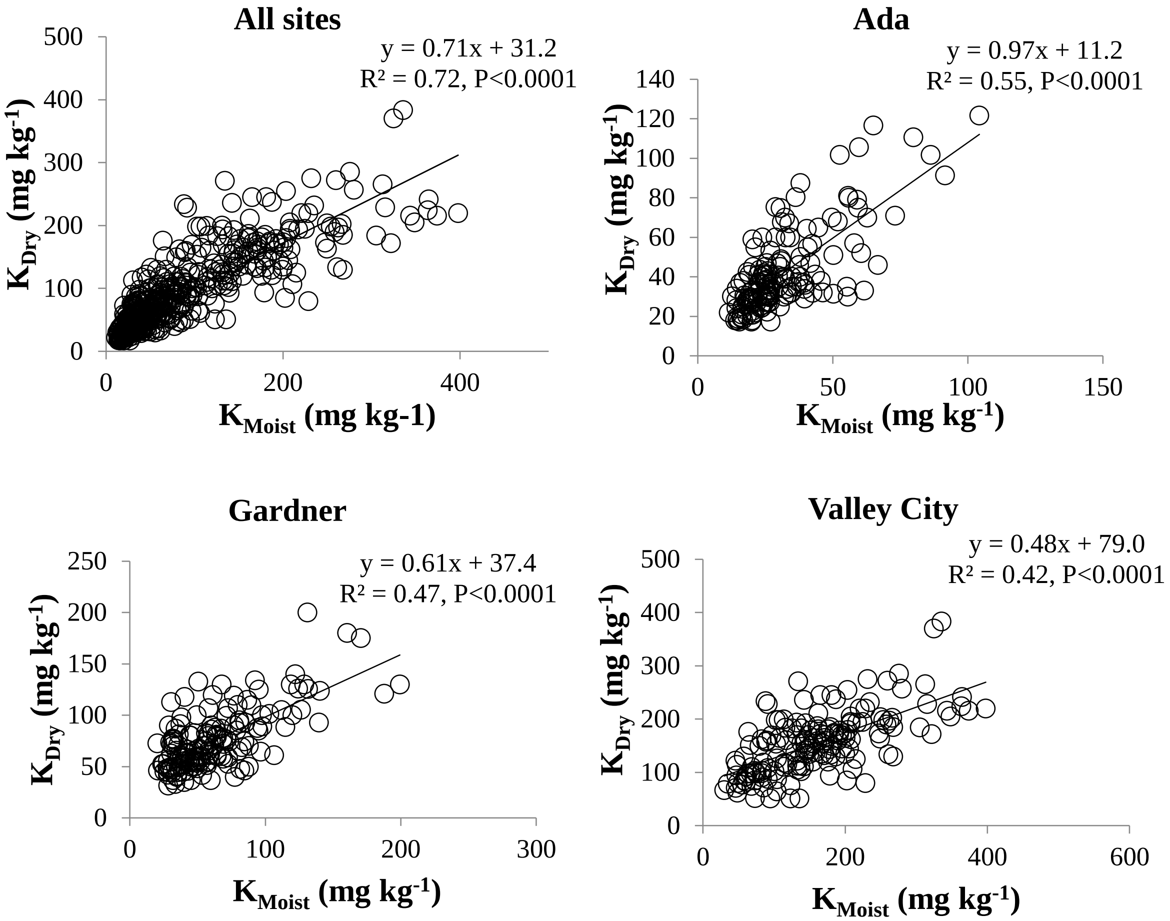
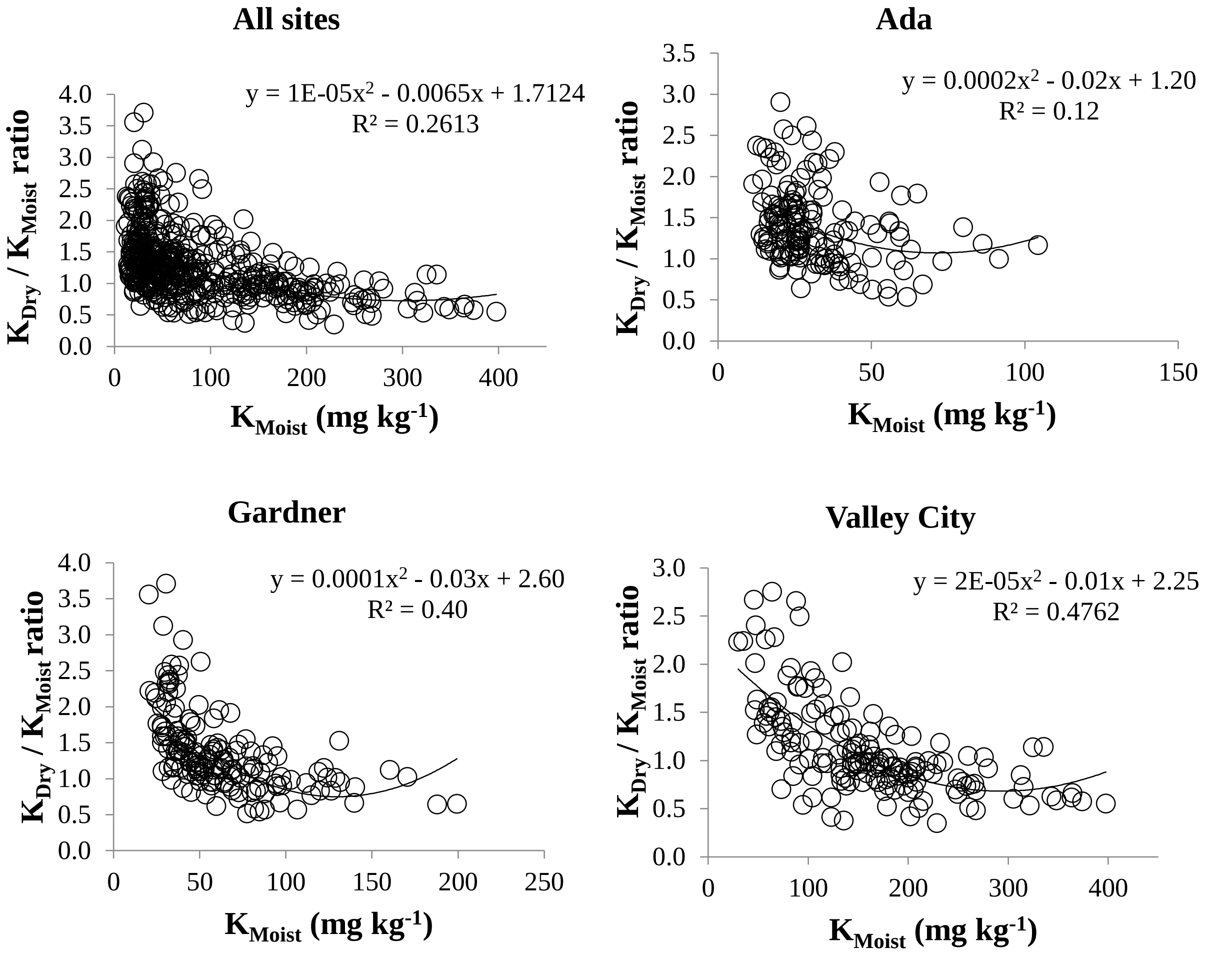


Figure 1. Relationship between NH_4OAc extracted exchangeable K based on field-moist(K_{Moist}) and air-dried (K_{Dry}) soil samples.


All sites

Ada

Gardner

Valley City

Figure 2. Regression analysis of ratio of K extracted from air-dried sample and field-moist sample and K extracted from field- moist soil sample at different experimental sites.

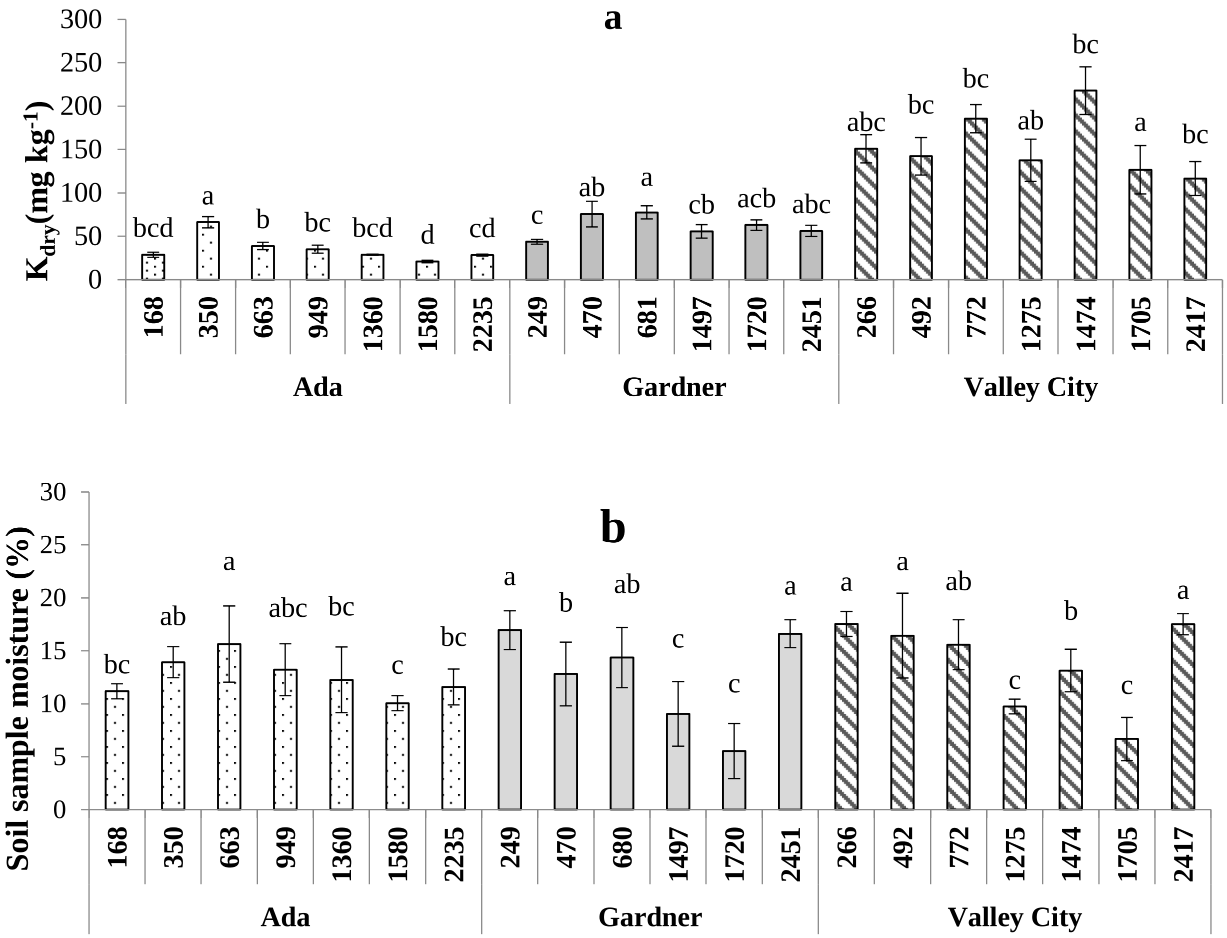


Figure 3. (a) Changes in plant available soil-K (mg kg^{-1}) of air-dried samples of K0 (control) treatment with growing degree days (GDD). Bars represent standard error (n=4). (b) Changes in gravimetric soil moisture (%) of soil samples of K0 (control) treatment with growing degree days (GDD). Bars represent standard deviation (n=4). Different lowercase letters within a site indicate significant differences at 0.05 significance level.

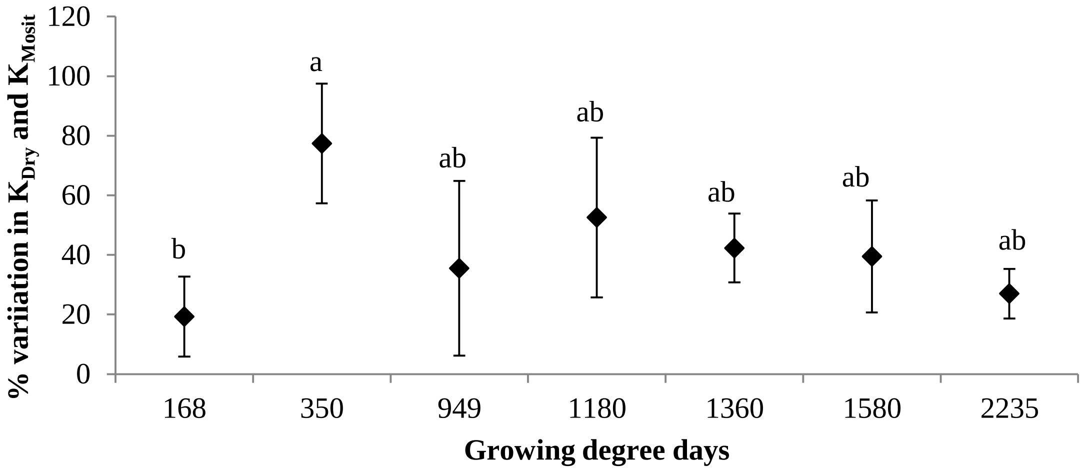
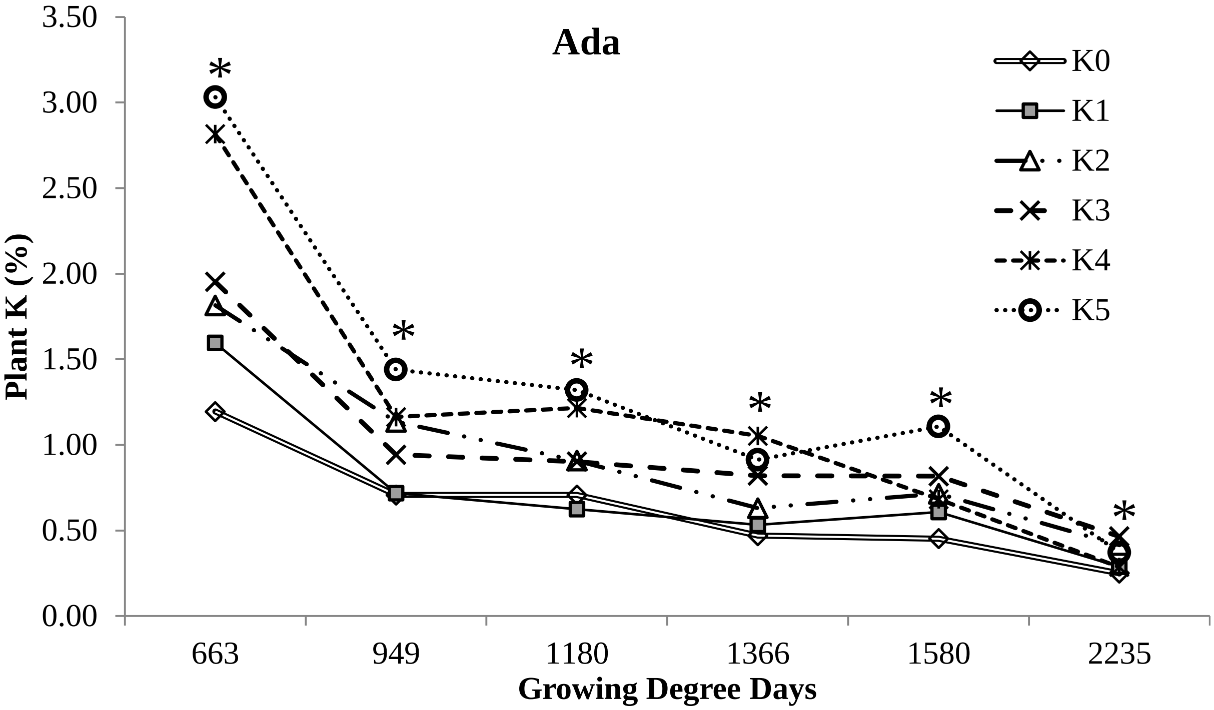


Figure 4. Percent variation of soil test K (mg kg^{-1}) due to air-drying soil as compared to field-moist soil-K ($K_{Moist)}$ during the growing season at Ada. Bars represent standard error (n=4). Different lowercase letters within a site indicate significant difference at 0.05 significance level.



* represents the significant differences of plant K (%) between different treatments on specified GDDs at 0.05 significance level.

Figure 5. Effect of application of different K-rates on plant concentration of K at particular corn growth stage (GDDs). Bars represent standard error (n=4). Different lowercase letters within a GDD indicate significant differences at 0.05 significance level.

EVALUATION OF SOIL POTASSIUM TEST FOR RECALIBRATION OF CORN RESPONSE CURVES

Abstract

Maintenance or improvement of soil fertility to ensure profitable yields is dependent upon the ability of soil testing procedures to predict relative crop response. The soil potassium (K) test methodology is under increased evaluation due to the soil sample drying effect, temporal variations of test results and inconsistent crop response to applied K fertilizers. Ten on-farm trials were conducted in 2014 in eastern North Dakota to determine the corn response to different K-fertilizer rates and to assess the variation of soil K test levels between air-dried (K_{Dry}) and field moist (K_{Moist}) soil samples during the corn growing season. Significant differences were observed between K_{Dry} and K_{Moist} soil K test results. The ratio of K_{Dry}/K_{Moist} showed high correlation with cation exchange capacity (r = 0.63), Organic matter (r = 0.61) and (Ca + Mg)/K ratio (r = 0.64) from the 1M ammonium acetate extractant, while pH, electrical conductivity, clay (%) and soil moisture showed non-significant correlation. On average, K_{Dry} resulted in higher soil K test levels than K_{Moist} and pattern of deviation was different for surface and sub-surface soil samples. Soil K analysis of samples collected during the fall and spring showed large enough variations to affect the soil test interpretation category which is used to make fertilizer recommendations. Corn yield increased significantly with applied K fertilizer at only three out of 8 sites with beginning K levels below the current critical level of 150 ppm, and one response was at a site with K level above the critical level. Therefore, use of either the K_{Dry} or K_{Moist} method alone may not be adequate to predict K response in some North Dakota soils.

Introduction

The corn growing belt of the United States is shifting north and west of the traditional Corn Belt due to changing climate patterns and improved corn hybrid varieties with short-season yield potential. In North Dakota, corn acreage has increased three fold in the last decade and farmers are becoming more interested in raising corn in some years due to higher economic returns compared to other crop choices (Fletcher, 2013). Corn yields have increased more than two folds in North Dakota in past three decades (NASS, 2011). The increase in corn yield in North Dakota is the net result of improved corn genetics and higher rainfall during the growing season (Ransom et al., 2004). Since higher yields are often accompanied with high nutrient removal from the soil (Bender et al., 2013), maintaining an adequate supply of nutrients is the next major challenge for the corn growers of North Dakota.

Providing an adequate supply of nutrients to corn is important for gaining yield benefits from other management practices. Corn is known to take up substantial amounts of K during the growing season. For instance, corn yielding 10.11 Mt/ha, can accumulate about 165 kg ha^{-1} of potassium (Hanway, 2007). Crop response to K is not as great as that of N, but K plays a vital role in every facet of crop growth. Positive correlation has been reported between K content of crops and photosynthesis, carbohydrate metabolism, lodging and disease resistance (Havlin et al., 2005). Potassium plays an important role in water uptake and helps in maintenance of yields in adverse climatic conditions such as drought (Hu and Schmidhalter, 2005; Cakmak 2005; Zorb et al. 2013). Therefore, maintaining an adequate level of K is important in the rain-fed agricultural system of North Dakota.

Soil testing is an important diagnostic tool for estimating nutrient supplying capacity of soils for growing crops. The most widely used procedure for estimating plant-available potassium is extraction of K from air-dried soil samples using 1*M* ammonium acetate (Haby et al., 1990). However, air-drying of soil samples is known to collapse or scroll up the clay lattice structure leading to release or entrapment of K depending upon soil solution K concentration and clay mineralogy (McLean and Watson, 1985), which can lead to over or under-estimation of soil-K levels (Wells and Dollarhide, 2000). To overcome this issue, Iowa State University has reintroduced the procedure of using field-moist soil samples for plant-available K analysis. Analysis of field-moist soil samples from Iowa for available K have resulted in improved correlation with corn yields compared to air-dried soil K analysis (Barbagelata et al, 2012). Therefore, performance of this new methodology needs to be reviewed with the soils of North Dakota.

Soil K results are not only subject to change due to the air-drying of soil samples, but K results may also vary depending on the date of sampling (Franzen, 2011).The seasonality effect is likely due to seasonal variability in moisture (high moisture in winters and comparatively low moisture towards the end of growing season when soils are driest), K leaching from crop residues, freezing and thawing, and microbial activity (Murrell, 2011). Switching from fall to spring sampling can lead to significant changes in soil K values, affecting the rate of K-fertilizer application (Vitko et al., 2010). Therefore, a better understanding of fluctuations of soil K level during the growing season will be helpful in improving K-fertilizer recommendations.

In North Dakota, fertilizer recommendations for corn were formulated in the late 1970’s and early 1980’s when yields were much lower than they are today. The new corn varieties for the region are much more productive and generally soil tests K levels are much lower today.

To address the increase in corn acres in North Dakota, the relevance of the current soil K test and response of modern corn hybrids to K fertilizer, a study was conducted with three main objectives:

1.) To compare soil K test values based on air-dried and field moist samples,

2) To determine the effect of sampling time on soil K test levels during the corn growing season.

3) To determine the corn response to applied K-fertilizer based on the predictability the soil K test.

Materials and methods

Site descriptions

During 2014, trials were conducted at ten locations in the eastern part of North Dakota including the Cass, Barnes, Richland and Sargent counties (Figure 6). All of these sites are involved in agricultural production with corn and soybean as the main crops. These areas have a humid-continental climate with mean precipitation about 55 cm and mean temperature varying about 5 ^{o}C (Mean of temperature and precipitation from 1981 to 2010).

Soil series descriptions are listed in Table 7. Most of these soils are developed from glacial lacustrine sediments, glacial outwash or till/moraines with somewhat poorly drained to well drained characteristics.

Experimental design

Each experimental location was established with a minimum distance of 30 m from the field edge. The experimental design of the trials was a randomized complete block design with six K-fertilizer treatments and four replications. Nine of the total sites received a fertilizer application of potassium chloride-KCl (0-0-60) at the rate of 0 (K0), 33.6 (K1), 67.2 (K2), 100.9

(K3), 134.5 (K4), K5-168.1 (K5) K_2O kg ha^{-1} while the Milnor site received K application of 0 (K0), 67.2 (K1), 134.5 (K2), 201.7 (K3), 269.0 (K4), K5-336.2 (K5) K_2O kg ha^{-1}. Dimensions of all plots were 9.14 m long by 3.05 m wide, with a 1.52 m of alley between each replication. The alleyways were cut out when the corn had 8-12 leaves. Planting and all agronomic and cultural operations were carried out by the farmers and were uniform for all plots within a location. Corn was produced at each site. Corn production practices are listed in Table 8. The farmer did not apply K fertilizer within the boundaries of experimental plots. When the grower applied K with N or P fertilizer, the plot area was excluded from his field application and N, P and any other nutrients determined necessary by the pre-plant soil test were broadcast applied by the researchers.

Soil sampling

Initial composite soil samples were collected from 0-15 cm depth from each site before planting and were analyzed for plant available nutrients and other basic soil properties. During the growing season, soil samples were collected from the control plots (plots with no K-fertilizer application) twice each month with an interval of about 15 days. A 2.5 cm diameter Hofer soil tube was used to take the samples from the 0-15 cm and 15-30 cm depth throughout the growing season .Soil samples were not taken from 15-30 cm on the second August sampling at Page and Valley City due to soil hardness. Soil samples were collected by taking four to five cores at each depth from the interior inter-row area within each plot. Samples from each depth were then composited and stored in zip-lock polythene bags to maintain the moisture level comparable to the field conditions. Samples were transported in a cooler to the laboratory and stored in laboratory refrigerator at 7 ^{o}C for one to three weeks.

Laboratory analysis

Initial soil samples

Initial composite soil samples were analyzed for pH, N, P, K, EC and organic matter by the NDSU Soil and Water Testing Laboratory using approved methods for the North Central Region of the USA (Table 9). Soil texture was determined by a hydrometer method (Elliot et al, 1999) and bulk density was analyzed using soil core method which involves taking soil sample with a soil probe with a defined volume and oven-drying the sample for at least 24 hours to obtain the mass of soil solids (Blake and Hartge, 1986**)**. Cation exchange capacity of the soil was determined by saturating the soil with 1*M* sodium acetate solution and then washing the soil with 90% ethanol solution and replacing the sodium ions from exchange complex using 1 *M* ammonium acetate (Chapman, 1965).

Methodology for K_{Dry} and K_{Moist}

Each soil sample was thoroughly mixed and subdivided into two sub-samples. One of them was analyzed with standard procedure of soil K test which involves air-drying of soil, grinding and passing through 2 mm sieve. Two grams of air-dried sample was extracted with 20 ml of 1*M* NH_40Ac, shaken for 5 min and filtered through Whatman No. 2 filter paper. Gravimetric water content of air-dried and field-moist soil was determined by oven drying a sub-sample at 105° C for at least 24 hours (Black, 1965). For K_{Moist}, sub-sample was not air-dried but was sieved through a 2 mm sieve. Two grams of sieved field-moist soil was treated with 20 ml of NH_4OAc by adjusting the molarity of extracting solution to 1*M* according to the moisture content of the sample. The resulting slurry was then shaken for 5 min and filtered through Whatman No-2 filter paper. Soil K concentration of filtrate was determined with necessary dilutions using a

Buck Scientific Atomic Absorption Spectrometer - Model 200A (Norwalk, CT, USA) using 766.5 nm wavelength.

Yield analysis

For yield analysis, corn ears were harvested from one of the middle two rows leaving first and last plant in each row. Ears were shelled and grain weight was measured in grams. Grain moisture and test weight were measured using Dickey-John Grain Moisture tester (GAC500 XT). Grain yield was calculated in kg ha^{-1} adjusted to 15.5% grain moisture content.

Statistical analysis

Statistical software - SAS 9.3 and SAS Enterprise Guide 4.3 were used for data analyses. A paired t-test was used to compare K_{Dry} and K_{Moist} results. Linear regression was imposed on K_{Dry} and K_{Moist} collectively over all sites as well as separately at very low, low, medium, high and very high K soil test K-levels. Pearson correlation coefficients were used to evaluate the relationship of K_{Dry}/K_{Moist} ratio with clay content, soil moisture, cation exchange capacity, organic matter, and (Ca + Mg)/K at $p<0.10$. Analysis of variance for yield response was calculated by SAS PROC GLM procedure using Randomized Complete Block Design with K-fertilizer rates as the main factor. Means of main effects were compared using Fisher’s least significant difference (LSD) at 90% confidence level.

Results and discussions

Basic soil properties

Initial soil test results of all experimental sites are presented in Table 9. The pH of soils ranged from moderately acidic to moderately alkaline (Soil Survey Division Staff, 1993). Based upon the EC levels, all sites had non-saline soils (Whitney, 1998). Seven of the total sites had sandy loam texture, while two of them had loam and one of the sites was categorized as loamy sand. Organic matter determined by loss of weight on Ignition method (Combs and Nathan, 1998) ranged from 1.5 % to 3.1%. The CEC level of soils varied from 10.6 to 23.1 cmol kg^{-1}.

Comparison of soil potassium test based upon air-dried and field moist samples

Soil test-K values of surface soil samples (0- 15 cm depth) determined by K_{Dry} ranged from 21 ppm to 824 ppm across all sites with an average of 93 ppm. The K_{Moist} test values had an average of 99 ppm with K values ranging from 14 ppm to 837 ppm. Based on the paired t-test results, overall K_{Dry} test results were significantly different from K_{Moist} levels for surface as well as subsurface soils. The results of paired-t test of each site at specific sampling time are listed in appendix (Table A). On average, K_{Dry} test of surface soils (0-15 cm) were 1.07 times higher in K compared to K_{Moist} values but the change of Soil K test varied between soils. Out of 366 soil samples, 47% showed a decrease in K content upon drying while 53% of samples showed an increase in K content. The ratio of K_{Dry}/K_{Moist} varied from 0.32 to 2.66 across all sites for surface soil samples. The K_{Dry} of sub-surface soil samples (15-30 cm) was 1.52 times greater in K content compared to K_{Moist}. Only 20% of the total samples showed a decrease in K content upon drying while 80% samples showed an increase in K values. The linear trend line deviated from the 1:1 line, with the greatest difference in the high and very high K range (Fig 7). Such variation in soil K levels of moist and dried soil samples had been observed in various earlier studies in Iowa (Luebs et al., 1956; Barbagelata and Mallarino, 2012)

Since the variation between K_{Dry} and K_{Moist} was different for different sites throughout the growing season, probable factors that might contribute to the difference in drying response were correlated to the K_{Dry}/K_{Moist} ratio and summarized in Table 10.

Soil moisture content was poorly correlated (r = -0.02) with K_{Dry}/K_{Moist} ratio. Similar conclusions were found by Barbagelata and Mallarino (2012) who determined $r^2 = 0.03$ between

K_{Dry} and K_{Moist} ratio and soil moisture in Iowa. Burns and Barber (1961) also showed no significant relation of soil moisture to release of exchangeable K upon soil drying.

Clay percentage of initial soil samples was not significantly correlated with ratio of K_{Dry}/K_{Moist} ($r = 0.45$, $p = 0.19$). Texture has previously been reported as the main factor for influencing of the degree of K release or fixation (Barber et al., 1961). However, clay type may have influenced the K_{Dry}/K_{Moist} ratio (Dowdy and Hutcheson, 1963). Presence of illite is usually responsible for release while montmorillonite (a smectitic clay) is known to fix K (McLean and Watson, 1985). Analysis of clay mineralogy of all these sites might be more helpful in explaining the release and fixation of K upon drying than the determination of clay content of soil *per se*.

Ratio of (Ca+Mg)/ K was significantly correlated with K_{Dry}/K_{Moist} with a correlation coefficient $r = 0.64$ ($p<0.10$). A relationship between (Ca+Mg)/ K and K_{Dry}/K_{Moist} was also reported by Barbagelata and Mallarino (2012). It signifies that the concentration of cations present in soil solution can affect the release and fixation of K upon drying. It occurs because cations such as calcium which show high affinity for negative charged clays can compete with potassium ions for K fixation inducing wedge zones within clay interlayers which results in a release of K ions into the soil solution (Sparks and Huang, 1985.)

K_{Dry} and K_{Moist} were significantly related for both depths (0-15 cm and 15-30 cm). Potassium levels of sub-soil samples were always lower in K compared to surface soil samples. Overall, sub-surface soils showed an appreciable increase in K levels in K_{Dry} compared to K_{Moist} tests of surface soil samples (Fig 7). Since the sub-surface soils are less prone to weathering compared to surface soils, thereby, they show a high potential of release of K upon drying (McLean and Watson, 1985).

K_{Dry} compared to K_{Moist} were significantly related in very low, low and very high category K soils (Fig. 8). When the K_{Dry} content was below 120 ppm, K was released upon drying. Dry K analysis gave lower K values when the soils had >120 ppm initial K. Barbagelata and Mallarino (2012) results agree with these data where an exponential decrease of K_{Dry}/K_{Moist} ratios was observed as soil K levels were increased.

Cation exchange capacity was correlated ($r= 0.63$, $p < 0.10$) with the K_{Dry}/K_{Moist} ratio. The CEC of a soil partially depends upon the amount and type of clay minerals. CEC was observed to be positively related to the change of K levels in the soil samples when exposed to drying (Barbagelata, 2006)

K_{Dry}/K_{Moist} ratio was significantly related to organic matter content with a correlation coefficient of $r = 0.61 (p<0.10)$. The relationship of organic matter (non- volatile organic compounds) to the release of K from soils upon drying is also noted by Welch and Flannery (1985) where organic compounds were found to retard the process of diffusion of K from interlayer of clay minerals.

As the season progressed, the difference between K_{Dry} and K_{Moist} also changed (Fig 9, 10 and 11). During April, with the exceptions of the Milnor and Arthur sites, K_{Moist} levels were greater than K_{Dry}. By late September, this trend was reversed; K_{Dry} levels were greater K compared to K_{Moist}.

Effect of time of sampling on soil K test results

Soil K_{Dry} levels of all sites decreased as the growing season progressed (Fig. 9, 10 and 11). This change was greater in Very high- K soils as compared to low K soils. There was a decrease of 265 ppm of K content at Valley City (Very high K –site) at the end of September as compared to those collected the previous April. In comparison, the decrease in K between April and September was only 25 ppm at Walcott West (Low K site). Greater variation of K levels in high K soils was also reported previously (Lockman and Molloy, 1984). Temporal change of soil K level was significantly correlated with soil moisture content at three sites (Buffalo, Walcott East and Wyndmere) while temporal changes of K at other sites were poorly correlated with soil moisture content. An increase in non-exchangeable K was also observed by September in all sites except at Valley City. The temporal variation of soil K can at least be partially attributed to changing soil moisture and a reversion of exchangeable K to non-exchangeable forms. In addition, plant uptake during the growing season and leaching of K after physiological maturity until harvesting have been reported as the other possible factors responsible for temporal K variations (Murrell, 2011).

Except at the Valley City site, soil K level of all sites dropped to Very low and Low categories with time (Table 11). Lower K levels during the fall may mislead farmers in applying fertilizer K rates for next year’s crop. However, soil K levels usually recover during the winter season due to freezing and thawing effect and leaching of K from the crop residues, and comparatively higher exchangeable K is observed in April and May (Fine et al, 1940; Mallarino, 2011). It may be necessary to construct critical levels for early fall and June soil sampling, where the soil K levels are more stable over a practical length of time.

Among the K_{Dry} and K_{Moist} soil test results, moist K soil levels were observed to be more variable within a corn growing season. Except for Arthur site, the coefficient of variation was greater for K_{Moist} soil results compared to K_{Dry} for all other sites (Table 12). Some possible reasons for higher variation in K_{Moist} results could be the manual error during molarity adjustments of extracting solution and while mixing of the moist samples to get a representative sample. This indicates that the current methodology used in determining soil K involving air-drying as a pre-treatment, have more potential in providing precise estimates of K levels over a growing season.

Corn response to applied K fertilizer rates

Experimental locations were quite variable in K- status, varying from 80 ppm to 485 ppm of plant available K_{Dry} levels. According to North Dakota's published K fertility categories (Franzen, 2010), five of the sites had medium soil K level, three had soil K levels in the very high category while low and high categories were represented by one site each. Potassium in the profile was stratified; surface samples (0-15 cm) had higher K levels than the sub-surface layer (15-30 cm). Corn grain yield was increased at four sites at the 10% probability level compared to plots receiving no K application. Maximum yield was obtained at 101 kg ha^{-1} fertilizer rate at 5 sites and at 67 kg ha^{-1} K rate over 4 out of 10 sites. None of the sites gave highest yield at maximum K fertilizer rate of 168 kg/ha of K. Only one site achieved maximum response at 134 kg/ha of K rate (Table 13).

The present K category recommendations based on K_{Dry} predicted crop response at only 3 of 10 locations. The K_{Moist} did not improve crop response prediction. In addition, the non-exchangeable K levels were not helpful in predicting crop response.

North Dakota experienced frequent rain in the spring and summer of 2014 (NDAWN, http://ndawn.ndsu.nodak.edu/) and good soil moisture conditions were maintained until August. Favorable soil moisture conditions promotes diffusion of K^+ ions (Schaff and skogley, 1982; Zeng and Brown, 2000; Mackay and Barber, 1985) and may have resulted in comparable yields of control plots as that of plots receiving K-fertilizer.

Based upon the observations of corn response to applied fertilizers, it can be concluded that a refined strategy is required to better predict corn yield response, or a different soil testing method is required for prediction improvement.

Summary and conclusions

Air-drying of soil samples prior to soil analysis of plant-available K significantly affected soil K test results. Change of soil K test levels due to air-drying was not consistently increased or decreased, and was found to be significantly related to cation exchange capacity, organic matter and (Ca+Mg)/K ratio of the soil samples. Soil moisture content, clay content, pH and EC showed minimal influence over K_{Dry}/K_{Moist} ratios. Time of soil sampling had considerable effect on soil K levels as well as K_{Dry}/K_{Moist} ratios. Temporal K- variations of soil samples collected in fall and spring were large enough to change the soil test interpretation category of a site for making fertilizer recommendations, unless soil test interpretations were constructed for different sampling times. Corn response to applied K fertilizer was site specific and only related to initial soil K levels at three of ten sites.

Based upon these results, it can be concluded that air-drying of soil sample prior to soil K analysis alters the actual plant available-K levels, but K_{Moist} is not a better predictor of corn yield response compared with K_{Dry}. The extent of K variation is dependent upon various factors and is likely to change over the time. Corn K response curves needs re-calibration in North Dakota. Moreover, soil K levels along with time of sampling, soil moisture dynamics and plant’s nutrient utilization potential should be taken into consideration when making K-fertilizer recommendations.

Table 7. Location and soil characterization information of K-experimental sites.

Location	Latitude and Longitude	Soil series	Taxonomic Classification
Buffalo	46º 55’ 12.582”N 97º25’18.338”W	Lankin-Gilby	Fine-loamy, mixed, superactive, frigid Pachic Hapludolls
Gardner	47º09’ 57.830”N 97º03’04.561”W	Galchutt	Fine, smectitic, frigid Vertic Argialbolls
Walcott E	46º 29’ 43.090”N 96º 53’05.196”W	Wheatville-Mantador-Delamere	Coarse-silty over clayey, mixed over smectitic, superactive, frigid Aeric Calciaquolls
Wyndmere	46º 15’38.809”N 97º 03’50.155”W	Glyndon	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Fairmount	45º 58’18.719”N 96º 37’08.665”W	Gardena	Coarse-silty, mixed, superactive, frigid Pachic Hapludolls
Milnor	46º 16’ 33.843”N 97º28’01.110”W	Embden-Wyndmere	Coarse-loamy, mixed, superactive, frigid Pachic Hapludolls
Walcott W	46º 35’16.546”N 97º02’50.090”W	Hecla-Garborg	Sandy, mixed, frigid Oxyaquic Hapludolls
Arthur	47º03’ 46.590”N 97º08’03.730”W	Glyndon- Tiffany	Coarse-silty, mixed, superactive, frigid Aeric Calciaquolls
Valley city	46º 53’17.843”N 97º 54’54.062”W	Barnes-Svea	Fine-loamy, mixed, superactive, frigid Calcic Hapludolls
Page	47º 09’38.226”N 97º 22’02.788”W	Swenoda	Coarse-loamy, mixed, superactive, frigid Pachic Hapludolls

Table 8. Corn production details for all experimental sites.

Site	Corn variety	Planting density	Sowing date	Harvesting date
		--seeds ha^{-1}--		
Buffalo	Dekalb DKC 36-30 RIB	80000	5/15/2014	10/8/2014
Gardner	NuTech 5B782	-	5/18/2014	9/24/2014
Walcott E	Dekalb DKC 36-30RIB	85000	5/30/2014	10/15/2014
Wyndmere	Dekalb DKC 43-10	87250	5/27/2014	10/14/2014
Fairmont	GC 95-33 VT3P	87340	5/23/2014	10/16/2014
Milnor	Pioneer 9917	81250	5/17/2014	10/14/2014
Walcott W	Dekalb 39-07	85000	5/23/2014	10/15/2014
Arthur	ProSeed 11-91 VT2P RIB	90000	5/18/2014	10/3/2014
Page	REA 2A550	-	5/25/2014	10/17/2014
Valley City	Crop Plan 2417 VT2	75000	5/5/2014	10/13/2014

Table 9. Soil test results of initial soil samples collected from 0-15 cm depth.

Location	**NO_3-N[†]**	**P[§]**	**K[¶]**	**pH [#]**	**EC[††]**	**OM[‡‡]**	**Clay[§§]**	**CEC[¶¶]**
	kg ha^{-1}	**---ppm--**			**dS m^{-1}**	**-----%-----**		**cmol kg^{-1}**
Buffalo	18	12	115	7.6	0.19	2.1	10.8	12.9
Gardner	10	13	110	5.9	0.09	2.2	11.3	12.5
Walcott E	6	3	105	7.4	0.45	2.3	11.5	12.1
Wyndmere	20	8	100	7.9	0.27	2.3	11.5	15.6
Fairmount	23	10	140	7.6	0.30	2.7	15.5	19.9
Milnor	9	18	110	6.2	0.43	2.2	7.30	14.1
Walcott W	10	16	80	5.8	0.10	1.5	4.50	10.6
Arthur	15	10	170	8.2	0.26	3.1	14.5	23.1
Page	20	12	200	7.5	0.48	2.4	10.0	14.9
Valley City	10	27	485	6.5	0.30	3.1	17.5	19.7

[†] NO_3-N extracted with water
[§] P extracted with Olsen procedure
[¶] K extracted with 1M ammonium acetate
[#] pH in water
[††] EC using 1:1 (soil: water) ratio
[‡‡] Organic matter – Ignition method
[§§] Clay (%) –Hydrometer method
[¶¶] Cation Exchange capacity estimated by 1N sodium acetate method.

Table 10. Relationship between various soil properties (0-15 cm depth) and ratio of soil test K result based upon air-dried and field-moist soil samples.

Soil properties	Number of observations (n)	Pearson correlation Coefficient (r)
Initial soil samples		
pH	10	0.29
Organic matter (%)	10	0.61*
Cation exchange capacity (cmol kg^{-1})	10	0.63*
Electrical Conductivity (dS m^{-1})	10	0.29
Clay (%)	10	0.45
Others		
† (Ca+Mg)/K ratio	40	0.64*
‡Soil moisture (%)	366	-0.02

* Significant at 90% confidence level

† Correlation of (Ca+Mg)/K ratio with K_{Dry}/K_{Moist} ratio of soil samples collected in first fortnight of September

‡ Correlation of soil moisture (%) with K_{Dry}/K_{Moist} ratio of all soil samples collected at fortnightly interval during the corn growing season.

Table 11. Changes in soil test K level between spring and fall soil sampling of control plots and its impact on soil test category.

Location	Change in Soil K level†	Soil Test Category*	
	ppm	Spring	Fall
Buffalo	84.5 ± 5.35‡	Medium	Very Low
Gardner	83.5 ± 4.44	Medium	Very Low
Walcott E	71.1 ± 2.87	Medium	Very Low
Wyndmere	67.2 ± 3.85	Medium	Very Low
Fairmount	107 ± 3.85	High	Very Low
Milnor	66.3 ± 2.53	Medium	Low
Walcott W	25.8 ± 10.6	Low	Low
Arthur	134 ± 9.66	Very High	Very Low
Page	139 ± 7.79	Very High	Very Low
Valley City	265 ± 67.0	Very High	Very High

† Change in soil test K level calculated as spring minus fall sampling soil K test results.
‡ Standard deviation of soil K change between four replications of a control plot (n = 4).
*Soil test categories are given for corn in Franzen (2010) Extension Bulletin which include five categories as Very Low (0-40 ppm), Low (41-80 ppm), Medium (81-120 ppm), High (121- 160 ppm) and Very high (161+).

Table 12. Summary of soil K tests based on air-dried and field –moist soil samples during the growing season.

Location	Dry Soil K test (ppm)			Moist Soil K test (ppm)		
	Average	Std. Dev.	CV	Average	Std. Dev.	CV
Buffalo	55.95	25.18	0.45	54.15	36.31	0.67
Gardner	54.78	25.39	0.46	59.93	47.59	0.79
Walcott E	63.50	24.39	0.38	63.59	29.73	0.47
Wyndmere	52.58	21.71	0.41	53.15	36.79	0.69
Fairmount	66.32	31.00	0.47	56.63	35.62	0.63
Milnor	83.70	28.03	0.33	81.45	30.56	0.38
Walcott W	53.06	14.68	0.28	64.04	35.24	0.55
Arthur	82.29	40.16	0.49	83.84	38.38	0.46
Page	115.9	47.96	0.41	156.9	109.2	0.70
Valley City	360.9	108.3	0.30	391.3	136.5	0.35

Table 13. Corn grain yield response of all sites to applied K-fertilizer treatments.

	Treatment (kg K_2O ha^{-1})						
Location	**0**	**33**	**67**	**101**	**134**	**168**	**LSD**
			------(Grain yield, Mg ha^{-1})-----------				
Buffalo	8.69 c	9.64 a	8.88 bc	9.86 a	9.38 ab	9.31 ab	0.57*
Gardner	8.94 b	10.91 a	9.75 ab	11.0 a	10.1 ab	9.84 ab	1.43*
Walcott E	7.07	7.80	7.26	7.93	7.05	7.32	ns‡
Wyndmere	8.44	9.42	10.56	8.42	9.31	8.18	ns
Fairmount	10.1 b	10.9 ab	11.3 ab	11.0 ab	11.4 a	10.8 ab	1.27*
Walcott W	8.18	8.40	8.85	8.54	8.78	8.70	ns
Arthur	9.77 c	10.2 bc	11.3 ab	11.7 a	11.1 ab	10.6 abc	1.26*
Page	9.34 a	8.85 ab	9.74 a	9.49 a	9.03 ab	8.32 b	1.01*
Valley City	9.74	9.62	9.64	11.1	10.0	10.7	ns
	Treatment (kg K_2O ha^{-1})						
	0	**67**	**134**	**202**	**269**	**336**	
Milnor	12.3 ab	13.0 a	11.9 b	11.8 b	12.0 ab	12.8 ab	1.06*

*Significant at 90 % confidence level

† Different letters indicate significant differences at specified significance level.

‡refers to non-significant corn yield response to applied K- treatments.

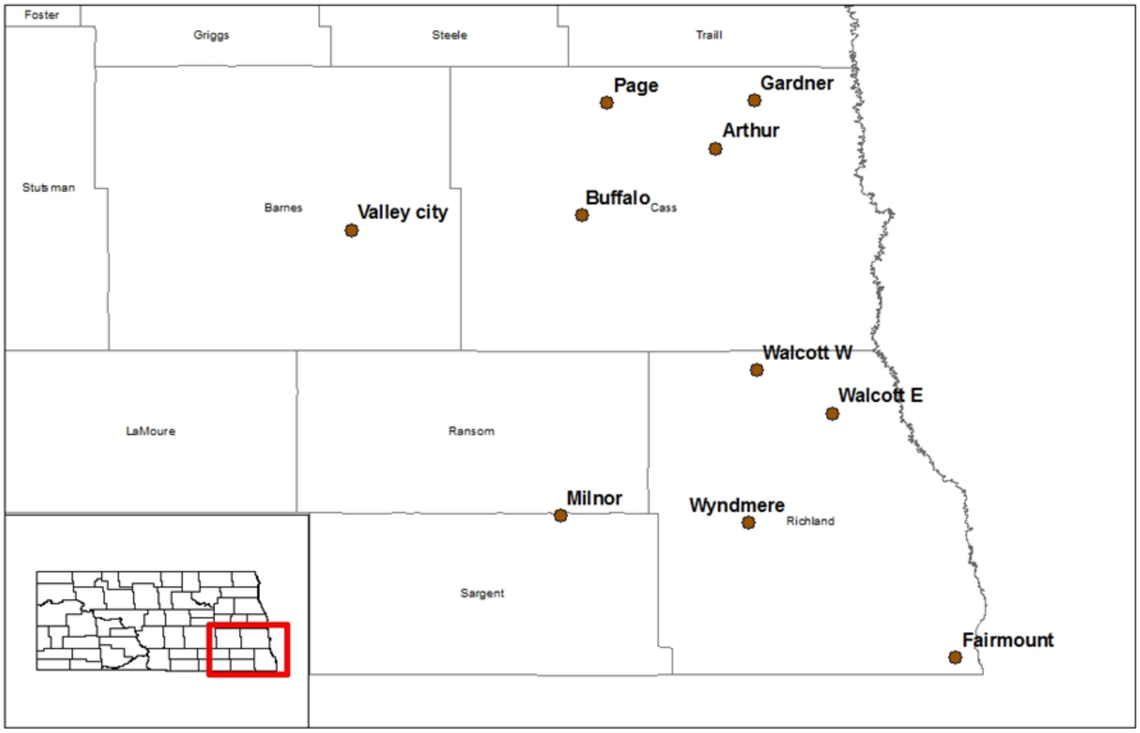


Figure 6. North Dakota map showing experimental sites of 2014.

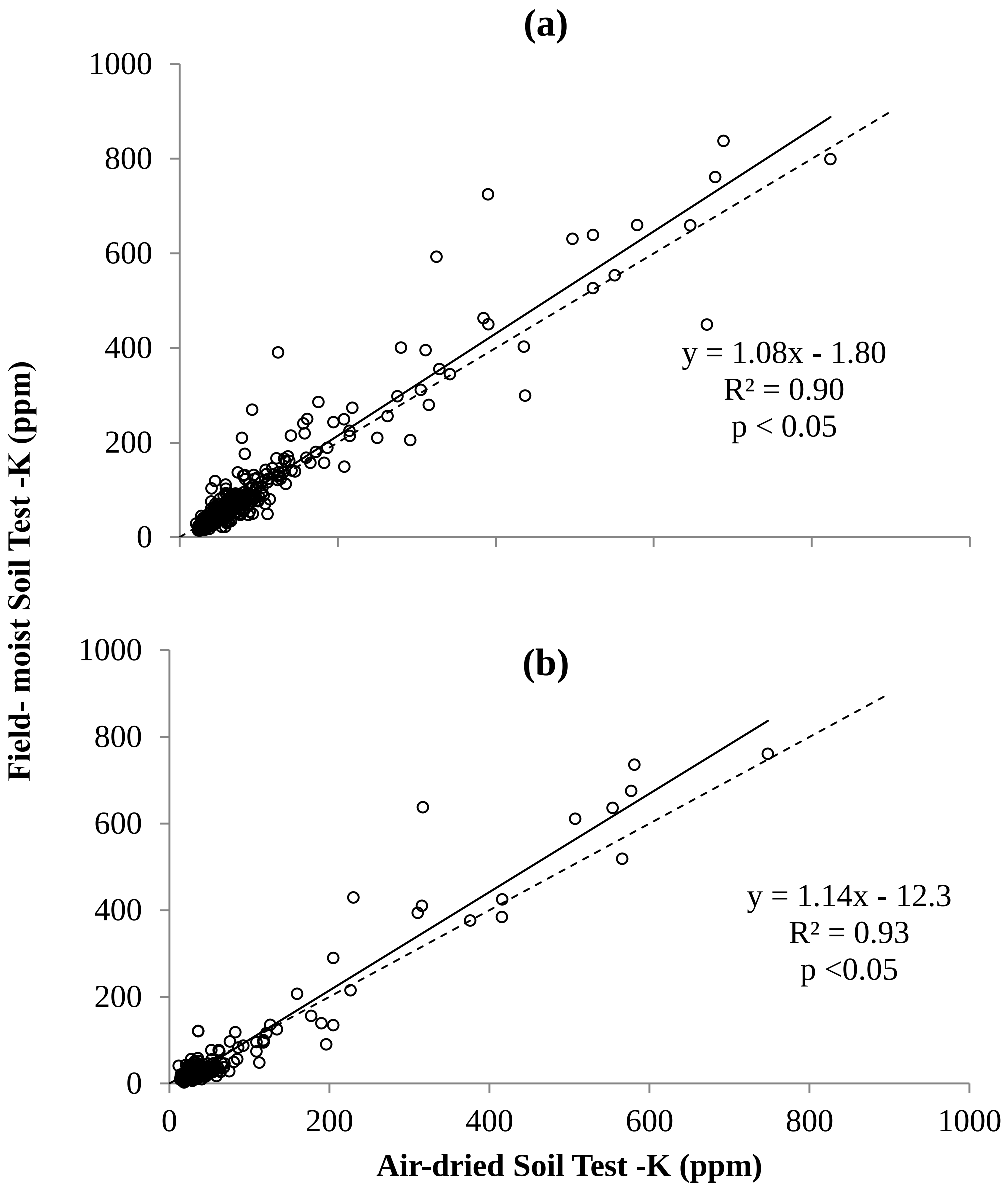
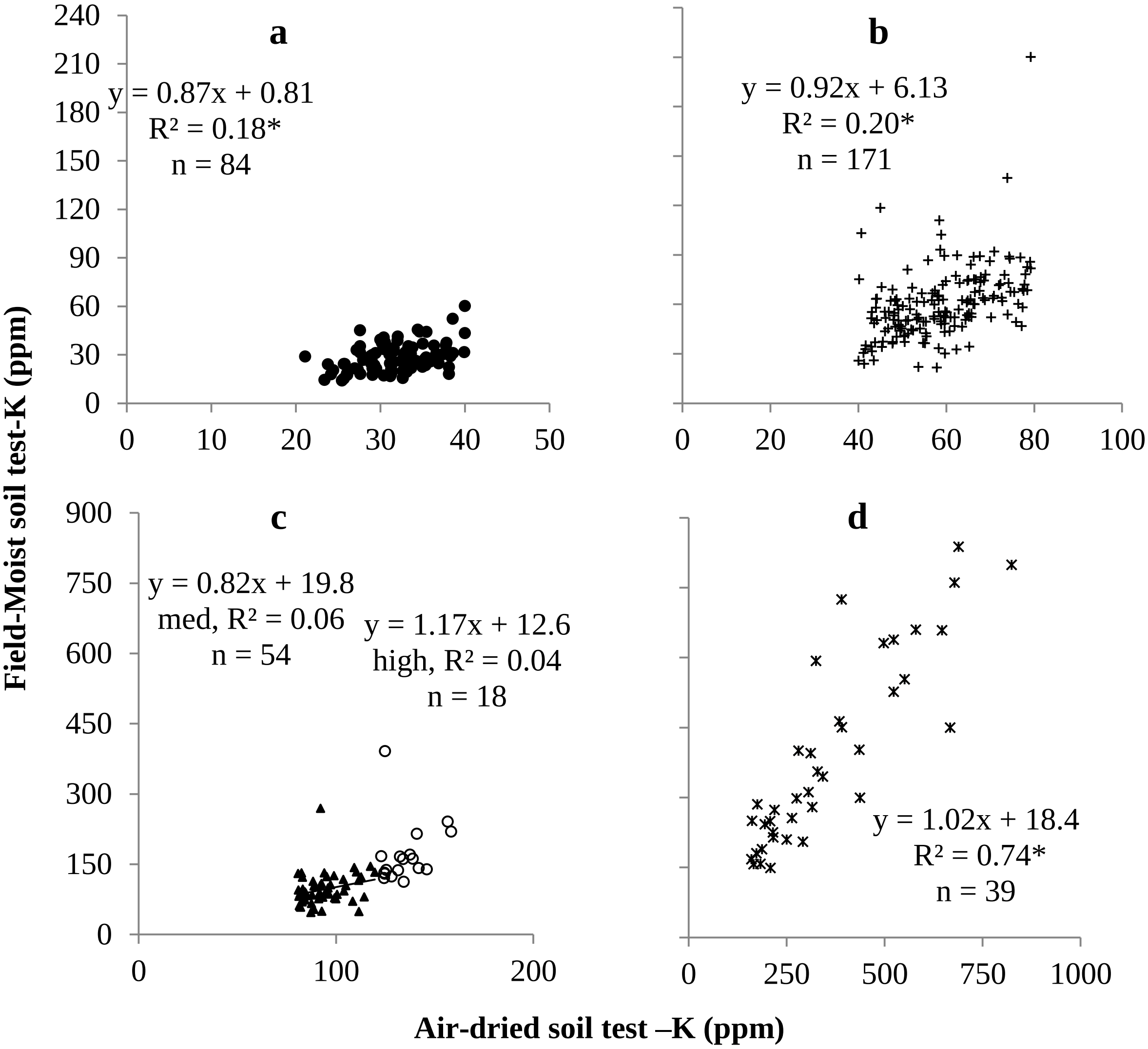


Figure 7. Relationship between soil K-test values based upon air-dried and field-moist soil samples of a) 0-15 cm and b) 15-30 cm depth.



*refers to significant relation between soil test K results based upon air-dried and field-moist soils at 95% confidence level.

Figure 8. Relation of soil test K results based upon air-dried and field-moist soil samples of

a) Very low (0-40 ppm) soil K samples
b) Low (41-80 ppm) soil K sample
c) Medium (81-120 ppm) and high (121-160 ppm) soil K samples
d) Very high (>161 ppm) soil K samples.

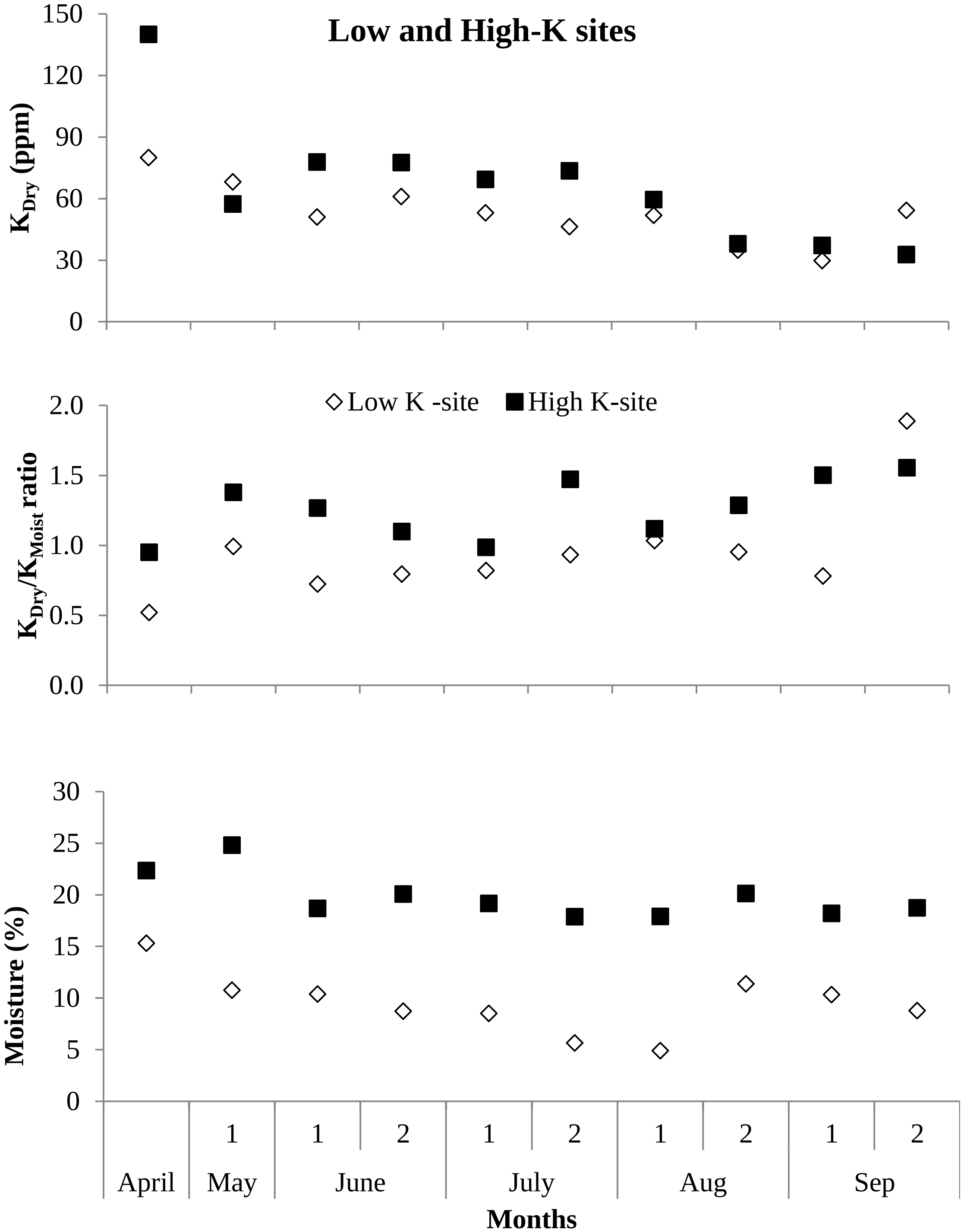


Figure 9. Effect of time of sampling on soil test- K (ppm), K_{Dry}/K_{Moist}t ratio and soil moisture (%) at Walcott W (low K site) and Fairmount (High K site.)

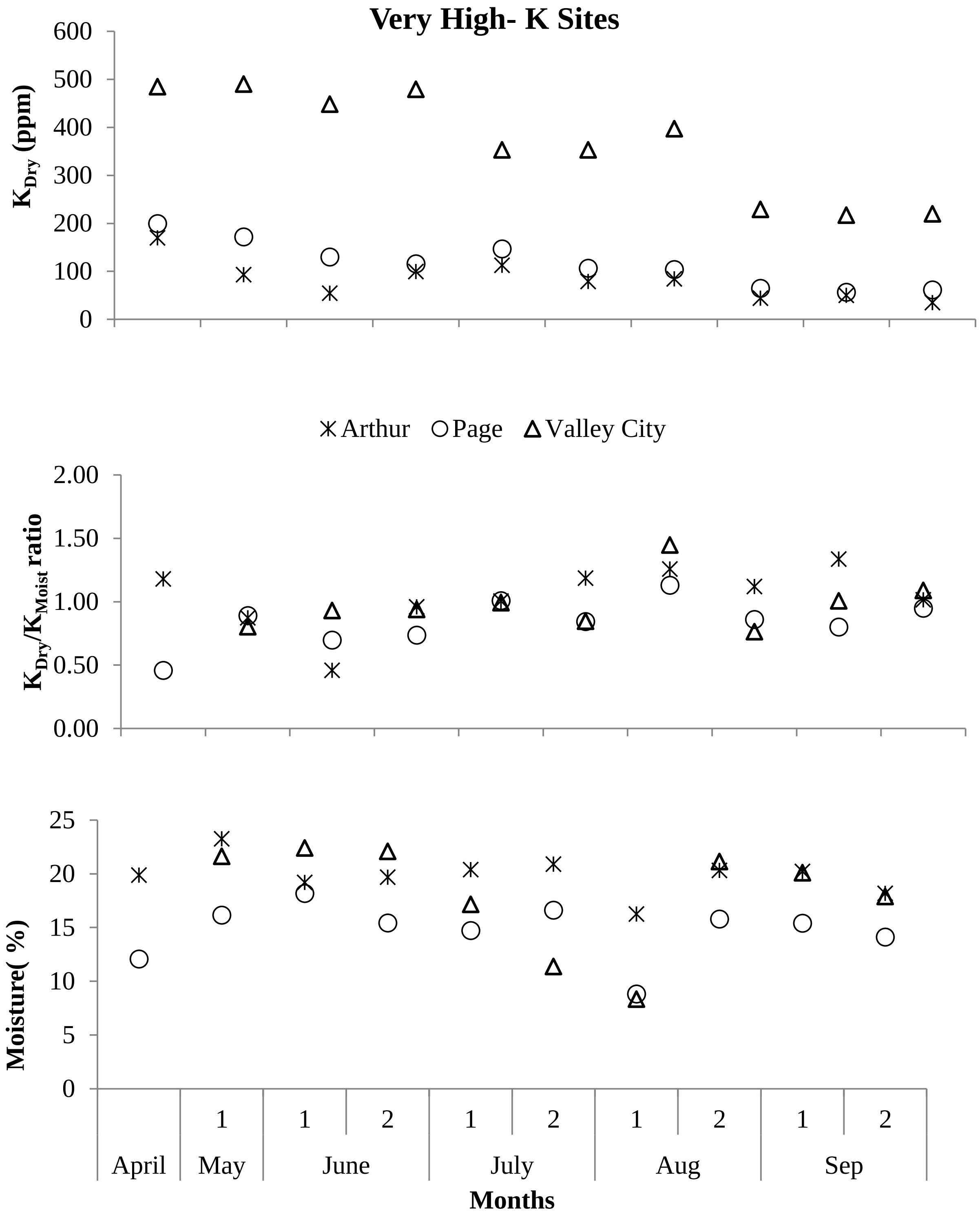


Figure 10. Effect of time of sampling on soil test- K (ppm), K_{Dry}/K_{Moist} ratio and soil moisture (%) at Very high K testing sites (Arthur, Page and Valley City).

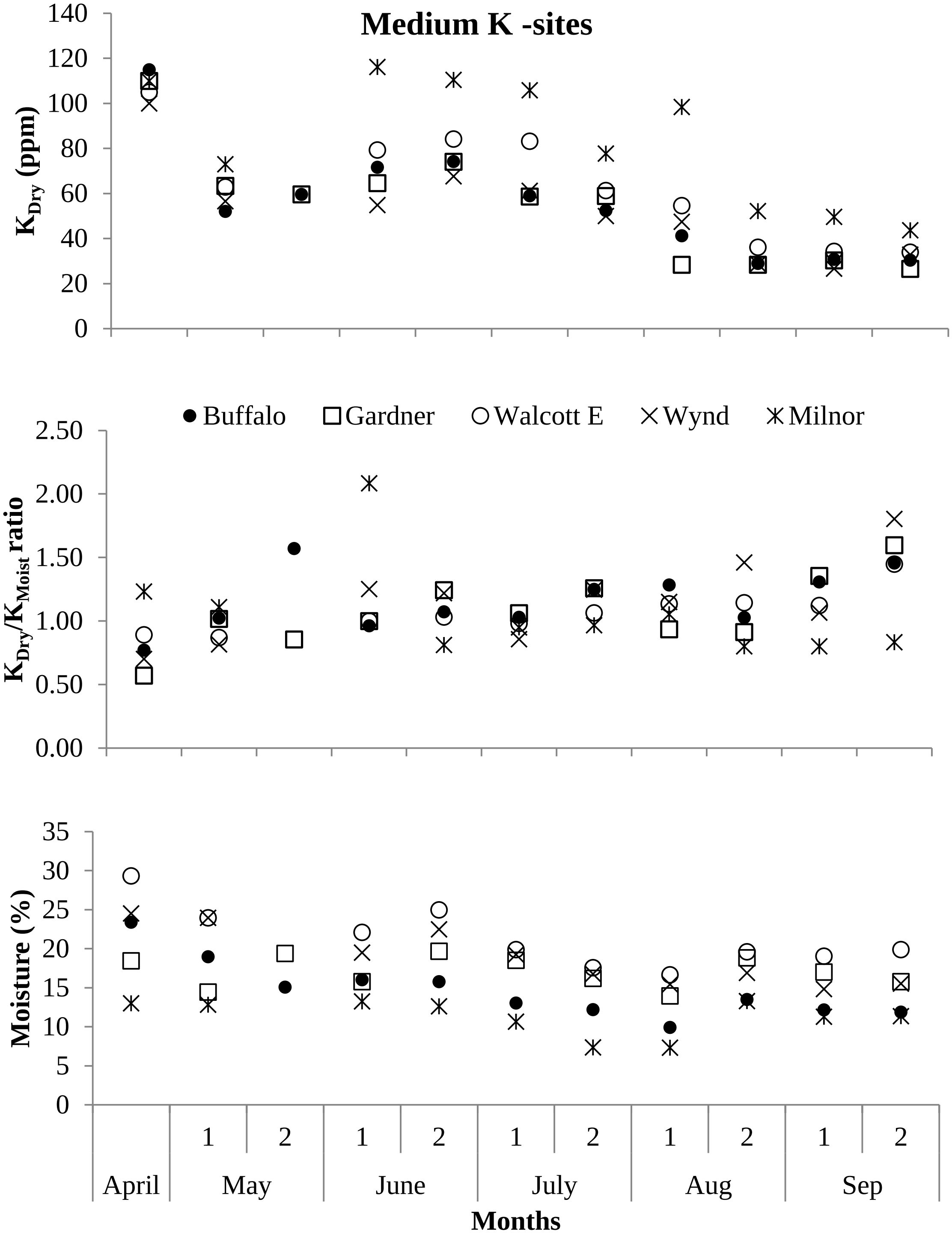


Figure 11. Effect of time of sampling on soil test- K (ppm), K_{Dry}/K_{Moist} ratio and soil moisture (%) at medium K testing sites (Buffalo, Gardner, Walcott E, Wyndmere and Milnor).

GENERAL SUMMARY AND CONCLUSIONS

The potassium fertilizer recommendation for corn needs a review in North Dakota. Significant differences between K_{Dry} and K_{Moist} levels revealed that air-drying of soil samples prior to the soil analysis can provide misleading levels of plant available K levels. Deviation of soil K results of air-dried and field-moist soils is dependent upon the specific soil properties such as concentration of other salts, soil sample depth, etc. The soil K levels show high temporal variations, therefore, time of soil sampling must be considered while formulating fertilizer recommendations. Corn's non-response to applied fertilizer at sites having less than the critical level of potassium has questioned the existing critical level of 150 ppm for corn. A more intensive collection of information such as soil mineralogy, soil moisture dynamics and plant nutrient efficiencies may prove to be more helpful in improving the predictability of corn yield response to applied fertilizers.

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APPENDIX. RESULTS OF PAIRED-T TEST OF EACH SITE AT SPECIFIC SAMPLING DATE

Sampling[†]	Depth	Buffalo	Gardner	W_E	Wyndmere	Fairmount	Milnor	W_W	Arthur	Page	V. City
	-cm-					p-values (α = 0.05)					
5-May	0-15	0.80	0.76	0.08	0.21	0.32	0.31	0.83	0.29	0.06	*
	15-30	*	*	0.90	0.80	0.10	0.59	0.77	0.22	*	0.06
22-May	0-15	0.09	*	NA[‡]	NA	NA	NA	NA	NA	NA	0.13
	15-30	*	*	NA	NA	NA	NA	NA	NA	NA	0.78
5-June	0-15	0.48	0.90	0.94	0.10	0.17	*	*	*	0.10	0.29
	15-30	*	*	*	*	*	0.16	0.24	*	0.12	0.65
24-June	0-15	0.43	*	0.89	*	*	0.12	*	0.17	0.07	0.54
	15-30	*	*	*	*	*	*	0.41	*	0.76	0.46
7-July	0-15	0.46	0.40	*	*	0.55	0.57	*	0.87	0.91	0.70
	15-30	*	*	*	0.35	*	*	0.27	*	*	0.30
21-July	0-15	0.11	*	0.92	*	*	0.51	0.22	*	0.16	0.06
	15-30	*	*	*	*	*	0.67	0.73	*	0.19	0.56
8-Aug	0-15	0.09	0.48	0.11	0.11	0.09	0.85	0.79	*	0.76	0.09
	15-30	*	*	0.32	0.15	*	0.18	0.38	*	NA	NA
27-Aug	0-15	0.09	0.10	0.96	*	*	*	0.27	0.45	0.12	*
	15-30	*	*	0.96	*	*	0.71	*	*	0.69	0.22
12-Sep	0-15	*	*	0.37	0.17	*	*	*	*	*	0.69
	15-30	*	*	*	*	*	0.83	0.87	*	0.06	0.87
30-Sep	0-15	*	*	*	*	0.09	0.20	*	0.87	0.41	0.11
	15-30	*	*	*	*	0.26	*	*	*	*	0.07

*refers to significant difference between the mean of K_{Dry} and K_{Moist} at α = 0.05.

[†] specified sampling date is variable by ±3 days.

[‡] NA – no soil sampling was done on that date.