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Alkaline Hydrolyzable-Nitrogen, Seeding Date, and Clay-Fixed Ammonium as Potential Indicators of Rice Response to Nitrogen Fertilization in Arkansas

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Alkaline Hydrolyzable-Nitrogen, Seeding Date, and Clay-Fixed Ammonium as Potential Indicators of Rice Response to Nitrogen Fertilization in Arkansas

A dissertation submitted in partial fulfillment
of the requirements for the degree of
Doctor of Philosophy in Crop, Soil, and Environmental Sciences

by

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ABSTRACT

Several potential indicators of rice (*Oryza sativa* L.) response to nitrogen (N) fertilization include the soil's alkaline hydrolyzable-N (AH-N) concentration, seeding date, and the soil's clay-fixed NH_4–N content. Three studies were conducted to: (1) correlate AH-N, determined using Illinois Soil Nitrogen Test (ISNT) or Nitrogen Soil Test for Rice (N-STaR), to plant parameters of interest and develop a fertilizer N rate calibration curve capable of predicting the 95% relative grain yield (RGY) fertilizer N rate for rice grown on clayey soils; (2) evaluate the influence of seeding date and N fertilization on rice plant growth and grain yield; and (3) evaluate the plant-available portion of clay-fixed NH_4–N using N-STaR and quantify total clay-fixed NH_4–N using dry combustion. Alkaline hydrolyzable-N, quantified using ISNT or N-STaR in the first study, explained 83% of the variability in calibration based on a 0 to 30-cm sampling depth. The second study revealed that grain yield exhibited a tendency to be greater for rice seeded in April or May compared to June seeding dates in Arkansas. Results of the third study indicated a low percentage (4–6%) of total clay fixed NH_4–N was predicted to be plant-available NH_4–N based on N-STaR. Fertilizer N rate recommendations based on the ISNT or N-STaR soil test methods evaluated in the first study revealed the potential of soil-based tests to improve economic, agronomic, and environmental aspects of rice produced on clayey soils. Seeding date selection can influence rice grain yield which may in turn influence fertilizer N management. However, the fertilizer N rate required to maximize grain yield when seeding date was delayed remained unclear following completion of the second study and continued research is required to identify the influence of seeding date on the yield-maximizing, fertilizer N rate for rice grown in Arkansas. The recovery of clay-fixed NH_4–N by N-STaR indicated a portion of total clay-fixed NH_4–N was expected to be available for plant uptake. However, additional research is required to verify the low recovery of clay-fixed NH_4–N and the influence of clay-fixed NH_4–N on the quantity of AH-N determined by N-STaR.

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CHAPTER ONE

INTRODUCTION

Rice (*Oryza sativa* L.) was harvested on 520,000 ha in Arkansas and accounted for over 47% of the estimated 1.1 million ha of rice produced in the United States during 2012 (National Agricultural Statistics Service, 2013). Nitrogen (N) is the plant nutrient that is most often applied in the greatest amount in U.S. rice production and urea [$CO(NH_2)_2$] fertilizer is the most common form of N applied in the drill-seeded, delayed flood production system due to its low cost and high N analysis (Norman et al., 2003). The high input cost of rice grown using drill-seeded, delayed flood cultural practices (e.g., seeding, irrigation, fertilization, herbicides, etc.) necessitates efficient management during the growing season to optimize producer profitability and environmental stewardship. Nitrogen fertilizer represents a major expense during the growing season and volatile urea fertilizer costs have producers continually searching for management strategies to improve the return on their investment in fertilizer N. Arkansas is the leading rice producing state in the U.S. and implementing sustainable agricultural practices, including efficient fertilizer N management, within the Arkansas rice industry could signal to other rice producing states that environmentally acceptable production practices are profitable and benefit the U.S. rice industry.

Sustainable management practices require economic, environmental, and social acceptance to remain viable for production agriculture. The ultimate goal of a soil-based N test is to prescribe site-specific fertilizer N rates that consistently maximize grain yield while limiting the misapplication of N fertilizers. Nitrogen soil test methods have typically identified the chemical forms of N utilized by the plant during the growing season and then developed an appropriate biological or chemical methodology to analyze this labile fraction of soil N. In general, biological and chemical methods have been used to provide an index of N availability that is correlated to mineralization of soil organic-N or to the fertilizer N responsiveness of agricultural crops. Biological methods, such as aerobic and anaerobic soil incubations, have historically provided the most consistent prediction of soil N mineralization. However, soil incubation procedures are time consuming, often requiring days to complete, therefore biological methods would not be conducive to the sample throughput required of a routine N soil test method.

Quantification of inorganic ammonium and nitrate ($NH_4^+ + NO_3^-$)–N has been the preferred chemical method of soil N analysis because the analytical method is simple, rapid, and can be performed at a low cost. Chemical methods are quicker to perform than biological methods and would be a preferred alternative for laboratories offering routine soil N testing services. However, inorganic-N is subjected to numerous loss mechanisms (e.g., leaching, denitrification, volatilization, clay-mineral fixation, etc.) which will influence the quantity of N available for plant uptake.

The disadvantages of inorganic-N analysis were quickly recognized and Magdoff et al. (1984) noted that concerns with the pre-sidedress soil nitrate test (PSNT) included the spatial and temporal fluctuation of soil inorganic-N which limits the time soil samples can be taken during the growing season. The logistical constraints of soil sampling for inorganic-N testing and the time constraints of biological methods of N soil testing led to the evaluation of chemical methods that could estimate potentially mineralizable soil N. Khan et al. (2001), with the development of the Illinois Soil Nitrogen Test (ISNT), stimulated interest in pursuing a chemical method that could provide an index of N mineralized during the growing season. The sample-to-sample variability and lengthy analysis time (~5 h) of ISNT initiated the development of the direct steam distillation (DSD) procedure of Bushong et al. (2008) that was subsequently identified as a suitable alternative to the ISNT (Roberts et al., 2009).

Fertilizer N use efficiency, assessed using ^{15}N isotopic tracer technics, of drill-seeded, delayed flood rice has been thoroughly documented and exhibited a range of 65 to 75% (Wilson et al., 1989; Norman et al., 1992; Guindo et al., 1994; Wilson et al., 1994; Bufogle et al., 1997) when fertilizer N was properly managed during the growing season. The identification of optimal seeding dates allows producers to make informed management decisions at planting that will determine the timing of fertilizer N application and other agronomic practices. Fertilizer N application timing is as important as the selection of the fertilizer N source and application rate to achieve efficient fertilizer management (Norman et al., 2003). The influence of seeding date on the rate of rice grain yield decline as well as disease incidence and severity has been documented in Arkansas (Slaton et al., 2003; Slaton et al., 2007). However, very little is currently known about the influence of seeding date on plant growth and grain yield in response to N fertilization for modern rice cultivars grown using the drill-seeded, delayed flood production system.

Nitrogen must be available for plant uptake during the rapid vegetative growth phase and should be adequately supplied or already taken up by the rice plant during reproductive growth to achieve maximum grain yield (Norman et al., 2003). Therefore, with as much as 80% of the total fertilizer N rate applied during the rapid vegetative growth phase (Roberts et al., 2013), management of fertilizer N applied early in the growing season is crucial to the efficient utilization of native soil and N applied later in the growing season for modern rice cultivars grown in Arkansas. Physiological stress, as a result of seeding date, may impact seed germination, stand uniformity, and biomass accumulation which could ultimately influence N use efficiency in the drill-seeded, delayed flood rice production system. The current lack of information regarding the response of modern rice cultivars to N fertilization as seeding date is delayed in Arkansas necessitates a review of the physiological response of rice to seeding date and the uptake of fertilizer N during physiologically important plant growth phases.

Rice response to N fertilization is influenced by the amount of N that is available or will become available for plant uptake during the growing season. Planting rice within an optimal time period is a management practice that can establish the environmental conditions necessary to optimize grain yield and seeding date selection may also influence fertilizer N use efficiency. However, all of the applied N will not be taken up by the rice crop even after implementing the best management practices due to microbial immobilization, leaching, denitrification, ammonia (NH_3) volatilization and clay mineral fixation (Norman et al., 2003). Clay mineral fixed NH_4–N will not readily leach through the soil profile and is not immediately available for microbial or plant utilization. Thus, even though clay-fixed NH_4–N remains in the soil it is the only mineral form of N that is considered to not be immediately available for crop uptake (Norman et al., 2003). Understanding the factors leading to the formation of clay-fixed NH_4–N may help identify fertilization practices (e.g., application of K^+ prior to N fertilization) which limit the amount of fertilizer located in the soil as clay-fixed NH_4–N. There are several factors that influence clay-fixed NH_4–N content and the most prominent factor controlling clay mineral fixation is the clay mineral composition of agricultural soils.

Nonexchangeable NH_4–N forms rapidly upon fertilizer N addition and compared to its rate of formation this “recently fixed” fertilizer N is released as an available form of N at a much slower rate (Kowalenko and Cameron, 1976; Sowden, 1976). The potential incorporation of fertilizer N into a fraction of total soil N that is not immediately available for crop uptake may influence the crop’s response to N fertilization. Consequently, numerous analytical methods were developed to gain an understanding of the factors (e.g., soil organic matter) that influenced clay mineral fixation of fertilizer N as well as the influence of soil depth on clay-fixed NH_4–N.

The removal of organic-N compounds along with clay-fixed NH_4–N has limited the precision of previous analytical methods which have attempted to quantify clay-fixed NH_4–N (Silva and Bremner, 1966). Historically, the method of Silva and Bremner (1966) was widely adopted for the quantification of clay-fixed NH_4–N due to the quantitative removal of inorganic and organic-N using alkaline potassium hypobromite (KOH–KOBr) prior to the determination of clay-fixed NH_4–N. The automation of elemental N analysis using modern laboratory instrumentation led Liang et al. (1999) to modify the method of Silva and Bremner (1966). The modification proposed by Liang et al. (1999) was shown to be highly correlated ($r^2 = 0.97$) to the original procedure, eliminated the use of strong chemical reagents and increased analytical precision for isotopic N analysis.

The following review describes the development of soil test methods which seek to identify plant-available forms of soil N, the influence of seeding date on plant growth and grain yield of rice, clay-fixed NH_4–N as a component of the N cycle, and analytical methods used to quantify clay-fixed NH_4–N. Specifically, the following review describes the development of two chemical methods, the ISNT method of Khan et al. (2001) and the DSD procedure of Bushong et al. (2008). Additionally, rice seeding date studies will be reviewed to examine environmental conditions (e.g., nighttime temperature) related to noticeable reductions in grain yield and how delayed seeding dates may influence rice response to N fertilization. Finally, the following review will evaluate the dynamics of clay fixed NH_4–N in field and laboratory studies as well as the chemical and physical mechanisms leading to the release of clay-fixed NH_4–N.

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CHAPTER TWO

LITERATURE REVIEW

Nitrogen Soil Test Methods

Several decades of soil fertility research have been devoted to identifying biological and chemical methods that can accurately predict soil nitrogen (N) mineralization or the fertilizer N requirements of agricultural crops. The ultimate goal of these biological and chemical N soil test methods is to provide an index of N availability which can be used to guide site-specific fertilizer N application rates. Three general categories of N soil test methods have been widely recognized and are typically considered as either i) biological methods (e.g., aerobic or anaerobic soil incubation); ii) chemical methods (e.g., neutral salt extraction); or iii) methods that directly analyze a specific fraction of soil N (Magdoff et al., 1984). There are numerous advantages and disadvantages associated with biological and chemical methods that attempt to quantify plant available-N. In general, biological methods were preferred despite the lengthy time required to complete analysis due to the belief that chemical methods were ineffective for quantifying a biologically labile fraction of soil N (Wang et al., 2001). However, the development of rapid chemical N soil test methods was pursued with the realization that chemical methods, rather than biological methods, would ultimately be preferred and utilized routinely by soil testing laboratories.

Biological Methods

Microbial organisms found in the soil carryout the heterotrophic phase of the N cycle through the process of mineralization (Jansson and Persson, 1982). Measurements of mineralization are taken by subjecting a soil subsample to salt extraction (e.g. 2 mol L^{-1} KCl) and net N mineralization is determined from the difference between final and initial inorganic-N concentrations of the extracted solution following incubation (Hart et al., 1994). The challenge facing researchers has been to accurately represent a predominately biological process, occurring under spatially and temporally variable conditions in the field, using the optimized conditions typically established and maintained in the laboratory during the period of soil incubation. Biological methods of soil analysis conducted in the laboratory, while not being free of analytical problems, have been developed and refined to provide an index of N availability for a wide range of soils. For example, utilizing a long-term aerobic incubation of 39 soils, Stanford and Smith (1972) were able to successfully introduce the concept of potentially mineralizable-N as a result of measuring net N mineralization during a 30-week study. The authors concluded that the cumulative amount of N mineralized during an aerobic incubation could be used to establish a rate of N mineralization. Since its development aerobic incubation has become the standard procedural method against which other methods are assessed and has been used to determine the impact of management practices on N mineralization (Schomberg et al., 2009).

Short-term anaerobic soil incubation is another biological method that has been widely adopted to assess potentially mineralizable-N and can be used for comparison of more rapid analytical procedures (Waring and Bremner, 1964). The reducing environment, which can be established and maintained for the period of anaerobic soil incubation, favors the formation of NH_4–N. This is advantageous due to the fact that NH_4–N is relatively stable in a highly reduced environment and should theoretically be subjected to fewer loss mechanisms than NO_3–N. Differences in N transformations were evaluated using a comparison of anaerobic and aerobic soil incubations by Zia et al. (2001). The authors found a lower concentration of NH_4–N (< 5 µg N g^{-1}) in the aerobic soil due to its oxidation to NO_3–N and the conversion of NH_4–N to NO_3–N was inhibited by the anaerobic environment of flooded soil. The rapid analysis and measurement of primarily NH_4–N under anaerobic soil conditions represents an improvement over long-term soil incubation. For example, Sims et al. (1967) modified the longer soil incubation procedure introduced by Waring and Bremner (1964) because Sims et al. (1967) found soluble plus extractable NH_4–N produced from anaerobic incubations of ≤ 12 d had better predictive value than longer incubation periods. Sims et al. (1967) also reported soluble plus extractable NH_4–N produced over shorter periods was a better index of soil N availability to rice.

Chemical Methods

Inorganic-N concentration has been ascertained by salt extraction (e.g., 2 mol L^{-1} KCl) of soil removed from fields suitable to agricultural production. Two soil-based N tests developed for upland crops are the pre-plant nitrate test (PPNT) and the pre-sidedress nitrate test (PSNT). The PSNT utilizes a sampling protocol which occurs during the vegetative growth stage of corn. Soil NO_3–N concentration during the vegetative growth phase of corn (i.e., V4–V6) represents a net balance between production (mineralization from soil organic matter, manure and/or fertilizers) and loss (leaching, denitrification and immobilization) because the plant has not begun rapid uptake of N (Meisinger et al., 1992b).

Magdoff et al. (1984) were the first to report a strong linear relationship ($r^2 = 0.74$) between silage yield and soil NO_3–N concentration for corn grown in Vermont by analyzing composite soil samples taken to a depth of 30 cm. This method was later termed the PSNT due to the fact that soil NO_3–N concentration is determined 1 to 2 wk before normal sidedressing of fertilizer N (Meisinger et al., 1992a). This chemical extraction is suitable for use in different climates and cultural systems encountered in the United States. For example, Fox et al. (1989) in Pennsylvania and Blackmer et al. (1989) in Iowa were able to establish a soil NO_3–N critical value (25 mg N kg soil^{-1}) above which corn was non-responsive to sidedressed fertilizer N.

The PSNT is conducted by subjecting air-dried soil to 2 mol L^{-1} KCl, shaking the solution, decanting and analyzing the extract for inorganic N using steam distillation (Meisinger et al., 1992a). The advantages of the PSNT is that it can be conducted using minimal laboratory space, non-hazardous chemical reagents are used for soil extraction and the short analysis time (~1.5 h) enables soil testing laboratories to analyze a large number of samples. However, due to the fact that PSNT is most reliable when samples are taken during the growing season, the inconvenience of refrigerated transportation to laboratory, required to limit microbial activity, as well as preparing the soil for laboratory analysis is a disadvantage for routine soil testing laboratories following the PSNT protocol.

Labile Soil Organic Nitrogen Analysis

A chemical method with the potential to overcome the logistical problems associated with the PSNT could be the chemical analysis of labile soil organic-N. The analysis of labile soil organic-N represents a quantifiable fraction of total N that is mineralized and becomes available for plant uptake during the growing season. In addition to inorganic-N found in soil, there are also soluble or easily mineralizable organic-N compounds that represent a potential source of N which the plant can assimilate (Davidescu, 1982). Chemical methods of soil analysis have focused on isolating the fraction of soil organic-N that is strongly correlated to either plant response in the field or the concentration of N mineralized during soil incubations conducted in the laboratory. For example, using a 14 d anaerobic incubation of Alfisol and Mollisol silt loam soils from rice producing regions of Arkansas, Wilson et al. (1994) found the amount of NH_4–N produced was adequately predicted with a more rapid (~1 h) chemical analysis using acidified potassium permanganate ($KMnO_4$) and acidified potassium dichromate ($K_2Cr_2O_7$). This study revealed that acidified $KMnO_4$ and acidified $K_2Cr_2O_7$ exhibited the strongest coefficients of determination (r^2) of 0.83 and 0.86, respectively, suggesting these chemical methods could have use for predicting N mineralization. Similarly, Bushong et al. (2007) compared the sensitivity of three chemical methods capable of quantifying labile soil organic-N to the amount of NH_4–N produced during a 14 d anaerobic incubation of 50 surface soils from across the South, central and Midwestern United States. The authors found that all of the methods evaluated (i.e., acid-oxidation, chemical extraction utilizing 1 mol L^{-1} KCl, and alkali diffusion) were predictive of NH_4–N mineralized during the incubation period with coefficients of determination ranging from 0.31 to 0.58.

By combining the techniques of acid hydrolysis and alkaline distillation researchers have been able to fractionate total soil organic-N in many agronomic studies (Stevenson, 1996). This method is very time consuming requiring a lengthy acid hydrolysis procedure and is therefore unsuitable for routine soil testing. However, this is a useful analytical method for identifying the various components of soil organic-N. The fractions of soil organic-N isolated using acid hydrolysis-diffusion include: total hydrolyzable-N, hydrolyzable NH_4–N, amino sugar + hydrolyzable NH_4–N, and alpha-amino acid N (Mulvaney and Khan, 2001). This procedure requires acid hydrolysis of 5 g soil using 3 or 6 mol L^{-1} HCl and heating to > 100ºC for 12 to 24 h. Once the acidic hydrolysate is neutralized, the aforementioned soil organic-N fractions can be quantified based on the amount of NH_3 volatilized using either steam distillation (Stevenson, 1996) or diffusion (Mulvaney and Khan, 2001).

Development of the Illinois Soil Nitrogen Test

Organic-N is a major pool of N utilized by crops; therefore, a logical soil-based N test would examine the portion of this pool mineralized during the growing season (Kowalenko and Babuin, 2009). The length of time needed to complete acid hydrolysis-distillation (12–24 h) and the recovery errors of isolated hydrolyzable-N fractions (Mulvaney and Khan, 2001) led to the development of an alkaline diffusion method performed directly on soil. This procedure was given the name Illinois Soil Nitrogen Test (ISNT) and has been successfully used in Illinois to separate responsive from non-responsive fields when corn was grown using sidedressed fertilizer N applications. The advantage of ISNT is the ability to accurately and precisely quantify amino sugar-N (AS-N) in soil. However, subsequent research indicated the ISNT was not sensitive to mineralizable-N but measured only a constant fraction of soil total N (Laboski et al., 2008). Since the development ISNT there have been both negative (Barker et al., 2006b; Laboski et al., 2008; Osterhaus et al., 2008; Spargo et al., 2009) and positive (Mulvaney et al., 2006;

Klapwyk and Ketterings et al., 2006; Bushong et al., 2007; Williams, 2007; Lawrence et al., 2009) evaluations of this alkaline diffusion soil test method.

Using data from 96 N rate trials from Illinois, Iowa, Michigan, Minnesota, Nebraska, and Wisconsin, Laboski et al. (2008) recorded a 48.3% failure rate (Type A), meaning that sites predicted as non-responsive according to the ISNT critical value (230 mg N kg soil^{-1}) were found to need additional fertilizer N. Undoubtedly, the inability of a soil N test to distinguish between responsive and non-responsive sites to the addition of fertilizer N would prohibit the method from gaining acceptance as a reliable N soil test method. The influence of soil depth and sampling time on AS-N concentration was investigated in Iowa (Barker et al., 2006a). From 2001 to 2003 Barker et al. (2006a,b) compared the difference in AS-N concentration using three sampling times (fall, early spring and late spring) and two sampling depths (0–15 cm and 0–30 cm) from 43 N rate trials in commercial production corn fields. The results presented by Barker et al. (2006a,b) revealed that with the exception of sites high in organic C or recent manure application, the AS-N concentration was similar for samples taken in the fall and spring. Regarding sampling depth, the authors recorded a significantly ($p < 0.01$) higher concentration of AS-N in the 0- to 15-cm soil depth with a mean (n = 505) concentration of 302 mg N kg soil^{-1} compared to 277 mg N kg soil^{-1} for the 0- to 30-cm depth.

The fact that AS-N concentrations are similar for samples taken in the fall and spring (Barker et al. 2006a) suggests producers could incorporate this soil test using a sampling schedule that coincides with chosen cultural practices. However, in an ensuing study, Barker et al. (2006b) came to the conclusion that AS-N concentration as determined using ISNT (Khan et al., 2001) could not be recommended in Iowa for adjusting fertilizer N rates or predicting corn N responsiveness. The lack of correlation between ISNT and N response measurements (i.e., leaf chlorophyll meter value, relative grain protein, relative yield or grain yield response to additional N) along with the inability of ISNT to be calibrated based on an economically optimal N rate (EONR) for corn was the basis for the negative evaluation (Barker et al., 2006b).

Spargo et al. (2009) examined 29 corn field trials from across Virginia and a weak relationship ($r^2 = 0.33$, $p = 0.0126$) was recorded when relative yield (RY) was regressed against ISNT values from those trials categorized as responsive (< 200 mg N kg soil^{-1}) to additional fertilizer N according to the ISNT soil test value. The authors came to the conclusion that this weak relationship may potentially limit the ability of ISNT to improve fertilizer N management for corn. Similarly, Osterhaus et al. (2008) in Wisconsin came to the conclusion that the ISNT was not a reliable predictor of the N requirements of corn. The authors of this study found a strong relationship between ISNT and soil organic matter ($r^2 = 0.88$) and suggested the test was measuring a constant fraction of soil organic-N rather than a mineralizable fraction of soil organic-N.

Despite the aforementioned negative evaluations, ISNT has been used to identify corn fields where sidedress fertilizer N application would produce a positive response in Illinois (Khan et al. 2001). Other researchers (Klapwyck and Ketterings, 2006; Lawrence et al., 2009) have suggested that when combined with additional measurements (i.e., soil organic matter) ISNT can be used to distinguish between fields of high and low native soil N fertility. Also, additional research has shown that ISNT has the ability to predict EONR for corn grown in the southeastern United States after separating sites based on drainage class (Williams et al., 2007).

By examining a total of 102 site-years gathered from two distinct sampling periods, the first from 1990 to 1992 and the second during 2001 to 2003, Mulvaney et al. (2006) were able to identify sites of N misapplication when a yield-based approach was used for corn production in Illinois. This study proposed the use of a soil-based approach to fertilizer N management and the authors revealed that of the 33 site-years characterized as non-responsive according to an ISNT critical value of 230 mg N kg soil^{-1}, 31 were correctly identified as non-responsive, while 50 of 69 site-years were predicted correctly as responsive to additional fertilizer N.

Klapwyck and Ketterings (2006) conducted 24 corn field trials from 2002 to 2004 in New York using samples (0–20 cm) with a mean soil organic matter concentration of 54 g kg^{-1}. This study found that N soil test critical values could be obtained when mean soil organic matter content was considered in addition to N availability as determined by ISNT. The authors came to the conclusion that the ISNT soil test value could be used to adjust fertilizer N recommendations for corn beyond a starter application when soil organic matter was also taken into consideration. Lawrence et al. (2009) conducted a similar study in New York using soil organic matter adjusted ISNT critical values. The authors found that soil organic matter adjusted ISNT critical values were 83% accurate in predicting N responsiveness of second year corn following sod or soybean (*Glycine max* L.). However, the authors did caution that soil samples not be taken for 5 wk following sod incorporation due to the dynamics of inorganic-N release following sod decomposition. The authors also suggested that sod N credits be added to accurately assess N responsiveness for corn that follows sod in rotation.

Beyond including other measures of soil fertility (e.g., soil organic matter) researchers have found that separating sites based on geographic location or soil properties (i.e., soil texture or drainage class) can improve the predictive ability of the ISNT (Bushong et al., 2007) and improve the relationship between EONR and ISNT (Williams et al., 2007). For example, Bushong et al. (2007) found the concentration of NH_4–N recovered following a 14 d anaerobic incubation of 50 clayey and loamy textured soils from different regions of the United States was adequately predicted ($r^2 = 0.45$) using ISNT but improved considerably ($r^2 = 0.71$) when only soils from Arkansas were evaluated. Also, Williams (2005) found that when soils were separated according to the four major crop growing regions of North Carolina, ISNT soil test values from the undulating landscape of the Piedmont region were negatively correlated to slope gradient. The author concluded that this relationship suggested that AS-N, as determined using a modified ISNT protocol of Khan et al. (2001), was sensitive to landscape position.

Illinois Soil N Test soil test values were also found to be weakly correlated to unfertilized wheat (*Triticum aestivum* L.) yield ($r = 0.47$) and fertilizer N response ($r = -0.67$) in Canada (Steckler et al., 2008). Steckler et al. (2008) found that the strength of the correlation varied according to landscape position (upper, middle, or lower slope position) and location, similar to the results of Williams (2005). Williams et al. (2007) using 34 N response trials from North Carolina recorded a strong relationship between ISNT and EONR when soils were separated into well drained ($r^2 = 0.87$) and poorly drained ($r^2 = 0.78$) categories. Williams et al. (2007) concluded that when soils were separated into their respective drainage classes, ISNT could be used to effectively predict EONR and effectively manage fertilizer N applications for corn grown in North Carolina.

The ISNT method, while offering several advantages, is still a rather time consuming (5 h) laboratory procedure. Also, the protocol outlined by Khan et al. (2001) utilizes electric griddles that can encumber the daily activities of a soil testing laboratory. Sample-to-sample variability is an additional concern associated with ISNT analysis and has been addressed by rotating sample jars 1.5 and 3 h from the beginning of the 5 h diffusion period and modifying the griddle's main temperature control (UIUC, 2003). Temperature differences between griddles along with differences in air-flow and daily room temperature in the laboratory could potentially influence reproducibility of ISNT sample analysis. Rapid analysis time (~ 8 min) and the widespread use of steam distillation by soil testing laboratories across the country led to the comparison of direct steam distillation (DSD) and ISNT (Ross, 2007; Bushong et al., 2008; Roberts et al., 2009).

Direct Steam Distillation

Hydrolysates obtained from N fractionation schemes have been analyzed for organic-N, with amino acids and amino sugars being the main identifiable forms of organic-N (Stevenson, 1982). Direct steam distillation represents an advantage over conventional hydrolysis-distillation or steam distillation performed on soil extracts due to the fact that DSD does not require the soil be pretreated or extracted prior to analysis. The DSD method conducted by Bushong et al. (2008) involves combining 1 g oven-dried soil and 10 mol L^{-1} NaOH in a distillation flask, steam distilling into 4% (wt./v.) H_3BO_3-indicator solution at an approximate rate of 5 mL distillate min^{-1} until a volume of 40 mL is reached and the N content of the distillate determined by acidimetric titration.

The successful development of the ISNT procedure as outlined by Khan et al. (2001) and its potential for incorporation as a routine soil N test procedure led Bushong et al. (2008) to evaluate DSD using differing strengths of an alkaline reagent (i.e. 2, 5, or 10 mol L^{-1} NaOH). Regression analysis revealed the strength of the linear relationship ($r^2 = 0.92$) between ISNT and 10 M NaOH was the strongest, with a slope (1.08) and intercept (1.10) that would suggest the two methods are quantifying a similar fraction of soil organic-N (Bushong et al., 2008). The authors conclude by suggesting separation based on soil texture and geographic origin may further improve the ability of ISNT and DSD to predict mineralized soil N during anaerobic incubation. Ross (2007) evaluated several chemical methods that attempted to quantify the portion of organic-N mineralized during the growing season including ISNT and 10 mol L^{-1} NaOH DSD. The author found a significant ($p < 0.0001$) relationship between ISNT and DSD across the four soil depths (0–15, 15–30, 30–45, and 45–60 cm) evaluated in the study. The finding of Ross (2007) provided evidence in support of using either ISNT or DSD to predict the fertilizer N rate required to achieve optimal yields for rice grown in Arkansas.

To evaluate whether ISNT and 10 mol L^{-1} NaOH DSD recover a similar suite of organic- N compounds, Roberts et al. (2009) quantified the amount of N recovered from soil following the application of purified organic-N compounds. The authors found that all four of the pure AS-N compounds evaluated had greater recoveries using ISNT than DSD although the authors did observe significantly ($p < 0.0001$) higher recovery of transition amino acids using DSD. This finding led the authors to the conclusion that even though the amount of N liberated from amino sugars and transition amino acids differs between the two methods, the alkaline hydrolyzable-N (AH-N) concentration measured is very similar using either method.

Rice Grain Yield Response to Seeding Date

Seeding date, variety selection, and seeding rate are among the most important decisions a producer will make prior to the growing season and when the appropriate management practices are selected can result in a more efficient harvest (Blanche et al., 2009). Choosing an appropriate seeding rate for any seeding date is an important decision due to the effect of temperature on stand establishment and the relationship between uniform stands and yield (Blanche et al., 2009). Seeding date selection is important because achieving a uniform stand can be difficult when rice is seeded early due to cooler air and soil temperature during germination and seedling development.

Seeding outside the optimal time period in central Arkansas, either prior to 10 April or after 15 May, can influence rice plant physiology as well as determine the timing of other cultural practices during the growing season. For example, a seeding date study was conducted by Gravois and Helms (1998) in Stuttgart, AR on a Crowley silt loam (fine, montmorillonitic, thermic, Typic Albaqualfs). The authors observed the time from seeding to flooding a field decreased as seeding date was delayed and was extended for early-seeded rice which resulted in more difficult and expensive weed control. This suggests that when seeding dates are selected that fall outside the optimal time period, more intensive management practices may increase production costs and lower rice productivity and profitability.

Influence of Seeding Date on Rice Grain Yield

The concept of growing degree-day has been used successfully to model the physiological development of rice (Keisling et al., 1984). The degree-day 10 (DD10) program, developed in the 1970s as a modification of the growing degree day concept, was introduced to predict when certain rice plant growth stages (e.g., internode elongation) would be reached during the growing season (Slaton and Norman, 2001). As seeding is delayed, the time of year during which vegetative (i.e., germination to panicle initiation) and reproductive (i.e., panicle initiation to heading) growth occur will necessarily shift in comparison to rice seeded within the optimum time period. These growth stages will therefore take place later in the season, although a potential overlap can occur depending on the extent of the delay. This shifted growth pattern can have a direct impact on plant physiology and the elevated day and nighttime air temperatures commonly encountered from June to mid-August in eastern Arkansas could negatively affect plant growth and yield.

In an attempt to further evaluate the effect of temperature on plant physiology and grain yield of rice, Peng et al. (2004) examined temperature trends over a period from 1979 to 2003 near Manila (14°11' N latitude, 121°15' E longitude) in the Philippines. The authors of this study found that for every 1°C increase in mean minimum nighttime temperature above 22°C rice grain yield was reduced by 10%. Even though Peng et al. (2004) acknowledged the role of increased plant maintenance respiration as a contributing factor to reduced yield as temperature increased, they also suggested the influence of day and nighttime temperature on tillering, leaf area expansion, stem elongation, crop development, and grain filling could also be contributing to reduced grain yield.

In an attempt to quantify yield loss associated with delayed planting, Slaton et al. (2003) evaluated yield trends from Stuttgart, AR and Crowley, LA for modern rice cultivars and cultivars commonly grown in the 1960s and 1970s. The authors found a predicted yield loss $\leq$ 10% when modern rice cultivars were seeded by 22 April or 26 May at the Louisiana and Arkansas locations, respectively. Whereas, yield losses of 35% were predicted for modern cultivars when seeded by 31 May or 4 July for the Louisiana and Arkansas locations, respectively, indicating yield declined substantially as seeding date was delayed during the growing season. The authors suggested rice grain yield decreased in response to a shorter vegetative growth phase when seeding was delayed during the growing season.

Water-seeded rice production allows for planting operations to proceed according to the producer’s schedule regardless of soil conditions, while offering control over red rice infestation (Smith, 1981). Because early seedling establishment and vigor may differ in comparison to drill-seeded rice, Sha and Linscombe (2007) evaluated the influence of seeding date on conventional, hybrid, and breeding rice lines in Crowley, LA under water-seeded conditions. The authors found the time required to achieve 50% heading was reduced by 18 d when rice was seeded in early June compared to early May. Although, the authors point out that the number of accumulated degree-days (>10ºC) from emergence to 50% heading was not different across seeding dates. This finding is in agreement with the earlier work of Norman et al. (1999, 2000, and 2001) which showed the accumulated degree-days required to reach different stages of plant growth (e.g., 50% heading) remains relatively constant for a given rice cultivar regardless of seeding date or year.

Blanche and Linscombe (2009) conducted a seeding date study in Crowley, LA using eight cultivars and seven seeding dates ranging from 27 February to 4 July to evaluate the effects of seeding date and cultivar on yield. The authors found Cypress and Clearfield 161 (CL161) performed consistently over a three-year trial while providing the highest whole kernel milling yields. Also, these cultivars had the two lowest coefficient of variation (CV) values over this same time period, suggesting that Cypress and CL161 exhibited greater yield stability even as environmental conditions changed from year-to-year.

Fertilizer Nitrogen Management of Late-Seeded Rice

Current N fertilization practices for rice production are based on variety, soil texture, previous crop, and stand density. Unfortunately, this approach can result in under- or over-fertilization of the crop, leading to inefficient fertilizer N management and environmental degradation. Previous research evaluating nitrogen fertilizer recovery of the drill-seeded, delayed flood rice cultural system has shown that N uptake can range from 53 to 63% of the total preflood N applied (Wilson et al., 1989). This suggests that N management early in the growing season is a critical component of drill-seeded, delayed flood rice culture.

Since environmental conditions are often quite variable during the growing season, an understanding of the plant growth stages during which yield components (i.e., number of panicles per unit land area, number of spikelets per panicle, percentage of filled grains, and weight of filled grains; Moldenhauer and Gibbons, 2003) are determined may be of importance when evaluating the relative influence of seeding date among rice cultivars. Tillers that develop during vegetative growth influence the total number of panicles a plant can produce, while reproductive growth determines the number of spikelets and number of seeds contained by each spikelet (DeDatta, 1981). Longer-season rice cultivars typically produce a greater number of tillers than early-season cultivars (Bruns, 2009) and therefore environmental conditions during the vegetative growth phase could potentially influence productivity.

Reddy and Patrick (1976), Wilson et al. (1989), and Guindo et al. (1994) found during vegetative growth the plant relied on N derived from fertilizer and during reproductive growth the rice plant derived a majority of N from the native soil N pool. These results highlight the general difference between the rice plant's utilization of applied fertilizer N and native soil N during the growing season. These results may also provide some insight into how a shorter vegetative growth period of late-seeded rice could alter fertilizer N response.

Nonexchangeable Ammonium

The N cycle can be divided into three general components: inputs, outputs, and internal (i.e., soil) N-cycling where N is neither gained nor lost (Havlin et al., 2005). Nonexchangeable ammonium (NEA) represents a fraction of NH_4–N separate from the solution and exchangeable inorganic-N pool and NH_4–N fixation is considered mainly a loss mechanism in the N cycle. The process of NH_4–N fixation is not entirely irreversible and the release of NEA into solution indicates NEA may influence other components of the N cycle.

Soil inorganic NH_4–N is known to exist in equilibrium among solution, exchangeable, and clay-fixed forms (Kowalenko and Cameron, 1976; Nommik, 1981). An increase in the solution concentration of NH_4–N can produce an increase in exchangeable NH_4–N as well as NEA which is categorized by "weakly" and "tightly" held fractions (Gouveia and Eudoxie, 2007). The fixation of NH_4–N following the addition of fertilizer N is a loss mechanism that has the potential to influence fertilizer N use efficiency. Recent fixation of added NH_4–N occurs relatively quickly and takes place in the sand, silt, and clay size fractions of soil although the release of NEA is much slower (Kowalenko and Ross, 1980). The slow rate at which NEA can replenish available NH_4–N in solution as well as the unpredictable fate of NEA upon release from the interlayer of clay minerals makes identifying the role of NEA in plant nutrition difficult to determine, however NH_4–N can readily transform to NEA upon fertilizer N addition.

Influence of Clay Mineralogy on Nonexchangeable Ammonium

Since soils are known to contain the clay minerals principally responsible for NH_4–N fixation (i.e., illite, vermiculite, and montmorillonite) it is apparent the retention of NH_4–N as NEA would occur in soil following N added either naturally or in fertilizer (Bremner, 1959). Kaolinite does not retain any appreciable quantity of NEA (Allison et al., 1953b) and would therefore be unlikely to influence the NH_4–N fixation capacity of soil. Also, fixation occurs following the replacement of interlayer cations by NH_4–N while the clay mineral structure is in an expanded state (Dhariwal and Stevenson, 1958), therefore the inability of 1:1 structured kaolinite minerals to expand while remaining intact would preclude cation replacement.

Mica and montmorillonite type clay minerals were evaluated by Barshad (1954a) to identify some of the factors regulating K and NH_4–N fixation which included particle size, interlayer charge, presence of exchangeable H^+, replacing cation, and presence of interlayer K. The results presented by the author indicated that for both unground (2–10 mm) and ground (< 0.1 mm) vermiculite the fixation capacity was greatly influenced by interlayer charge. For unground vermiculite having an interlayer charge of between 210 to 220 $cmol_c$ kg^{-1}, nearly complete fixation of added NH_4–N (> 90%) occurred subsequent to N added as neutral ammonium acetate (NH_4Ac). An interlayer charge of 106 $cmol_c$ kg^{-1} was unable to fix any added NH_4–N for either the ground or unground vermiculite. Ground vermiculite exhibited a similar relationship between interlayer charge and fixation capacity. However, the fixation capacity was lower, achieving a maximum of 86% of total added NH_4–N compared to 96% for unground vermiculite when both had an interlayer charge of 220 $cmol_c$ kg^{-1}.

Barshad (1954b) also examined particle size influence on cation exchange properties of both pure and mixed vermiculite clay minerals. Cation exchange experiments were conducted on both unground (2–10 mm) and ground (< 0.1 mm) vermiculite clay minerals by leaching the adsorbent with a salt solution containing the cation of interest and subsequently determining the concentration of the replaced cation in the filtrate. The results of Barshad (1954b) indicated that the inability of interlayer cations to migrate out of unground vermiculite due to contraction of the clay mineral edges would result in these interlayer cations becoming fixed and therefore resistant to replacement by cations in solution. Thus, the inability of the replacing cation to gain edgewise entry into the lattice would suggest that accessibility along edges and discontinuities in the mineral structure are important factors regulating cation adsorption and retention.

When isomorphous substitution of aluminum (Al^{3+}) or silicon (Si^{4+}) occurs during crystallization, the clay mineral possesses a net negative charge which must be balanced by cations either inside or outside the mineral structure (Nommik and Vahtras, 1982). The results of Barshad (1954a) indicated that layer charge was one factor that directly influenced fixation of NH_4–N and because the magnitude of the interlayer charge greatly influences the strength of the interlayer bond (Nommik and Vahtras, 1982), the ability of the 2:1 phyllosilicates to retain NEA may be related to the attractive forces which exist in the interlayer positions of these minerals. For example, montmorillonites with a weak interlayer bond expand readily to allow water and cations to gain access to the interior of the clay mineral. Whereas for vermiculites with an intermediate layer charge, certain cations (i.e., Ca^{2+}, Mg^{2+} and Na^{+}) are considered exchangeable when located in the mineral interior, while other cations (i.e., NH_4–N and K^{+}) are considered nonexchangeable (Nommik and Vahtras, 1982).

Ammonium Fixation

While illite and vermiculite can fix added NH_4–N under dry or moist conditions, montmorillonite can fix added NH_4–N only upon drying (Nommik and Vahtras, 1982). The wetting–drying cycle experienced by agricultural soils that are predominately composed of montmorillonite clay minerals may exhibit a different pattern of NH_4–N fixation and release than illite or vermiculite dominated soils. Schneiders and Scherer (1998) carried out a series of soil incubations to identify the dynamics of NEA under flooded conditions. An Entisol containing smectite, illite, and vermiculite and an Ultisol containing only illite and chlorite were subjected to a continuous flood for 8 wk.

Schneiders and Scherer (1998) found during an initial 8 wk incubation 26 and 84% of the mineralized organic-N was transformed to NEA for the Ultisol and Entisol soils, respectively under a continuous flood. The author stated the substantial difference in the ability of soil to retain NH_4–N in a nonexchangeable form could be explained in part by the fact that the Ultisol lacked the expandable clay minerals necessary for fixation. During the second 9 wk incubation the soil was allowed to air dry, $(NH_4)_2SO_4$ was applied (60 mg N kg soil^{-1}), and the soil was flooded for a second time. The results of the second incubation indicated the Ultisol and Entisol fixed 14 and 39% of the total added NH_4–N, respectively. The results of Schneiders and Scherer (1998) indicated that under flooded conditions NH_4–N fixation still occurred in both the Ultisol and Entisol, although a much smaller portion of the total quantity of mineralized N tended to be fixed in soils without expanding 2:1 phyllosilicate clay minerals. The results presented by the authors suggested mineralization can lead to an accumulation of recently fixed NH_4–N derived from soil organic-N during incubation.

Dynamics of Nonexchangeable Ammonium

While soil incubation studies conducted in the laboratory can be useful in identifying N transformations among soil inorganic-N fractions under optimal conditions, the removal, homogenization and leaching of incubated soil may inhibit the full expression of the microbial diversity of field soil. Also, the typically favorable moisture and temperature conditions that are readily imposed and maintained in the laboratory are not feasibly recreated in the field. Furthermore, the variation of temperature and moisture as well as the influence of the rhizosphere could alter the rate of NEA fixation and release in the field. Installation of field plots can result in the alteration of soil structure in comparison to intact soil, however, the exposure of soil to the solute and gas fluxes which occur in the field suggest field studies could provide a more representative account of N transformations.

Kowalenko (1978) established a 17 mo field incubation study on a Bainsville (Orthic Humic Gleysol) clay loam soil in Canada to identify the dominate processes that regulated the fate of ^{15}N-labeled ammonium sulfate [$(^{15}NH_4)_2SO_4$] applied to surface (0–15 cm) soil. The results of this study revealed NH_4–N fixation occurred very rapidly followed by a slower rate of release which was in agreement with earlier studies (Kowalenko and Cameron, 1976; Sowden, 1976) of laboratory incubated soil. The mean daily release of NEA was 1.70 kg N ha^{-1} over a 42 d period. The results also revealed 66% of the NEA derived from fertilizer N is released 85 d after incorporation while the remaining portion of NEA was retained possibly forming a stronger fixation to clay minerals over the remaining 426 d.

Kowalenko (1978) suggested the continued decrease of extractable NH_4–N without a concurrent release of NEA indicated a portion of recently fixed NEA had achieved greater stability within a depth of 0- to 15-cm. The author points out the wetting–drying and freezing–thawing cycles which occurred during field incubation could have led to the increased stability of NEA. The change in NEA as influenced by wetting, freezing, and drying of soil has been reported by Walsh and Murdock (1960) and the change in concentration of strongly fixed NEA in response to soil moisture content (Gouveia and Eudoxie, 2007) indicated that a conversion of recently fixed NEA to a form that exhibits greater stability is one potential fate of NH_4–N derived from fertilizer sources.

Liu et al. (1997) conducted a 30 yr fertility experiment established on a Plano (fine-silty, mixed, mesic, Typic Argiudolls) silt-loam soil near Arlington, WI to identify the response of NEA to nutrient management practices. The results of Liu et al. (1997) revealed a nonsignificant difference ($p > 0.05$) between the concentration of NEA of the 0 and 168 kg N ha^{-1} fertilizer N rate treatments, which suggested the capacity of the soil to fix added NH_4–N remained unchanged. The saturation of available fixation sites for NH_4–N might have been expected to occur over a 30 yr period of continued NH_4–N fertilizer input and thus influence the ability of the soil to fix added NH_4–N. However, the authors came to the conclusion that because NH_4–N in solution was unable to accumulate, the release of recently fixed NEA inhibited the accumulation of NEA and therefore the NH_4–N fixing capacity was not affected by fertilizer N application.

Subsoil Nonexchangeable Ammonium

The presence of native NEA in the subsoil has been documented to a 122 cm depth (Hinman, 1964). Also, the ability of subsoil to fix a greater amount of NH_4–N than surface soil (Allison et al., 1953a) indicates NEA may influence N cycling within both surface soil and subsoil. Lower NH_4–N fixation of surface soil compared to subsoil is thought to be due to NH_4–N and K^+ occupying available fixation sites, the typically greater clay content of subsoils, as well as greater organic matter content.

Soon (1998) examined the difference in the transformation of NEA in surface soil and subsoil by comparing 0–15, 15–30, 30–55, and 55–80 cm depths under the influence of either continuous barley (*Hordeum vulgare* L.), continuous bromegrass (*Bromus inermis* Leyess.), or barley since 1991 in Canada. During the growing season there were significant ($p < 0.05$) differences in NEA concentration among sampling depths suggesting NEA concentration was not the same across the entire 0- to 80-cm sampling depth. The difference in NEA concentration at the beginning and end of the growing season indicated NEA in the 55- to 80-cm depth decreased by 12 mg N kg^{-1} and 17 mg N kg^{-1} in 1994 and 1995, respectively. Soon (1998) documented less of a change in NEA concentration in the surface soil compared to the subsoil and net release of NEA occurred late in the growing season (i.e., grain fill). The author indicated the fluctuation of NEA in subsoil could have been a result of the migration of soluble organic matter to the subsoil where mineralization of organic-N increased NH_4–N concentration leading to the temporary increase of NEA.

Hinman (1964) sampled soil profiles (0–122 cm) in Canada to identify the relationship between soil texture and NEA. Soil textural classes ranged from fine sandy loam to clay. The results of the study revealed NEA content tended to increase with depth and the profile contained 34 to 76% of NEA in the clay fraction, while 21 to 47% of NEA was in the silt fraction. Hinman (1964) noted the ratio of native NEA to total N increased with sampling depth. The increase in NEA with depth indicated that NEA could be an important factor of the N cycle in subsoil.

Quantification of Nonexchangeable Ammonium

A majority of the methods used to quantify NEA have selected an acid digestion process to dissolve the clay mineral structure and release interlayer NH_4–N. While several methods have gained general acceptance, the inability of these methods to specifically isolate NEA has generated some concern regarding the potential overestimation of NEA as a result of organic-N released during acid hydrolysis. While procedural modifications have been made to avoid the handling of hazardous materials, the potential for labile organic-N to be released remains an analytical problem which can prohibit the accurate determination of NEA.

A review of some of the existing methods which attempted to quantify NEA was undertaken by Bremner (1959) to identify the advantages and disadvantages associated with each method. The methods of Rodrigues (1954) and Allison and Roller (1955) were evaluated by Bremner (1959) along with the effect of various soil pretreatments on the release of NEA. The results indicated that following the procedure of Rodrigues (1954) up to 52% of total N found in the soil extract was in the form of acid hydrolyzable NH_4–N while the greatest recovery of NEA was approximately 28% of total soil N. The reason acid hydrolyzable-N was believed to be much greater than NEA was because the use of a strong acid mixture of 4:1 (v/v) 40% HF–50% H_2SO_4 resulted in the formation of acid soluble-N and NH_4–N derived from organic compounds.

The duplicate distillation method of Allison and Roller (1955) quantified < 3.3% of total soil N in the form of NEA. The author noted that less NEA was released using duplicate distillation compared to the amount of NEA released using 40% HF–50% H_2SO_4. Bremner (1959) acknowledged the possibility that K^+ released into solution during NaOH distillation could have exerted a blocking effect on the subsequent release of NEA which resulted in the lower quantification of native NEA.

To limit the interference of organic-N, a potassium hydroxide (KOH) pretreatment was evaluated since a substantial portion of organic matter and labile-N compounds (e.g., glutamine, asparagine, and glucosamine) dissolve in heated KOH solution (Bremner, 1959). The quantification of NEA was very similar before and after pretreatment with differences not exceeding 2% of total soil N. This result suggested the inclusion of a KOH pretreatment prior to the quantification of NEA using acid digestion would not be expected to substantially influence the release of NEA and may reduce the interference of organic-N compounds.

Following the identification of a potential soil pretreatment to be used in conjunction with acid hydrolysis for the determination of NEA, Silva and Bremner (1966) continued to examine different soil pretreatments to identify a method that could be applied to soil regardless of organic matter content. Specifically, the pretreatment examined by Silva and Bremner (1966) consisted of using an alkaline potassium hypobromite (KOBr–KOH) solution prior to 5 mol L^{-1} HF–1 mol L^{-1} HCl acid extraction for 24 hr. The total N concentration of the 10 soils examined by the authors was in the range of 813 mg N kg^{-1} to 9120 mg N kg^{-1} and the organic carbon (C) content of the soils ranged from 0.79 to 10.2%. After evaluating a range of pretreatment reaction times (2–12 hr) followed by boiling for a maximum of 30 min Silva and Bremner (1966) found that reaction of soils with KOBr–KOH for 2 hr followed by boiling the soil-solution mixture for 5 min was necessary to completely remove alkali-labile compounds in the soil. The KOBr–KOH pretreatment was able to remove >92 and >96% of the total organic-C and organic-N in the soil, respectively. Using the KOBr–KOH pretreated soil the quantification of NEA was highly reproducible with an approximate standard deviation of 1 mg N kg^{-1}. The near complete removal of both organic-C and organic-N led to the conclusion that KOBr–KOH pretreatment could be used to obtain a more reliable estimate of NEA. However, Silva and Bremner (1966) acknowledged that providing definitive results regarding the ability of KOBr–KOH pretreatment to be completely free from the interference of additional N compounds was beyond the scope of the study.

The Silva and Bremner (1966) procedure has been applied to a wide variety of soils for the quantification of NEA (Paramasivam and Breitenbeck, 2000; Eudoxie and Gouveia, 2001). However, the method of Silva and Bremner (1966) is time consuming, requiring approximately 26 h to complete analysis, and modifications of the original procedure have been proposed to reduce analysis time and limit use of concentrated chemical reagents. Because the method of Silva and Bremner (1966) is time consuming, requiring approximately 26 h to complete analysis, modifications to the original procedure have been proposed to reduce analysis time and limit use of concentrated chemical reagents. One modification proposed by Liang et al. (1999) substituted dry combustion for the 5 mol L^{-1} HF–1 mol L^{-1} HCl acid digestion of clay minerals to quantify NEA.

Liang et al. (1999) compared the 5 mol L^{-1} HF–1 mol L^{-1} HCl procedure of Silva and Bremner (1966) to a proposed procedure utilizing combustion of soil following KOBr–KOH pretreatment. The proposed procedure was a modification of the HF–HCl procedure, whereby NEA is released using dry combustion at 1030ºC subsequent to the pretreatment of soil as outlined by Silva and Bremner (1966). Liang et al. (1999) reported the proposed dry combustion method was highly correlated ($r^2 = 0.97$) to the HF–HCl method with a slope of 1.03. The nearly 1:1 linear relationship between the two methods led the authors to suggest that the proposed method could serve as a suitable alternative to the original method of Silva and Bremner (1966).

Nonexchangeable Ammonium Released During Alkaline Hydrolysis

Acid hydrolysis is capable of releasing a greater amount of total NEA present in soil compared to alkaline hydrolysis (Greenfield, 2001). Alkaline distillation is effective at removing NEA because the NH_4–N released from interlayer positions in the clay mineral is continuously removed, thereby preventing the accumulation of exchangeable NH_4–N (Nommik and Vahtras, 1982). However, the presence of a low concentration of K^+ in solution has been shown to effectively block the release of fixed NH_4–N during alkaline hydrolysis (Leggett and Moodie, 1963). Therefore, even though fixed cations (i.e., K^+ or NH_4^+) can be replaced by cations in solution that expand the clay mineral lattice (e.g., Na^+; Barshad 1954a) the concurrent presence of K^+ has the ability to limit the release of NEA during distillation.

Hanway et al. (1957) subjected pure vermiculite and bentonite clay minerals to an alkaline distillation using 0.1, 1, or 2 mol L^{-1} NaOH solution containing varying amounts of K^+. Ammonium-saturated vermiculite containing 82 $cmol_c$ kg^{-1} was distilled in the absence of K^+ and the amount of NEA released increased with the concentration of sodium hydroxide (NaOH). Complete recovery (i.e., 82.3

$cmol_c\ kg^{-1}$) was obtained following distillation in 2 mol L^{-1} NaOH for 1 h, suggesting that in the absence of additional fixable cations (i.e., K^+) quantitative removal of NEA can be achieved with 2 mol L^{-1} NaOH solution. Distillation of NH_4-saturated vermiculite also revealed that when the K:Na ratio increased from 0.005 to 0.05 the amount of NEA released by 1 *M* NaOH decreased from approximately 67 $cmol_c\ kg^{-1}$ to 5 $cmol_c\ kg^{-1}$.

While the identification of NEA released from purified clay minerals can be used to isolate the effect of a single competing ion such as K^+ on the release of NEA during alkaline hydrolysis, the release of NEA from soil is complicated due to the change of K^+ concentration with depth. Also, the variable clay mineralogical composition and clay content of surface soils along with the change in clay content with depth present additional challenges when evaluating the release of NEA during hydrolysis. Therefore, the release of NEA may change in response to the concentration of solution and exchangeable K^+ found at depths deeper in the soil profile.

In Iowa, Hanway and Scott (1956) examined the amount of NEA released during alkaline hydrolysis of selected soil profile samples. Using duplicate distillation and HF extraction the authors compared the influence of soil depth on the quantification of NEA using profile samples of an Edina (fine, montmorillonitic, mesic Vertic Argialbolls) silt loam and profile samples of a Webster (fine-loamy, mixed, super-active, mesic Typic Endoaquolls) silty clay loam. The HF extraction resulted in the release of 2.2 $cmol_c\ kg^{-1}$ of NEA from the silt loam surface (0–13 cm) soil while 1.8 $cmol_c\ kg^{-1}$ of NEA was released by the silty clay loam surface (0–15 cm) soil. A greater amount of NEA was present in the A horizon of both soil profiles based on HF extraction, however a greater concentration of NEA was found in the B horizon when duplicate distillation was used to release NEA.

Hanway and Scott (1956) were able to determine the greatest difference in the amount of NEA released between HF extraction and duplicate distillation occurred when surface soil was analyzed. There was a difference of 1.8 $cmol_c\ kg^{-1}$ and 1.6 $cmol_c\ kg^{-1}$ of NEA between the two procedures for the silt-loam soil and silty clay loam soil, respectively. The subsoil samples removed from the B and C horizon of the two soil profiles indicated the two methods used to quantify native NEA differed by less than 0.3 $cmol_c\ kg^{-1}$. Hanway and Scott (1956) concluded by stating that the methods evaluated released a similar amount of NEA from subsoil. The higher concentration of solution and exchangeable K^+ in surface soil could have interfered with the removal of NEA during NaOH distillation thereby reducing the recovery of NEA in comparison to HF extraction.

SUMMARY

A soil test that accurately accounts for the fraction of organic-N mineralized during the growing season could provide a site-specific index of native soil N fertility. Chemical and biological methods have been utilized to ascertain the portion of total soil N which provides the most accurate indication of plant available N. Biological methods have historically provided the most accurate prediction of N mineralization; however, lengthy analytical procedures have limited the use of biological methods (i.e., soil incubation) as routine N soil test methods. Chemical methods offer the advantage of timely analysis; however, the prevailing belief that chemical methods cannot reproducibly provide an index of biologically labile soil N impeded the wide-spread use of chemical N soil test methods. The development of the ISNT by researchers at the University of Illinois reinvigorated the search for a rapid, accurate, and consistent soil-based N test method. The rigorous evaluation of ISNT by researchers across the country produced conflicting conclusions regarding the ability of ISNT to provide meaningful adjustments to N fertilizer rate recommendations for corn. The influence of soil texture and sampling depth on N recovery led to the comparison of ISNT and DSD for rice grown on silt-loam soils.

Rice yield has been shown to decline as seeding date is delayed; however, the influence of seeding date on rice response to N fertilization is not as well understood for drill-seeded, delayed flood rice. Nitrogen uptake of late-seeded rice may differ from rice planted within an optimal time period due to morphological responses (i.e., tillering, dry matter accumulation, etc.) established during the vegetative growth phase. Modifying fertilizer N management decisions based on seeding date may be necessary to efficiently manage fertilizer N under environmental conditions that potentially influence plant growth and N uptake of rice grown in Arkansas.

While native NEA is considered unavailable or only very slowly available to plants and microorganisms, recently fixed NH_4–N exhibits a tendency to become available during the growing season. The dynamic equilibrium between solution, exchangeable, and NEA suggests that a change in the concentration of one form of NH_4–N in soil can lead to a readjusted equilibrium among the other forms of soil inorganic NH_4–N. Nonexchangeable ammonium concentration changes in subsoil following the growing season. Acid digestion was initially utilized to quantify clay fixed NH_4–N and subsequent modifications of the original acid digestion procedure sought to avoid interference by acid soluble organic-

N compounds and thereby provide a more accurate representation of native NEA. Alkaline distillation has also been used to quantify NEA. However, the presence of a high concentration of solution and exchangeable K^+ typically found in surface soil limited the quantity of NEA released during alkaline distillation.

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CHAPTER THREE

ALKALINE HYDROLYZABLE-NITROGEN OF CLAYEY SOILS PREDICTS NITROGEN REQUIREMENT OF RICE IN ARKANSAS

ABSTRACT

The Illinois Soil Nitrogen Test (ISNT) and the Nitrogen Soil Test for Rice (N-STaR) quantify alkaline-hydrolyzable N (AH-N) and can potentially improve nitrogen (N) fertilization of cereal crops by predicting an accurate and site-specific fertilizer N rate; however, neither method has been evaluated when rice (*Oryza sativa* L.) is grown on clayey soils in Arkansas. Therefore, 16 N response trials were conducted from 2007 to 2012 in order to correlate ISNT and N-STaR to indicators of rice response to N fertilization and to develop a fertilizer N rate calibration curve capable of predicting the fertilizer N rate required to achieve 95% relative grain yield (RGY). Soil samples were collected to a 60-cm depth in consecutive 15-cm intervals and selected rice response parameters were regressed on the AH-N concentration of individual as well as depth averaged soil sampling intervals (i.e., 0–30, 0–45, and 0–60 cm). The percentage of RGY when no N was applied (% RGY_{0N}) was significantly and positively correlated to AH-N concentration measured at each sampling depth, except for the 45- to 60-cm depth. The coefficient of determination (r^2) was maximized for % RGY_{0N} using ISNT ($r^2 = 0.80$) and N-STaR ($r^2 = 0.82$) when clayey soils were sampled to a 30-cm depth. Calibration of the fertilizer N rate required to achieve 95% RGY revealed that AH-N was able to explain 83% of the variability in calibration based on a 0- to 30-cm sampling depth for ISNT and N-STaR. Results of this study indicate that when clayey soils are properly sampled at 0- to 30-cm, ISNT and N-STaR are capable of accurately predicting fertilizer N responsiveness of rice grown in Arkansas.

INTRODUCTION

Ensuring the long-term sustainability of U.S. domestic crop production will require a critical evaluation of the social, economic, and environmental impact of currently acceptable agricultural production practices. Nitrogen fertilizer represents a substantial input cost for producers and when properly managed can positively affect profitability while minimizing the potentially detrimental influence that over-fertilization can have on the environmental quality of contiguous water resources (e.g., lakes, streams, reservoirs, etc.). A yield goal approach to N fertilizer management relies on the assumption that a positive relationship exists between percentage of the RGY and fertilizer N rate. However, positive as well as negative relationships have been documented for rice yield (Roberts et al., 2008) indicating that this approach is not suitable for all soils and environmental conditions. The development of site-specific, soil-based N tests such as ISNT by researchers at the University of Illinois and N-STaR by researchers at the University of Arkansas signified the need to provide an alternative to the yield goal approach to fertilizer N management. The ISNT and N-STaR soil test methods attempt to quantify the available NH_4–N as well as labile soil N, which is mineralized during the growing season, to predict the crop's fertilizer N response.

The ISNT was initially developed as a simple chemical procedure performed directly on soil without prior extraction or acid hydrolysis to reproducibly quantify AH-N, mainly as amino sugar-N (Roberts et al., 2009a). The relationship that existed between ISNT values and indicators of corn (*Zea mays* L.) response to N fertilization such as fertilizer N use efficiency (FNUE) indicated that ISNT may be a suitable site-specific N soil test method (Mulvaney et al., 2006). Therefore, the ISNT method was originally envisioned as a way of improving FNUE, increasing profits, and limiting the negative environmental impacts of excessive N fertilization associated with corn production (Khan et al., 2001). Additional field studies however produced positive (Mulvaney et al., 2006; Klapwyk and Ketterings et al., 2006; Bushong et al., 2007; Williams, 2007; Lawrence et al., 2009) as well as negative (Barker et al., 2006; Laboski et al., 2008; Osterhaus et al., 2008; Spargo et al., 2009) conclusions regarding the ability of the ISNT to provide site-specific and yield-maximizing fertilizer N rate recommendations for corn. These conflicting results in addition to the conclusion of Wang et al. (2001) widely confirmed the belief that a chemical method such as ISNT could not reliably quantify a biologically labile fraction of soil N across diverse sites and geographies.

The separation of corn fields based on fertilizer N responsiveness was made using ISNT critical soil test values developed using a 30-cm sampling depth. This limited sampling depth protocol only considered a portion of the rooting depth of corn which has been shown to extend to an average depth 90-cm (Dwyer et al., 1988) and fluctuations in plant available N in the subsoil could not be accurately represented by the ISNT soil test value. The inability of the ISNT to accurately predict fertilizer N response due to soil moisture stress, high seeding rate or the use of an inappropriate sampling depth was recognized by Mulvaney et al. (2006) and could be a prominent factor contributing to the mixed results of the ISNT critical value for soils across the U.S. Corn Belt observed by Laboski et al. (2008). Separation of sites based on soil drainage class or previous crop did not improve the correlation between ISNT and the economically optimal N rate (EONR) when soil was sampled to a 15-cm depth; thus Laboski et al. (2008) concluded that the ISNT was not suitable for predicting the N requirement of corn. The compilation of sites from across a wide geographic region without separating sites based on regional or soil textural differences as well as the use of a limited sampling depth could have contributed to the weak correlation (r^2 = 0.17) observed between ISNT and EONR for corn. Research with rice indicated the use of a sampling depth which corresponded to the majority of a rice crop's effective rooting depth (i.e., 0–45 cm) was necessary to develop a fertilizer N rate calibration curve (r^2 = 0.66) for ISNT when rice was grown on silt-loam soil in Arkansas (Roberts et al., 2011).

The N-STaR soil test method was developed to provide a rapid and more precise alternative to ISNT (Roberts et al., 2009a). This method utilizes 10 mol L^{-1} NaOH direct steam distillation (DSD) to quantify labile organic-N mainly in the form of amino sugar-N and amino acid-N, as well as inorganic-N in the form of NH_4–N (Roberts et al., 2009a), which provides a similar measure of AH-N as ISNT (Bushong et al., 2008). Initially, a comparison of the amount of AH-N quantified using 2, 5, and 10 mol L^{-1} NaOH DSD was found to be significantly correlated to the amount of N mineralized during a 14 d anaerobic incubation using 25 surface (i.e., 0–15 cm) soils from the south-central and Midwestern U.S. (Bushong et al., 2008). Further comparison of the ISNT and 10 mol L^{-1} NaOH DSD methods produced a nearly 1:1

linear relationship, which indicated that DSD could adequately quantify AH-N across different cropping systems and geographic regions.

Chemical soil test methods which have accurately predicted the N responsiveness of rice grown in the greenhouse have not been able to provide similar accuracy when evaluating rice response to N fertilization in the field (Wilson et al., 1994). Therefore, prior to the implementation of N-STaR in field trials, AH-N was quantified to a 60-cm depth and this sampling depth protocol was based on previous research indicating the rooting depth of rice grown on silt loam soils (Beyrouty et al., 1988). Roberts et al. (2011) were able to show that N-STaR soil test values obtained using a 0- to 45-cm sampling depth provided the most accurate prediction of a yield maximizing fertilizer N rate for rice grown on silt loam soils. Several possible explanations for the observed accuracy of the N-STaR N rate recommendations were put forth by Roberts et al. (2011), including: the high and consistent FNUE of rice grown using the drill-seeded, delayed flood production system; a relatively constant N mineralization rate of flooded rice soils; proper representative soil sampling depth from the rice crop's effective rooting zone; and the ability of N-STaR to provide a precise measurement of AH-N.

The identification of an appropriate N-STaR sampling depth protocol led to the successful field validation of this method for drill-seeded, delayed flood rice produced on silt loam soils (Roberts et al., 2013a). Validation trials were useful in identifying sites where the current fertilizer N rate recommendation failed to produce a maximum grain yield due either to under- or over-fertilization of rice. Roberts et al. (2013a) indicated that for sites with a high concentration of native soil N, the adoption of N-STaR-based fertilizer N rates would have achieved a significantly greater grain yield in comparison to the traditional university research-based recommended N rate (i.e., 168 kg N ha^{-1}) while reducing total fertilizer N addition by as much as 118 kg N ha^{-1} in some cases. The incorporation of N-STaR into routine soil testing has the benefit of offering site-specific N rate recommendations to commercial rice producers while avoiding the higher uncertainty associated with a yield goal approach to fertilizer N rate recommendation.

Arkansas is the largest producer of rice in the nation with approximately 500,000 ha^{-1} harvested annually. Rice produced on silt loam soils accounts for a majority of the commercial production hectarage; however, in 2012 an average of 43% of rice grown in Arkansas was produced on clay and clay loam soil (Hardke and Wilson, 2013). Continued success of Arkansas rice production requires the efficient management of fertilizer N on both silt-loam soil and clayey soils to optimize producer profitability across a majority of the commercial rice production hectarage. Fertilizer N movement is restricted in fine-textured soils due to the limited diffusion and high cation exchange capacity of clayey soils. Therefore, current fertilizer N recommendations for rice grown on clayey soils include an additional 34 kg N ha^{-1} to achieve optimum grain yield in comparison to rice grown on silt loam soils (Norman et al., 2013).

Despite the successful implementation of N-STaR N rate recommendations for rice grown on silt loam soils, it has not been possible to utilize the N-STaR fertilizer rate calibration curve developed for silt loam soils when rice is grown on clayey soils in Arkansas. The results of Williams et al. (2007) and Bushong et al. (2007) clearly indicated the ability of AH-N soil test methods to predict fertilizer N rates were improved by separating sites based on drainage classification and geographic region, respectively. The ability of N-STaR to predict fertilizer N rates is directly linked to soil texture and rice rooting depth. As a result of these relationships and shifts toward more rice production on clayey soils, it has become clear there is a growing need for a fertilizer rate calibration curve specifically for clayey soils (Roberts et al., 2011; Roberts et al., 2013a). Therefore, the first objective of this study was to utilize ISNT and N-STaR soil test methods to correlate the concentration of AH-N to rice N response parameters including total N (TN) uptake, check plot grain yield, as well as % RGY_{0N}. The second objective was to develop 95% RGY N fertilizer rate calibration regression equations based on ISNT and N-STaR soil test values for clayey soils cropped to rice in Arkansas.

MATERIALS AND METHODS

Fertilizer Nitrogen Response Trials

Sixteen fertilizer N rate trials were conducted with rice grown on clayey soils in Arkansas from 2007 to 2012 at experiment stations (9 site-years) and in commercial production fields (7 site-years). The year of each N rate trial, soil series, and previous crop are presented in Table 3.1. Sufficiency of Mehlich-3 extractable nutrients (i.e., P, K, Ca, Mg, S, Na, Fe, Mn, Zn, and Cu; Mehlich, 1984) for samples from each experimental site were interpreted in accordance with University of Arkansas Cooperative Extension Service guidelines for the production of drill-seeded, delayed-flood rice (Hardke, 2013) to ensure N was the only potentially yield limiting nutrient. Weed, disease, and insect pest control recommendations were also utilized in accordance with University of Arkansas Cooperative Extension Service guidelines for the production of drill-seeded, delayed flood rice (Hardke, 2013). The rice cultivar selected at each experiment station was Wells while the fertilizer N rate trials conducted in commercial production fields included hybrid or pure-line rice cultivars which were selected by the producer but would require a similar yield-maximizing N rate as Wells. At each experiment station field, rice was drill-seeded at a rate of 134 kg ha^{-1} in plots that were nine rows wide (19 cm row spacing) and 4.9-m in length. The fertilizer N source was urea (460 g N kg^{-1}) and the total N rates were split applied with the first application immediately prior to flooding, termed preflood (PF), by broadcasting fertilizer N onto a dry soil surface when the rice plant was at a four- to five-leaf growth stage. The second application occurred at beginning internode elongation (BIE) by broadcasting urea fertilizer directly into the floodwater. Ammonia volatilization from surface-applied urea fertilizer to clayey soils can represent a substantial N loss mechanism when more than 7 d are required to establish a permanent flood (Roberts et al., 2011). Therefore, to limit ammonia volatilization when N rate trials were conducted in commercial production rice fields, urea fertilizer was treated with the urease inhibitor NBPT (n-butyl-thiophosphoric tiramide) by blending 1 kg of prilled urea with 3.1 mL of Agrotain Ultra (Koch Agronomic Services, LLC., Wichita, KS). The N treatments at each site were arranged as a randomized complete block with four blocks using total N (PF + BIE) rates of: 0, 101 (51 + 50), 134 (84 + 50), 168 (118 + 50), 202 (152 + 50) and 235 (185 + 50) kg N ha^{-1} from 2007 to 2010; fertilizer N rates of: 0, 50, 101 (51 + 50), 151 (101 + 51), 202 (152 + 50) and 252 (202 + 50) kg N ha^{-1} were used from 2011 to 2012. A permanent flood was established following PF fertilizer N application and was maintained for the duration of the growing season until the rice plant reached physiological maturity.

Soil Sampling and Analysis

Four soil cores were collected immediately prior to PF fertilizer N application when rice was at a four- to five-leaf growth stage from each of the no N (0 kg N ha^{-1}) control plots in consecutive 15-cm intervals to a 60-cm depth using a Dutch Auger Probe (AMS Inc., American Falls, Idaho). The 16 individual soil cores were not composited, but remained separated according to the individual sampling depth (i.e., 15 cm depth interval) and the blocking structure established in the field. Soil samples were dried at 60°C within 24 to 48 h after removal from the field and subsequently ground to pass through a 2-mm sieve prior to chemical analysis. Inorganic NH_4–N and NO_3–N were determined colorimetrically

(Mulvaney, 1996) and soil TN was determined by dry combustion (Bremner, 1996) using an Elementar CN Variomax (Elementar Americas, Inc., Mt. Laurel, NJ). Soil pH 1:2 (w/v) was determined in the supernatant (Thomas, 1996) by Ag/AgCl pH electrode. Alkaline hydrolyzable-N was determined using ISNT (Khan et al., 2001) and N-STaR (Bushong et al., 2008; Roberts et al., 2009a) soil test methods. The ISNT procedure utilizes mild temperature and alkaline conditions (i.e., 2 mol L^{-1} NaOH at 50°C) during a 5 h diffusion period and N-STaR utilizes 10 mol L^{-1} NaOH DSD during an approximate 7 min distillation period. Both methods use boric acid [4% H_3BO_3 (w/v)] to trap the NH_3 released during alkaline hydrolysis. Following either diffusion or distillation, the quantity of N was determined by titrating (719 S Titrino, Metrohm Ltd., Herisau, Switzerland) the boric acid to a predetermined pH endpoint with 0.01 mol L^{-1} H_2SO_4. Mean pH, inorganic-N, and TN of the soils utilized in this study are presented in Table 3.2.

Measurement of Total Nitrogen Uptake and Grain Yield

Total N (TN) uptake was determined by hand harvesting the aboveground plant biomass from a 0.91-m section of the first inside bordered row of each unfertilized plot at 50% heading, the time representing the maximum accumulation of aboveground N in rice tissues (Norman et al., 1992). Plant tissue samples were dried at 60°C within 24 to 48 h after removal from the field. The dry biomass was weighed and a subsample ground to pass a 1-mm sieve for analysis at the University of Arkansas Diagnostic Laboratory. Total N in the plant tissue was determined by combustion (Campbell, 1992) using an Elementar Rapid N (Elementar Americas, Inc., Mt. Laurel, NJ). Total N uptake was calculated as plant tissue N (g N g tissue^{-1}) multiplied by the dry aboveground biomass and scaled to an area basis.

The harvested grain from five center rows of each plot was weighed and a subsample removed to determine moisture content using a Grain Moisture Tester (DICKEY-john Corp., Auburn, IL). Grain weights were adjusted using a moisture content of 120 g H_2O kg^{-1} and the harvested grain yield reported on an area basis (i.e., kg rough rice ha^{-1}). Relative grain yield was determined by dividing the yield of the unfertilized plot by the maximum grain yield obtained at that location and multiplying by 100 to express the result as a percentage of the RGY. The fertilizer N rate required to achieve 95% RGY was obtained by fitting a linear regression model to the yield curve obtained from each N response trial.

Statistical Analysis

Regression analysis was performed using PROC REG in SAS v9.2 (SAS Institute Inc., Cary, NC) to evaluate the significance ($P < 0.05$) of the linear and quadratic coefficients of the regression models. The regression models were evaluated for outliers by examining the distribution of studentized residuals ($-2 \leq$ studentized residual ≤ 2) and outliers were removed when deemed appropriate and this is reflected in the number of observations used to fit each regression model. Correlation of TN uptake with AH-N values was conducted on 10, 11, or 12 of the 16 sites used in this study. Sites 1, 9, and 16 were not included in the regression analysis because TN uptake values were not recorded. Site 16 was identified as non-responsive to N fertilization based on a nonsignificant ($P > 0.05$) one-way analysis of variance for N rate. Therefore, site 16 was removed from the % RGY_{0N} regression curves as well as the 95% RGY fertilizer N rate calibration regression curves and this is reflected in the number of observations used to fit these linear regression models. The concentration of AH-N was determined within individual 15-cm depth intervals as well as depth averaged soil sampling intervals of: 0- to 30-, 0- to 45-, and 0- to 60-cm (Roberts et al., 2011). The rice plant N response parameter of interest was regressed on the mean AH-N concentration for individual 15-cm depth intervals as well as depth averaged soil sample intervals. In order to develop the linear, fertilizer N rate calibration regression equations, the N rate required to achieve 95% RGY was regressed on the AH-N concentration as determined by ISNT or N-STaR for each sampling depth.

RESULTS AND DISCUSSION

Across sites and soil depths AH-N concentration ranged from 38 to 378 mg N kg soil^{-1} using the ISNT method and 52 to 364 mg N kg soil^{-1} with the N-STaR method (Table 3.3). The range of ISNT soil test values in this study exceeded the range of ISNT values reported by Wall et al. (2010) for clayey soils of North Carolina. However, a stratification of ISNT values was observed for clayey soils sampled to a 60-cm depth which was similar to the results of Wall et al. (2010). There was a tendency for AH-N, as quantified by ISNT or N-STaR, to decrease as sampling depth increased to 60-cm across a majority of the sites (i.e., Sites 1, 2, 5, 6, 7, 9, 10, 13, 15, and 16) evaluated in this study although this was not a consistent trend. Two of the 16 sites evaluated exhibited slight numerical increases in AH-N concentration as determined by ISNT or N-STaR as sampling depth increased from 0- to 15- to 15- to 30-cm and then decreased when depths > 30-cm were evaluated. Two of the 16 sites evaluated using ISNT and 1 of the

16 sites evaluated using N-STaR essentially exhibited no change in AH-N concentration with depth. The N-STaR method tended to quantify a greater concentration of AH-N compared to ISNT across a majority of sites and soil depths which is in agreement with the results of Roberts et al. (2011) who evaluated the analytical methods (ISNT and N-STaR) for silt loam soils.

The sites included in this study were selected to represent a wide range in native soil N fertility which was reflected by differences in TN uptake, check plot grain yield, % RGY_{0N}, and fertilizer N rate required to achieve 95% RGY when rice was grown on clayey soils in Arkansas. Total N uptake measured at 50% heading represents the maximum accumulation of N in plant tissue that occurs during the growing season (Norman et al., 1992). Total N uptake by rice when no fertilizer N was applied ranged from 24 to 219 kg N ha^{-1} indicating native soil N availability varied across sites (Table 3.3). Additionally, a wide range of fertilizer N responsiveness was measured across sites as displayed by a range of 27 to 100% RGY when no fertilizer N was applied and a range of 0 to 218 kg ha^{-1} of fertilizer N required to achieve 95% RGY.

Correlation of Rice Response to Alkaline Hydrolyzable-Nitrogen

A significant and positive relationship was observed between AH-N, quantified by ISNT and N-STaR, and TN uptake for rice grown on clayey soils for each sampling depth evaluated with the exception of the 45- to 60-cm depth (Table 3.4). Site 14 was determined to be an outlier based on the value of the studentized residual; therefore, this site was removed from the 0- to 15-cm depth ISNT linear regression and the model was refit (Figure 3.1A). Sites 13 and 14 were identified as outlying observations for the 0- to 15-cm depth N-STaR linear regression; therefore, these two sites were removed and the model was refit (Figure 3.2A). For all remaining depth averaged correlations of TN uptake versus AH-N, sites 13, 14 and 15 were statistical outliers and not included in the regression analysis (Figure 3.1B–D; Figure 3.2B–D).

Sites 13, 14, and 15 were located in commercial production rice fields in 2012. The measured TN uptake was greater at sites 13, 14, and 15 than the predicted TN uptake. The specific reasons why these sites responded differently are not fully known, but interplant competition varies greatly with changes in row spacing or seeding rate (Jones and Snyder, 1987) and reduced interplant competition within the row as a result of lower seeding rates could have contributed to greater TN uptake in these commercial production rice fields.

Roberts et al. (2011) reported a significant and positive correlation between AH-N, quantified by ISNT or N-STaR, and TN uptake of rice when silt loam soils were sampled to a 60-cm depth in 15-cm intervals. The results of the present study revealed the greatest r^2 value for an individual 15-cm sampling depth interval occurred at 15- to 30-cm for ISNT and at 0- to 15-cm for N-STaR (Table 3.4). The 0- to 30-cm sampling depth produced the greatest r^2 value of 0.60 and 0.64 for ISNT and N-STaR, respectively, when depth intervals were combined in this study.

Check plot grain yield regressed on the ISNT soil test value produced a significant and positive linear relationship for each of the sampling depth intervals evaluated with the exception of the 45- to 60-cm depth (Table 3.4). Regression analysis revealed that the ability of ISNT to predict check plot grain yield improved as sampling depth increased to 30-cm and then decreased when depths > 30-cm were evaluated. The 15- to 30-cm depth produced the greatest r^2 value of 0.73 of any individual 15-cm sampling depth interval while AH-N concentration of the 0- to 30-cm depth produced the greatest r^2 value of 0.64 when depth intervals were combined.

Check plot grain yield regressed on the N-STaR soil test value produced a significant linear regression for each of the sampling depth intervals evaluated with the exception of the 0- to 15-, 45- to 60-, and 0- to 60-cm depths (Table 3.4). Soil sampling depth had an apparent influence on each of the N-STaR linear regression models and as sampling depth increased the r^2 values increased to a 30-cm depth and then decreased as sampling depth increased to 60-cm. The influence of sampling depth on the accuracy of N-STaR grain yield correlation and fertilizer N rate calibration regression models has previously been demonstrated by Roberts et al. (2011). The results of this study indicated the 15- to 30-cm depth produced the greatest r^2 value of 0.71 for any individual 15-cm sampling depth and when depth intervals were arithmetically combined the AH-N concentration (i.e., ISNT or N-STaR values) of the 0- to 30-cm depth produced the greatest r^2 value of 0.61.

Factors other than nutrient availability, such as environmental conditions, cultivar, and seeding date can influence rice grain yield (Slaton et al., 2003). Yield comparisons among sites based on % RGY_{0N} can account for non-nutrient limiting factors thus providing a better representation of yield response to N fertilization (Roberts et al., 2011). Percentage of RGY_{0N} regressed on AH-N concentration as determined by ISNT or N-STaR was significant and positive across all of the sampling depths evaluated with the exception of the 45- to 60-cm depth (Table 3.4; Figure 3.3A–D and 3.4A–D). A similar influence of sampling depth on the ability of AH-N to predict % RGY_{0N} was observed with AH-N prediction of check plot grain yield.

The ISNT and N-STaR methods indicated the greatest r^2 value was obtained when % RGY_{0N} was regressed on AH-N concentration of the 0- to 30-cm depth. Previous research has emphasized the importance of identifying an appropriate soil sampling protocol during the correlation and calibration of N soil test methods (Roberts et al., 2009b; Roberts et al. 2013b). Root growth has been documented to a 40-cm depth and > 90% of the total root system was located within the 0- to 20-cm depth when transplanted rice was grown on clay or clay loam soils (Sharma et al., 1987; Mambani et al., 1989). The sampling protocol utilized in this study accounted for N availability at a soil depth > 15-cm which could potentially contribute to N assimilation and rice grain yield. Alkaline hydrolyzable-N concentration of the 0- to 30-cm depth, determined by ISNT or N-STaR, was positively correlated with TN uptake, check plot grain yield, and % RGY_{0N} and exhibited the greatest r^2 values among the depth averaged soil sampling intervals.

Fertilizer Nitrogen Rate Calibration

Calibrating a soil test method is necessary to determine whether the method is capable of providing accurate and meaningful adjustments to current fertilizer rate recommendations. The 0- to 45-cm sampling depth used to develop a fertilizer N rate calibration curve for N-STaR ($r^2 = 0.89$) was also used successfully to field validate N-STaR prescribed fertilizer N rates for rice grown on silt-loam soil in Arkansas (Roberts et al., 2013a). Identification of economically optimal N rates for rice produced in Arkansas indicated that current N rate recommendations were within a profitable range for sites exhibiting a positive response to fertilizer N addition (Watkins et al., 2010). However, to accurately identify an economically optimal N rate the native soil N availability has to be taken into consideration. The ISNT and N-STaR methods attempt to provide an index of potentially mineralizable-N which may become available over the course of the growing season. The ultimate goal of the ISNT and N-STaR soil test methods is to provide site-specific, yield maximizing fertilizer N rate recommendations.

Only sites that were responsive to fertilizer N addition were used to calibrate the ISNT and N-STaR methods. Site 16 had a native soil N availability that supported maximum rice yield without the addition of fertilizer N and was the only site that did not exhibit a positive response to N fertilization. Site 16 was pastureland for approximately 50 yr prior to the initiation of the fertilizer N rate trial and had the greatest concentration of exchangeable NH_4–N, soil TN, and AH-N in the surface (0–15 cm) soil of all sites evaluated (Tables 3.2 and 3.3).

Calibration of the fertilizer N needs for rice produced on clayey soils was achieved by regressing the fertilizer N rate required to achieve 95% RGY on the AH-N concentration, analyzed using either the ISNT or N-STaR method, of each sampling depth (0–15, 15–30, 30–45, 45–60, 0–30, 0–45, and 0–60 cm). A significant and negative linear relationship was identified across all of the sampling depths evaluated when the N rate required to achieve 95% RGY was regressed on ISNT values (Table 3.5; Figure 3.5A–D). The greatest r^2 value (0.84) among the individual 15-cm sampling depth intervals was achieved when the N rate required to achieve 95% RGY was regressed on the AH-N concentration of the 15- to 30-cm depth. Among the depth averaged soil sampling intervals, the regression model r^2 value improved as sampling depth increased, achieving the greatest r^2 values of 0.83 at the 0- to 30-cm depth and 0.84 at the 0- to 45-cm depth, but then decreased to 0.74 at the 0- to 60-cm depth.

Similar to the results of the ISNT fertilizer N rate calibration, a significant and negative linear relationship was identified for all of the sampling depths evaluated when the N rate required to achieve 95% RGY was regressed on N-STaR values (Table 3.5; Figure 3.6A–D). The greatest r^2 value (0.79) among the individual 15-cm sampling depth intervals occurred at the 15- to 30-cm depth, while among the depth averaged soil sampling intervals, the r^2 value reached a maximum of 0.83 at the 0- to 30-cm depth, but then decreased to 0.76 and 0.67 as sampling depth increased from 0- to 45- to 0- to 60-cm, respectively. Similar to the correlation of depth-averaged N-STaR values to rice response parameters (i.e., check plot TN uptake, check plot grain yield, and % RGY_{0N}), the greatest r^2 value for N-STaR calibration occurred using a 0- to 30-cm sampling depth. The results of this study are similar to the results of Roberts et al. (2011) who indicated that, among depth averaged soil sampling intervals, the 0- to 45-cm depth maximized the r^2 value for correlation of rice response as well as the calibration of N rate to achieve 95% RGY linear regression models using N-STaR when rice was grown on silt loam soils. The results of the present study using N-STaR for rice grown on clayey soils are encouraging and among the depth averaged soil sampling intervals, the greatest r^2 value of the correlation and calibration linear regression models coincided at the 0- to 30-cm sampling depth.

Alkaline hydrolyzable-N concentration averaged over sampling depth provided r^2 values which tended to increase as sampling increased to a 45-cm depth for ISNT and to a 30-cm depth for N-STaR indicating that sampling deeper than the surface 15-cm of soil improved the calibration of the ISNT and N-STaR soil test methods (Table 3.5; Figure 3.5A–C and Figure 3.6A–B). Alkaline hydrolyzable-N concentration of the 0- to 45-cm depth nominally improved the ISNT N rate calibration in comparison to the 0- to 30-cm depth. Therefore, the predictive ability of ISNT based on a 0- to 30- or 0- to 45-cm sampling depth would be expected to be approximately equivalent. Sample depth influence on N-STaR values indicated that increasing sampling depth from 0- to 15- to 0- to 30-cm improved N rate calibration and explained 83% of the variability in the fertilizer N rate required to achieve 95% RGY.

Watkins et al. (2010) identified an economically optimal N rate for rice grown at two sites on two different clay soils of 181 and 187 kg N ha^{-1} which is substantially lower than the current standard fertilizer N rate recommendation of 202 kg N ha^{-1} for rice grown on clayey soils in Arkansas. Differences such as those observed by Watkins et al. (2010) between the economically optimal N rate and standard N rate indicates a need for a N soil test to ensure the University of Arkansas N rate recommendations are accurate from an economic as well as an agronomic and environmental perspective. The 15 N responsive sites used to calibrate the fertilizer N rate required to achieve 95% RGY with AH-N (ISNT or N-STaR values) resulted in predicted fertilizer N rates ranging from 62 to 218 kg N ha^{-1} (Table 3.3). There were three sites (i.e., sites 7, 8, and 13) that would require an equivalent or even greater fertilizer N rate than the current research-based N rate of 202 kg N ha^{-1}, while 12 of the 15 sites evaluated in this study required less than the standard fertilizer N rate recommendation to achieve 95% RGY.

CONCLUSIONS

Alkaline hydrolyzable-N concentration, as estimated by ISNT and N-STaR methods, decreased throughout the soil profile to a 60-cm depth for a majority of clayey soils cropped to rice in Arkansas. However, there were some sites (i.e., Sites 4 and 14) that exhibited slight numerical increases in AH-N from 0- to 15- to 15- to 30-cm and then decreased as depth increased further to 60-cm, while a few sites (i.e., Sites 3, 11, and 12) basically exhibited no change with depth. This indicated the greatest AH-N concentration was not restricted to the surface (i.e., 0–15 cm) soil. Check plot TN uptake was significantly and positively correlated with AH-N, determined by ISNT or N-STaR, for each sampling depth evaluated with the exception of the 45- to 60-cm depth. Additional rice response parameters such as check plot grain yield and % RGY_{0N} were also positively correlated to ISNT and N-STaR values. When AH-N was quantified using either ISNT or N-STaR, the 0- to 30-cm depth was shown to produce linear regression models for TN uptake, check plot grain yield, and % RGY_{0N} that exhibited the greatest r^2 value among depth averaged sampling intervals.

Both the ISNT and N-STaR methods were successfully calibrated to predict the N rate required to achieve 95% RGY based on the AH-N concentration of clayey soils. The sites utilized to develop the correlation and calibration curves all exhibited a positive response to N fertilization and also exhibited a wide range among the fertilizer N rates required to achieve 95% RGY. The trend among r^2 values was similar between the ISNT and N-STaR correlation curves developed using depth averaged soil sampling intervals. The 0- to 30-cm depth produced the greatest r^2 value for the correlation to rice response compared to the 0- to 45- or 0- to 60-cm sampling depth when AH-N was quantified by ISNT or N-STaR. Whereas, the 0- to 30-cm depth produced the greatest r^2 value (0.83) among the depth averaged N-STaR calibration curves while the 0- to 30-cm and 0- to 45-cm depths produced the greatest r^2 values of 0.83 and 0.84, respectively, among the depth averaged ISNT calibration curves.

Further development of the ISNT and N-STaR fertilizer N rate calibration curves may require the identification of sites that require low amounts of N to optimize grain yield of rice grown on clayey soils, yet still exhibit a positive response to N fertilization. However, the relatively wide range of fertilizer N rates required to achieve 95% RGY observed in this study enabled the development of a fertilizer N rate calibration curve for ISNT and N-STaR. Sampling depth influenced fertilizer N rate calibration and the

ISNT and N-STaR fertilizer N rate calibration curves explained 83% of the variability in the 95% RGY fertilizer N rate when clayey soils were sampled at a 0- to 30-cm depth. The results of this study are encouraging and suggest the use of a proper sampling depth suitable for rice grown on clayey soils is crucial to the identification of correlation and calibration curves that maximize the predictive ability of the ISNT and N-STaR methods.

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Table 3.1. Year, soil series, taxonomic classification, field location, and previous crop for 16 sites sampled in Arkansas from 2007 to 2012.

Site	Year	Soil Series	Classification	Field Location[†]	Previous Crop
1	2007	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Soybean[‡]
2	2008	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Soybean
3	2008	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Rice[§]
4	2009	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Soybean
5	2009	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Rice
6	2009	Jackport	fine, smectitic, thermic, Chromic Epiaquerts	CP	Soybean
7	2009	Desha	very-fine, smectitic, thermic, Vertic Hapludolls	ES	Soybean
8	2010	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	CP	Soybean
9	2010	Jackport	fine, smectitic, thermic, Chromic Epiaquerts	CP	Soybean
10	2010	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Soybean
11	2010	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Rice
12	2012	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	ES	Rice
13	2012	Perry	very-fine, smectitic, thermic, Chromic Epiaquerts	CP	Soybean
14	2012	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	CP	Rice
15	2012	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	CP	Soybean
16	2012	Sharkey	very-fine, smectitic, thermic Chromic Epiaquerts	CP	Pasture

[†] ES = Experiment Station, CP = Commercial Production;
[‡] *Glycine max* L.
§*Oryza sativa* L.

Table 3.2. Mean soil pH_w, inorganic (NH_4 and NO_3)-N and total nitrogen (TN) of 16 sites sampled to a 60 cm depth in Arkansas from 2007 to 2012.

	pH†				NH_4–N‡				NO_3–N‡				TN§			
	Soil sample depth (cm)															
Site	0-15	15-30	30-45	45-60	0-15	15-30	30-45	45-60	0-15	15-30	30-45	45-60	0-15	15-30	30-45	45-60
					(mg N kg $soil^{-1}$)											
1	7.3	7.4	7.4	7.3	1	1	1	1	1	<1	<1	<1	1074	890	851	847
2	7.2	7.0	7.0	6.9	2	1	1	1	1	1	1	1	1124	946	966	899
3	7.0	7.0	7.0	6.9	1	1	1	<1	<1	<1	<1	<1	972	965	900	892
4	6.6	6.4	6.6	6.7	4	3	2	2	1	1	<1	<1	1125	1115	1008	975
5	7.4	7.3	7.1	6.6	3	2	2	2	3	3	2	2	1218	1063	970	906
6	6.8	6.6	6.3	6.5	4	2	1	2	2	<1	<1	<1	987	640	510	412
7	7.2	7.1	6.8	6.4	<1	<1	<1	<1	1	2	1	2	986	735	564	509
8	6.4	6.1	5.9	5.8	4	3	4	3	5	3	1	2	907	759	685	681
9	7.1	6.5	5.2	5.1	3	2	4	5	8	7	<1	1	957	565	500	509
10	6.8	6.8	6.7	6.6	2	1	1	<1	4	3	4	<1	-	-	-	-
11	7.0	6.9	6.9	6.8	1	<1	<1	<1	2	1	2	2	983	923	887	869
12	7.9	7.6	7.4	7.1	3	3	4	3	5	5	3	3	1001	973	954	937
13	6.8	7.2	6.9	6.6	4	3	3	3	3	3	3	3	1098	694	630	630
14	6.5	6.6	6.8	7.0	2	5	7	2	2	4	3	3	1222	1291	1155	791
15	7.0	6.4	5.2	5.1	3	3	5	5	2	1	1	3	1315	820	725	663
16	5.8	5.9	6.1	6.3	57	7	1	3	4	3	4	3	2444	1271	887	705

†pH determined on a 1:2 (m:v) soil-to-water suspension (Thomas, 1996).

‡Determined using salicylate colorimetric techniques (Mulvaney, 1996).

§Determined using dry combustion (Bremner, 1996)

Table 3.3. Mean and standard error of Illinois Soil Nitrogen Test (ISNT) and Nitrogen Soil Test for Rice (N-STaR) values, check plot total N (CPTN) uptake, check plot grain yield (CPGY), percentage of relative grain yield when no N fertilizer was applied (% RGY$_{0N}$), and the N rate required to achieve 95% RGY for 16 sites in Arkansas where rice was grown on clayey soils.

	ISNT†				N-STaR†				CPTN ‡	CPGY‡	RGY$_{0N}$‡	95%RGY N rate
	Soil sample depth (cm)											
Site	0-15	15-30	30-45	45-60	0-15	15-30	30-45	45-60				
	(mg N kg soil^{-1})								kg N ha^{-1}	kg rice ha^{-1}	%	kg N ha^{-1}
1	149 ± 4.6	142 ± 2.4	139 ± 7.8	118 ± 5.6	161 ± 3.4	139 ± 2.2	134 ± 2.3	131 ± 3.0	-	4389	68	123
2	156 ± 2.6	140 ± 9.5	137 ± 2.6	102 ± 5.2	173 ± 0.9	152 ± 3.4	175 ± 7.7	122 ± 6.9	95	5448	72	119
3	116 ± 5.6	115 ± 3.9	111 ± 2.2	105 ± 14.9	130 ± 5.9	130 ± 2.9	136 ± 4.0	126 ± 17.9	50	3329	46	176
4	142 ± 5.4	154 ± 6.7	138 ± 8.9	127 ± 2.0	151 ± 2.0	158 ± 5.8	140 ± 3.5	133 ± 3.6	96	8404	80	90
5	141 ± 4.9	130 ± 7.7	121 ± 9.2	113 ± 4.6	155 ± 3.7	149 ± 4.7	129 ± 11.4	127 ± 5.2	50	4712	54	129
6	133 ± 14.4	88 ± 3.5	67 ± 11.4	38 ± 4.5	140 ± 12.5	106 ± 3.1	75 ± 9.8	52 ± 4.6	58	4691	48	155
7	107 ± 3.5	83 ± 10.4	65 ± 8.4	55 ± 9.6	121 ± 3.5	100 ± 12.5	76 ± 13.0	67 ± 6.9	24	2220	27	218
8	100 ± 4.6	80 ± 4.3	68 ± 3.1	61 ± 0.9	127 ± 9.4	109 ± 3.7	80 ± 1.4	90 ± 6.8	53	3279	37	202
9	123 ± 9.6	79 ± 2.5	66 ± 4.0	60 ± 2.0	149 ± 17.3	93 ± 2.9	76 ± 5.3	70 ± 5.7	-	3329	41	185
10	147 ± 6.5	131 ± 13.4	110 ± 12.7	101 ± 10.1	164 ± 11.6	132 ± 7.7	123 ± 19.0	106 ± 17.0	76	4580	57	146
11	129 ± 1.8	126 ± 9.7	125 ± 1.2	124 ± 5.0	130 ± 6.5	140 ± 10.5	127 ± 6.9	134 ± 8.5	49	2865	38	173
12	127 ± 1.2	123 ± 3.7	129 ± 3.9	129 ± 2.8	134 ± 2.4	133 ± 2.4	135 ± 1.6	131 ± 2.1	63	4641	46	134
13	125 ± 14.4	69 ± 6.5	58 ± 4.7	54 ± 3.9	127 ± 17.0	83 ± 7.2	65 ± 4.9	59 ± 7.5	108	5700	54	202
14	156 ± 10.0	173 ± 2.8	163 ± 14.9	92 ± 21.7	154 ± 7.6	178 ± 11.8	161 ± 12.3	108 ± 24.3	219	10089	84	62
15	165 ± 3.5	95 ± 5.0	82 ± 7.6	66 ± 1.8	183 ± 1.8	108 ± 4.9	89 ± 5.4	72 ± 1.3	144	5650	53	180
16	378 ± 25.1	177 ± 16.1	114 ± 13.5	85 ± 7.8	364 ± 27.7	193 ± 13.7	125 ± 16.6	92 ± 10.0	-	8855	100	0

† Sites 1, 3-16 mean of four replicate determinations; site 2 mean of two replicate determinations; ‡ Mean of four replicate determinations

Table 3.4. Linear regression equations describing the relationship between check plot total N (TN) uptake, check plot grain yield and percent relative grain yield when no N fertilizer was applied (% RGY_{0N}) with alkaline hydrolyzable-nitrogen (AH-N) as quantified by the Illinois Soil Nitrogen Test (ISNT) and the Nitrogen Soil Test for Rice (N-STaR) for clayey soils cropped to rice in Arkansas from 2007 to 2012.

Dependent Variable	Depth (cm)	ISNT			N-STaR		
		n¶	Regression Equation	r^2	n	Regression Equation	r^2
Check plot TN Uptake†	0-15	12	$-91.3 + 1.23x$	0.51^{**}	11	$-130.7 + 1.37x$	0.74^{***}
	15-30	10	$-13.6 + 0.64x$	0.54^{*}	10	$-37.6 + 0.76x$	0.48^{*}
	30-45	10	$9.8 + 0.48x$	0.41^{*}	10	$7.4 + 0.45x$	0.45^{*}
	45-60	11	$57.2 + 0.09x$	0.01	11	$61.9 + 0.04x$	0.00
	0-30	10	$-41.7 + 0.84x$	0.60^{**}	10	$-81.1 + 1.04x$	0.64^{**}
	0-45	10	$-23.3 + 0.72x$	0.55^{*}	10	$-41.1 + 0.78x$	0.57^{*}
	0-60	10	$-5.5 + 0.60x$	0.43^{*}	10	$-24.3 + 0.68x$	0.47^{*}
Check plot grain yield‡	0-15	15	$-4467 + 69.6x$	0.40^{**}	15	$-1945 + 46.6x$	0.18
	15-30	15	$17603 – 278.8x + 1.4x^2$	0.73^{**}	15	$26091 – 393.3x +1.7x^2$	0.71^{***}
	30-45	15	$13860 – 231.6x + 1.3x^2$	0.58^{*}	14	$22424 – 389.7x + 1.9x^2$	0.59^{**}
	45-60	15	$1130 + 81.5x – 0.40x^2$	0.05	15	$3815 + 10.5x$	0.02
	0-30	15	$25311 – 406.5x +1.9x^2$	0.64^{**}	15	$52860 – 791.1x + 3.2x^2$	0.61^{**}
	0-45	15	$20406 – 331.6x + 1.6x^2$	0.63^{**}	15	$-1550 + 49.7x$	0.31^{*}
	0-60	15	$29124 – 525.6x +2.6x^2$	0.55^{**}	15	$61.9 + 39.4x$	0.20
% RGY_{0N}§	0-15	15	$-34.3 + 0.65x$	0.57^{**}	15	$-23.6 + 0.53x$	0.36^{*}
	15-30	14	$155.4 – 2.4x + 0.01x^2$	0.72^{**}	15	$-1.3 + 0.43x$	0.50^{**}
	30-45	15	$18.3 + 0.34x$	0.50^{**}	15	$17.9 + 0.31x$	0.45^{*}
	45-60	15	$34.6 + 0.21x$	0.16	15	$32.3 + 0.21x$	0.15
	0-30	15	$127.9 – 1.9x + 0.01x^2$	0.80^{***}	14	$-70.2 + 0.89x$	0.82^{***}
	0-45	15	$-6.1 + 0.51x$	0.65^{***}	15	$-15.8 + 0.54x$	0.58^{***}
	0-60	15	$184.1 – 3.1x + 0.02x^2$	0.69^{***}	15	$236 – 3.7x + 0.02x^2$	0.66^{**}

*, **, and *** denote significance at the 0.05, 0.01, and 0.001 level of probability, respectively.

†x, Alkaline hydrolyzable-N (mg N kg $soil^{-1}$); y, TN Uptake (kg N ha^{-1}) from unfertilized plots;

‡x, Alkaline hydrolyzable-N (mg N kg $soil^{-1}$); y, check plot grain yield (kg grain ha^{-1}); ¶n, number of sites utilized for regression equations.

§x, Alkaline hydrolyzable-N (mg N kg $soil^{-1}$); y, percentage of relative grain yield when no N fertilizer was applied (% RGY_{0N});

Table 3.5. Linear regression equations describing the relationship between the fertilizer N rate required to achieve 95% relative grain yield (95% RGY) and the concentration of alkaline hydrolyzable-nitrogen (AH-N) as determined by the Illinois soil nitrogen test (ISNT) and the Nitrogen Soil Test for Rice (N-STaR) for clayey soils sampled to a 60 cm depth in Arkansas from 2007 to 2012.

Dependent Variable	Depth (cm)	ISNT			N-STaR		
		n[‡]	Regression Equation	r^2	n	Regression Equation	r^2
95% RGY[†]	0-15	14	442 – 2.2x	0.72^{***}	14	430 – 1.9x	0.49^{**}
	15-30	15	301 – 1.3x	0.84^{***}	15	338 – 1.5x	0.79^{***}
	30-45	15	270 – 1.1x	0.76^{***}	15	269 – 1.0x	0.63^{***}
	45-60	15	228 – 0.8x	0.35^{*}	15	239 – 0.8x	0.34^{*}
	0-30	15	373 – 1.8x	0.83^{***}	14	440 – 2.1x	0.83^{***}
	0-45	15	336 – 1.5x	0.84^{***}	15	366 – 1.6x	0.76^{***}
	0-60	15	316 – 1.5x	0.74^{***}	15	339 – 1.5x	0.67^{***}

[*] Statistical significance at $P < 0.05$; [**] Statistical significance at $P < 0.01$; [***] Statistical significance at $P < 0.001$;
[†] x, Alkaline hydrolyzable-N (mg N kg soil^{-1}); y, fertilizer N rate (kg N ha^{-1}) required to achieve 95% relative grain yield (95% RGY);
[‡] n, Number of sites utilized for development of regression equations.

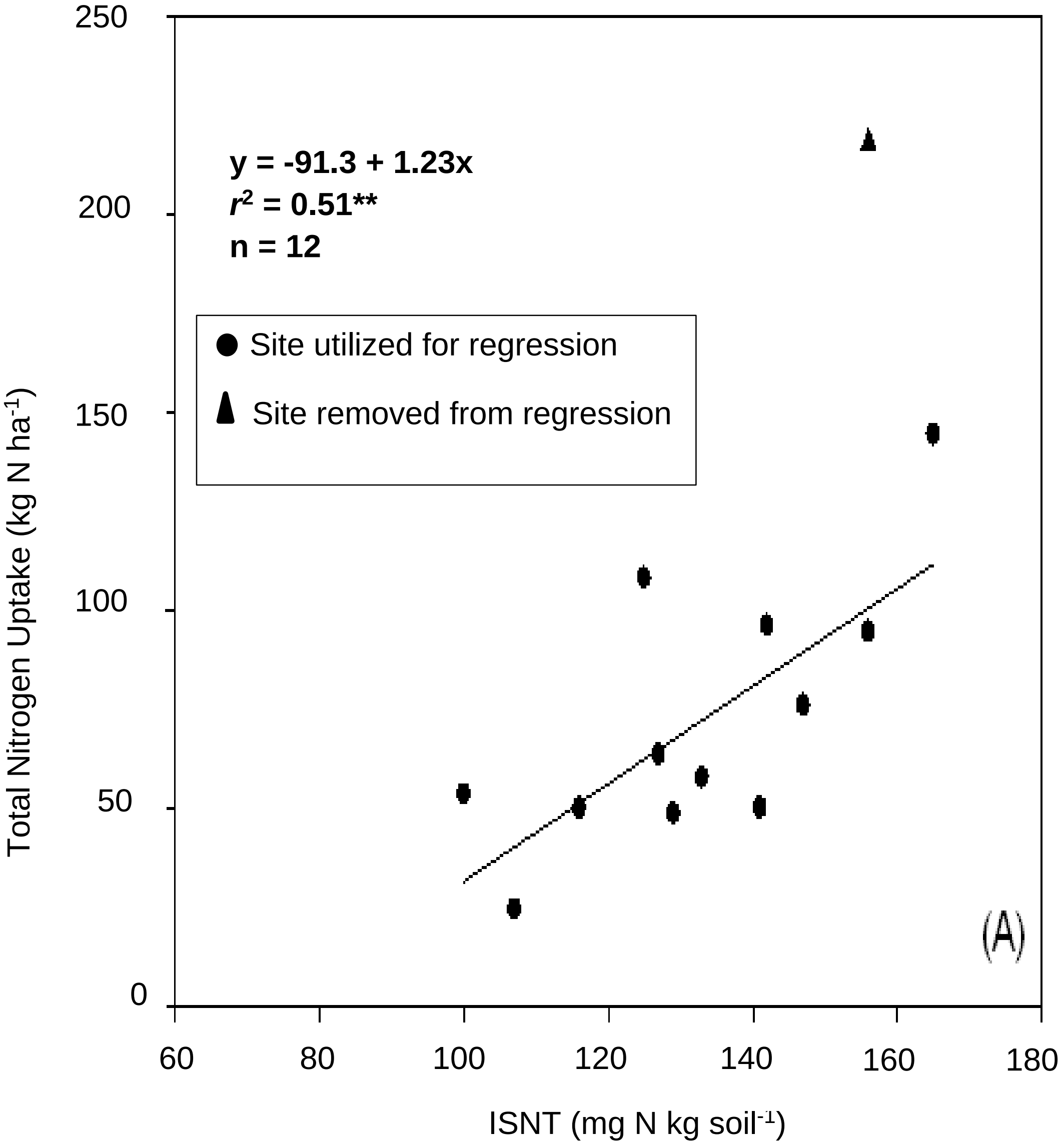

Figure 3.1A. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with regression equations developed using a soil sampling depth of 0- to 15-cm. ** indicates a significant linear regression at the 0.05 and 0.01 level of probability, respectively.

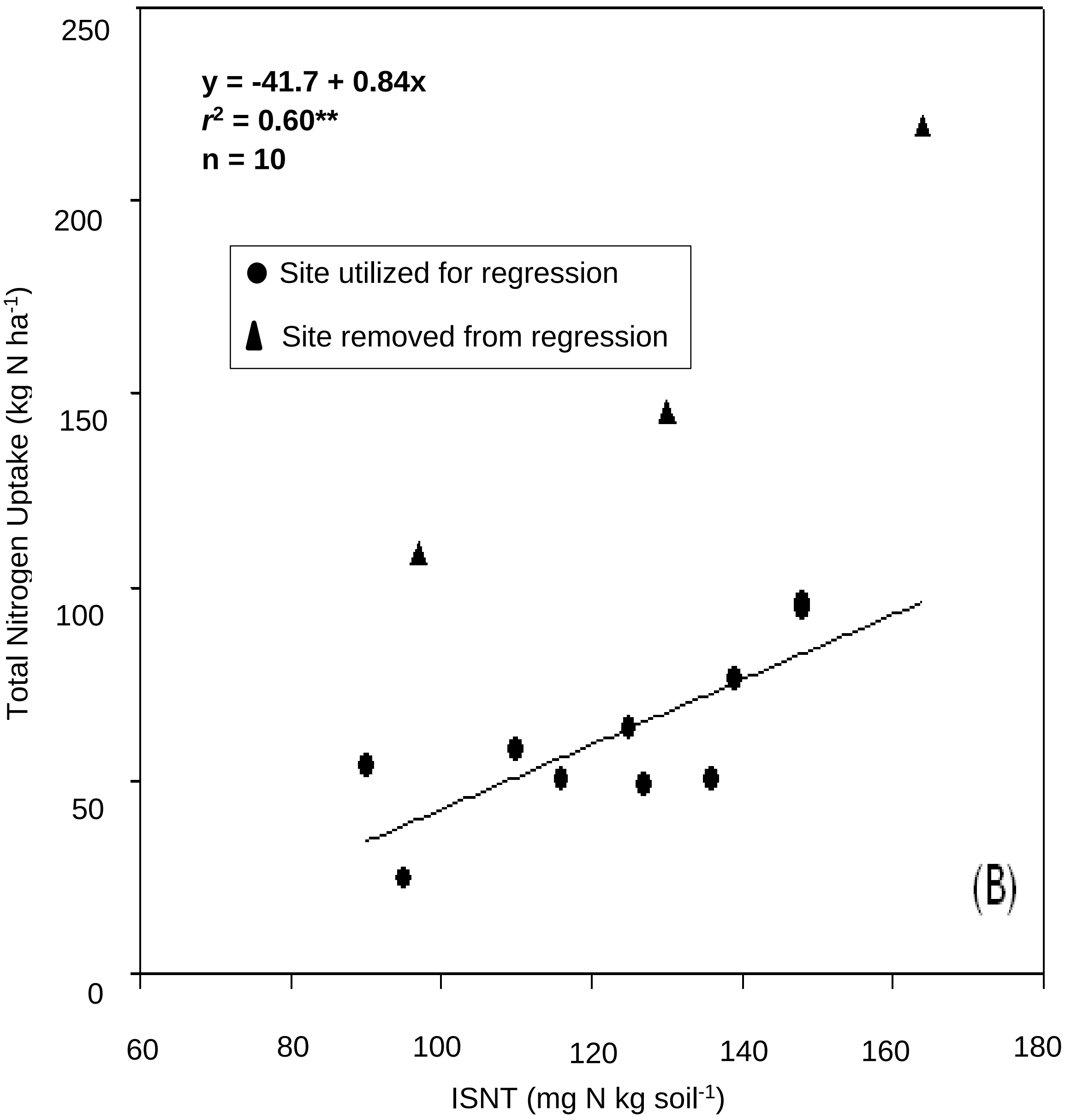

Figure 3.1B. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with regression equations developed using a soil sampling depth of 0- to 30-cm. ** indicates a significant linear regression at the 0.01 level of probability.

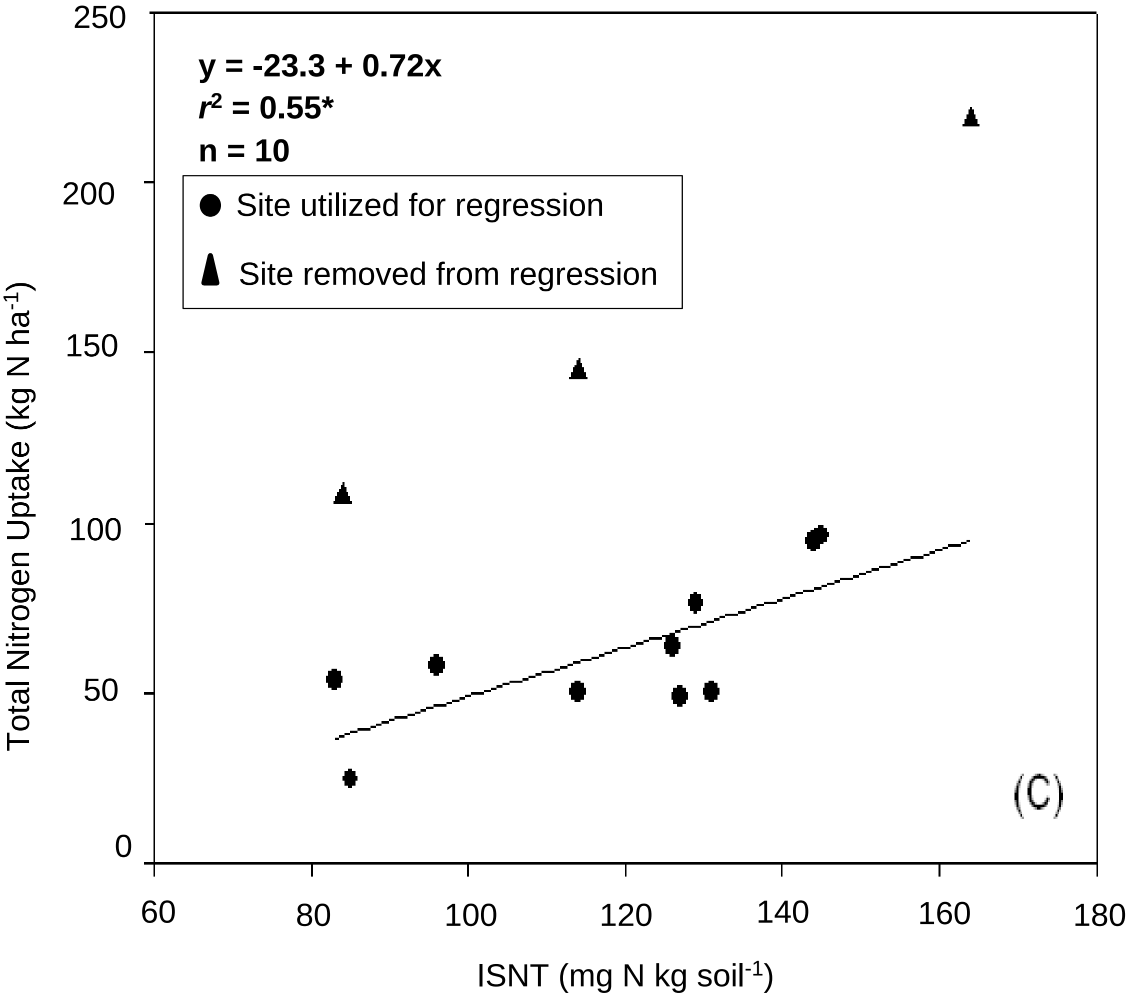

Figure 3.1C. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with the regression equation developed using a soil sampling depth of 0- to 45-cm. * indicates a significant linear regression at the 0.05 level of probability.

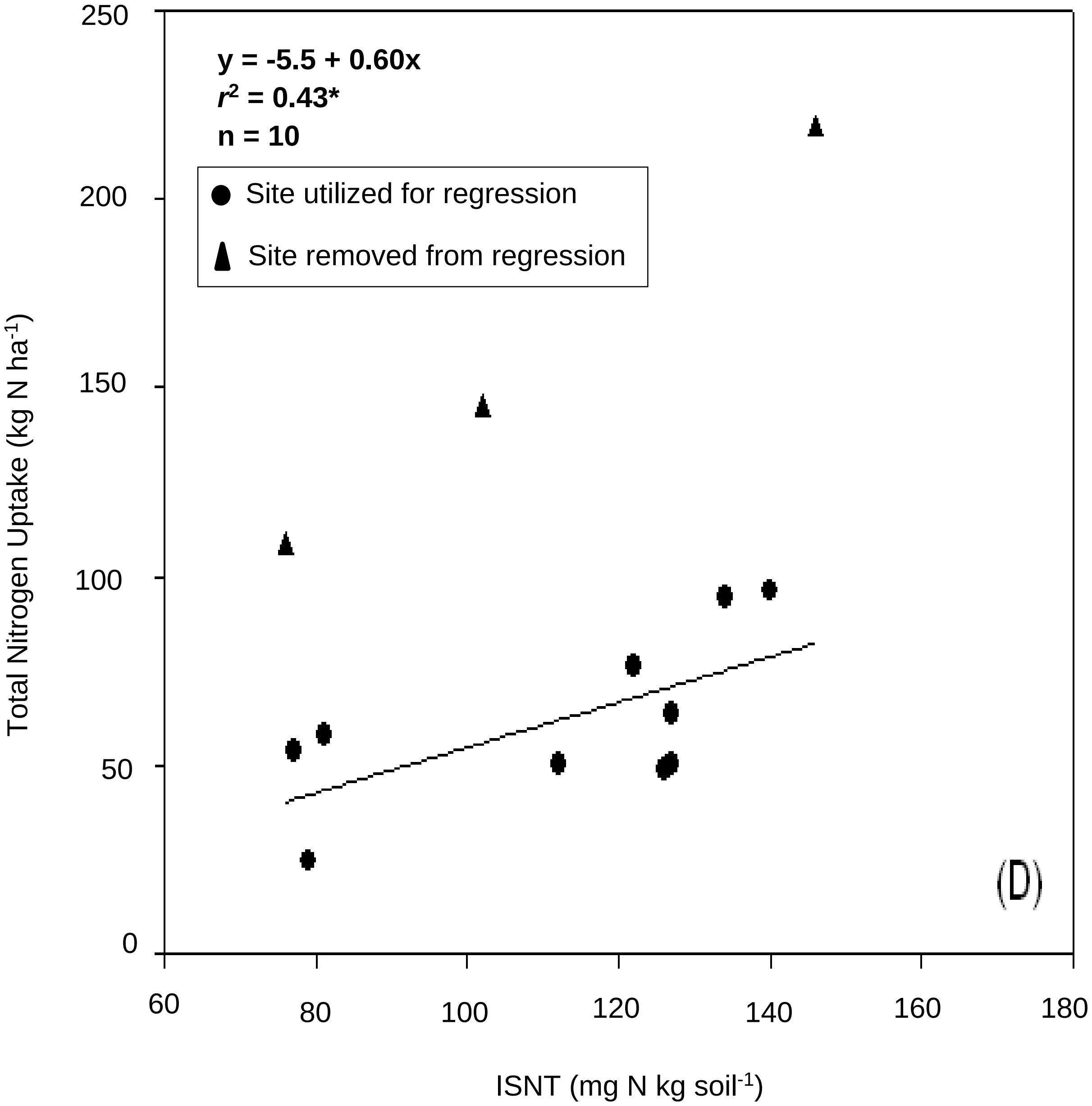

Figure 3.1D. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with the regression equation developed using a soil sampling depth of 0- to 60-cm. * indicates a significant linear regression at the 0.05 level of probability.

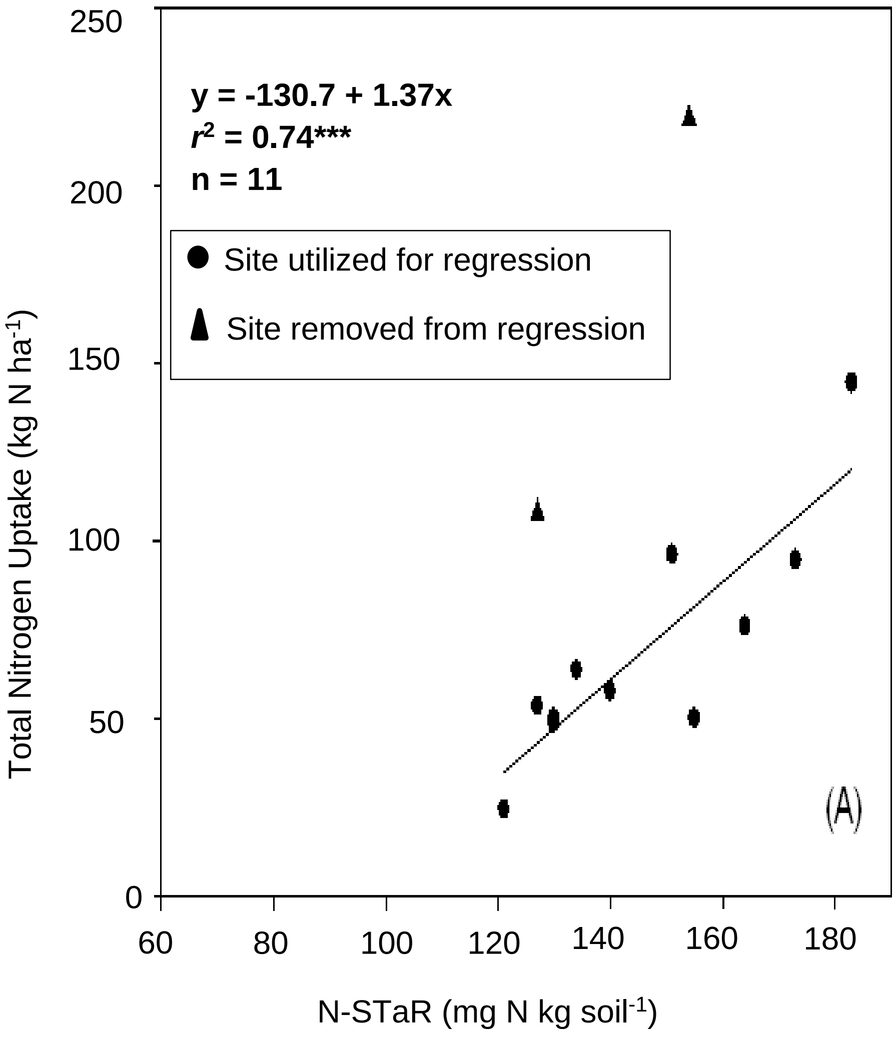

Figure 3.2A. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using a soil sampling depth of 0- to 15-cm. *** indicates a significant linear regression at the 0.001 level of probability.

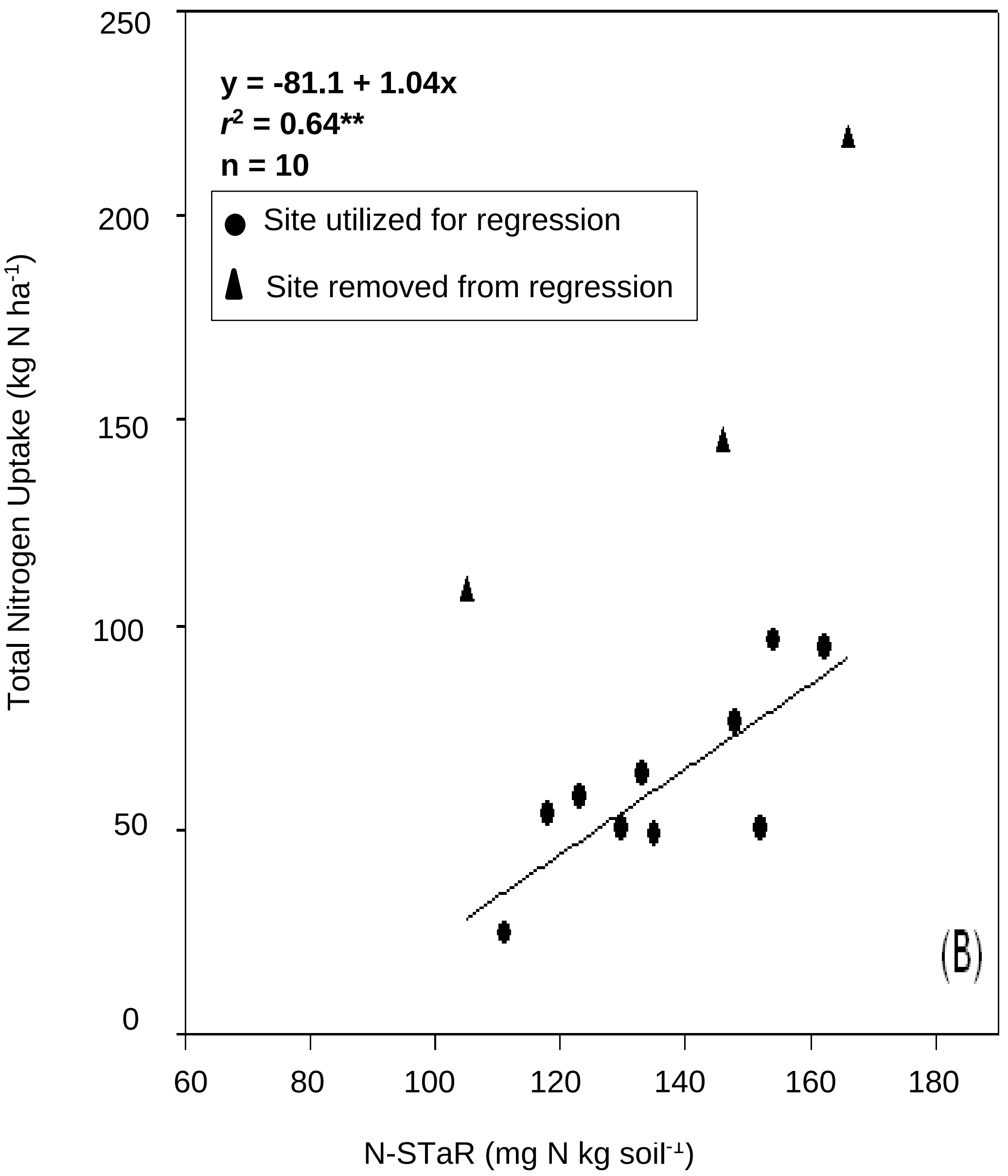

Figure 3.2B. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using a soil sampling depth of 0- to 30-cm. ** indicates a significant linear regression at the 0.01 level of probability.

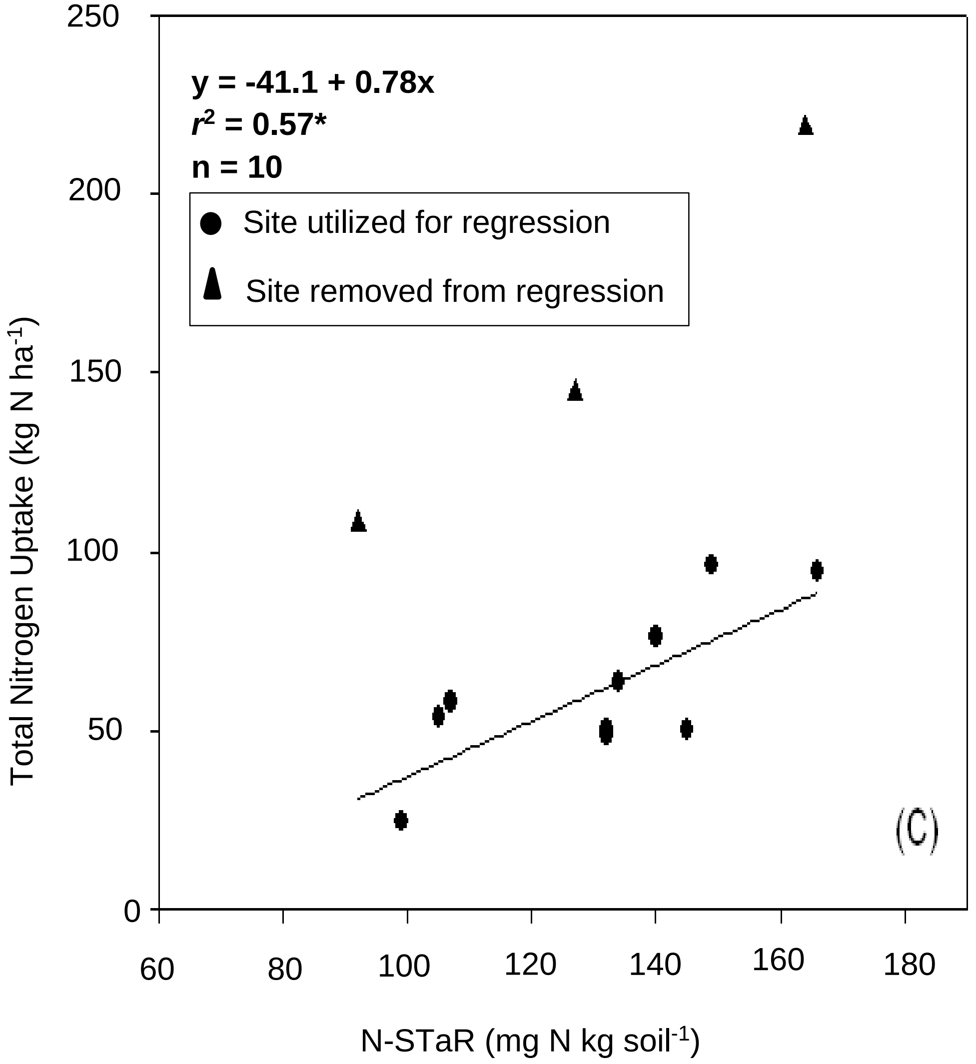

Figure 3.2C. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using a soil sampling depth of 0- to 45-cm. * indicates a significant linear regression at the 0.05 level of probability.

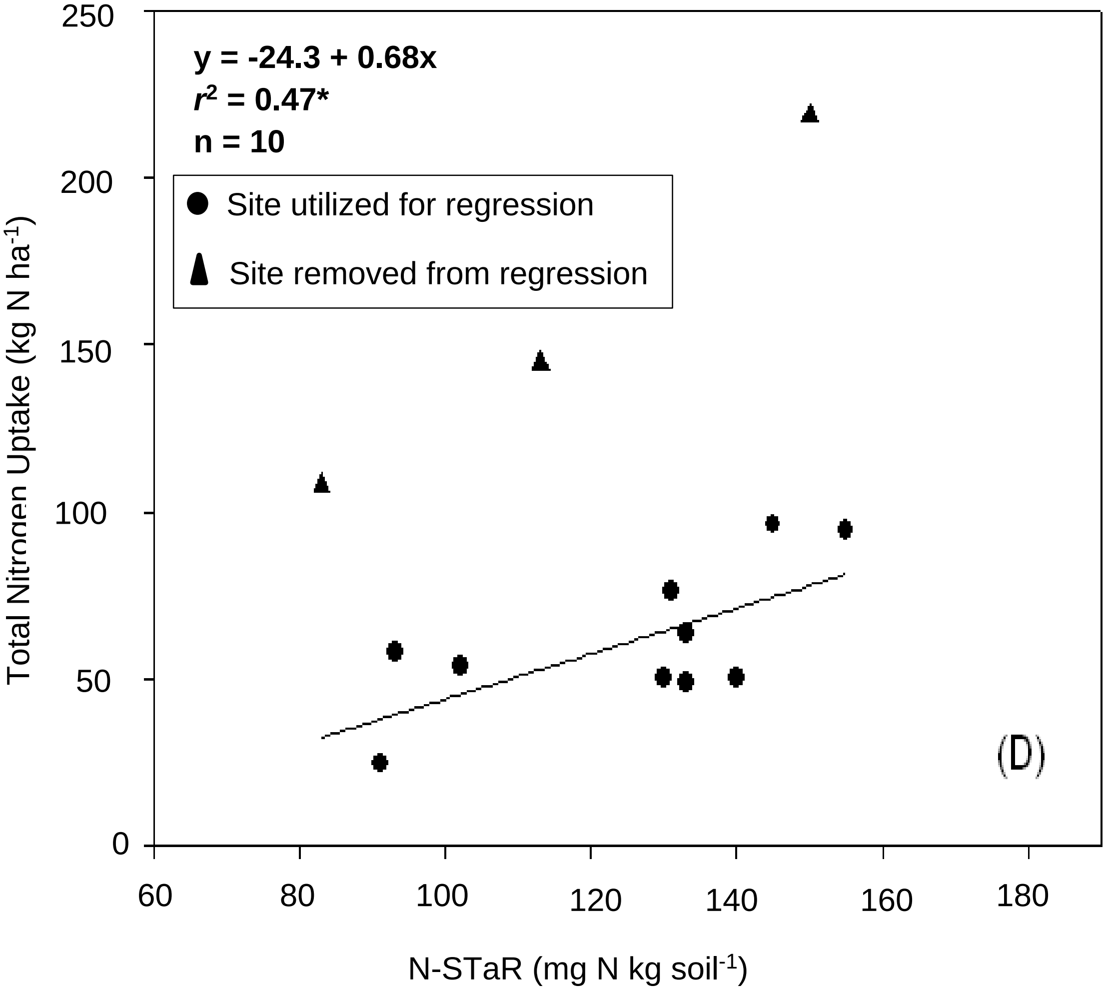

Figure 3.2D. Total nitrogen (N) uptake of rice receiving no fertilizer N regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using a soil sampling depth of 0- to 60-cm. * indicates a significant linear regression at the 0.05 level of probability.

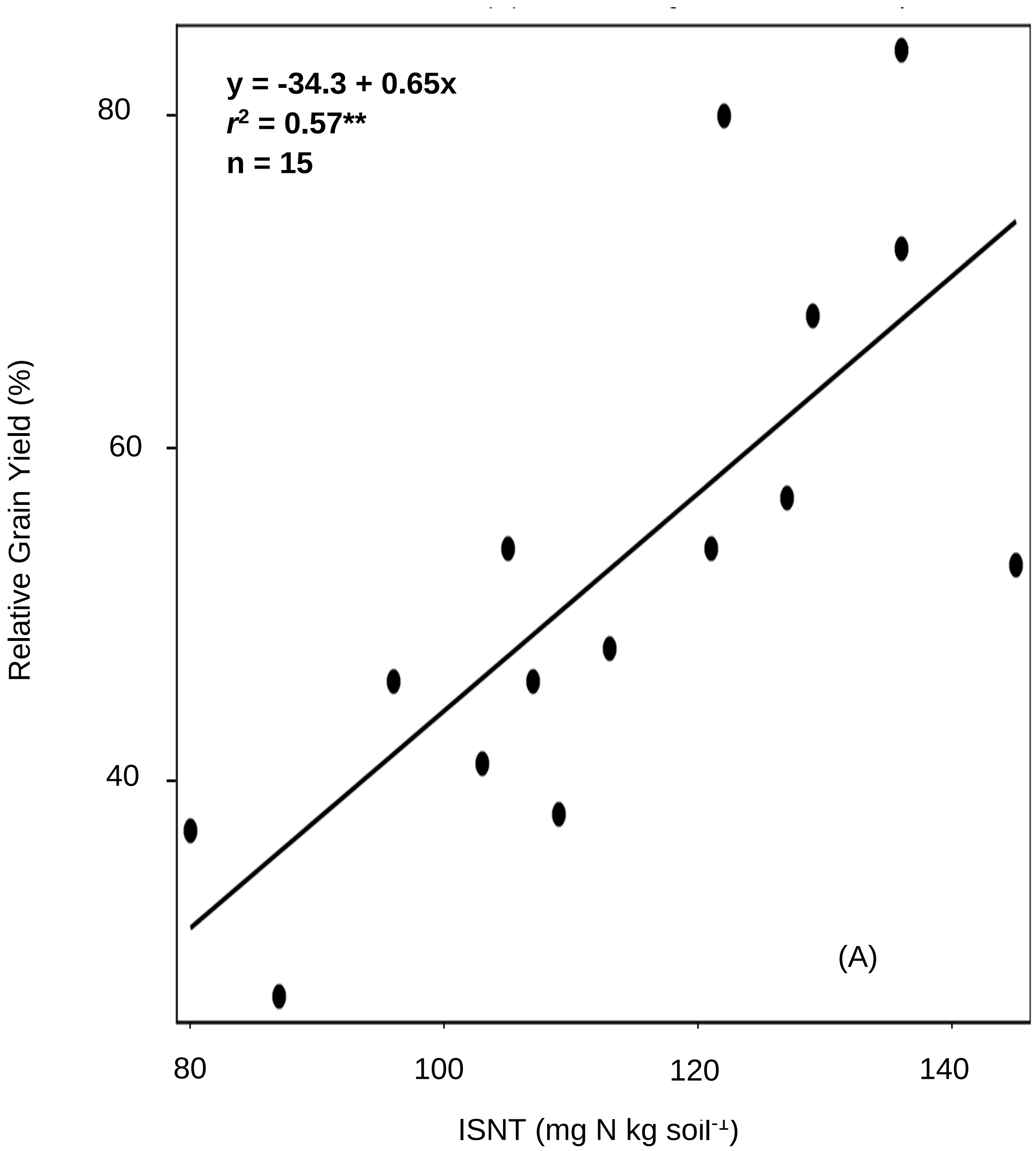

Figure 3.3A. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with the regression equation developed using the soil sampling depth of: 0- to 15-cm. ** indicates a significant linear regression at the 0.01 level of probability.

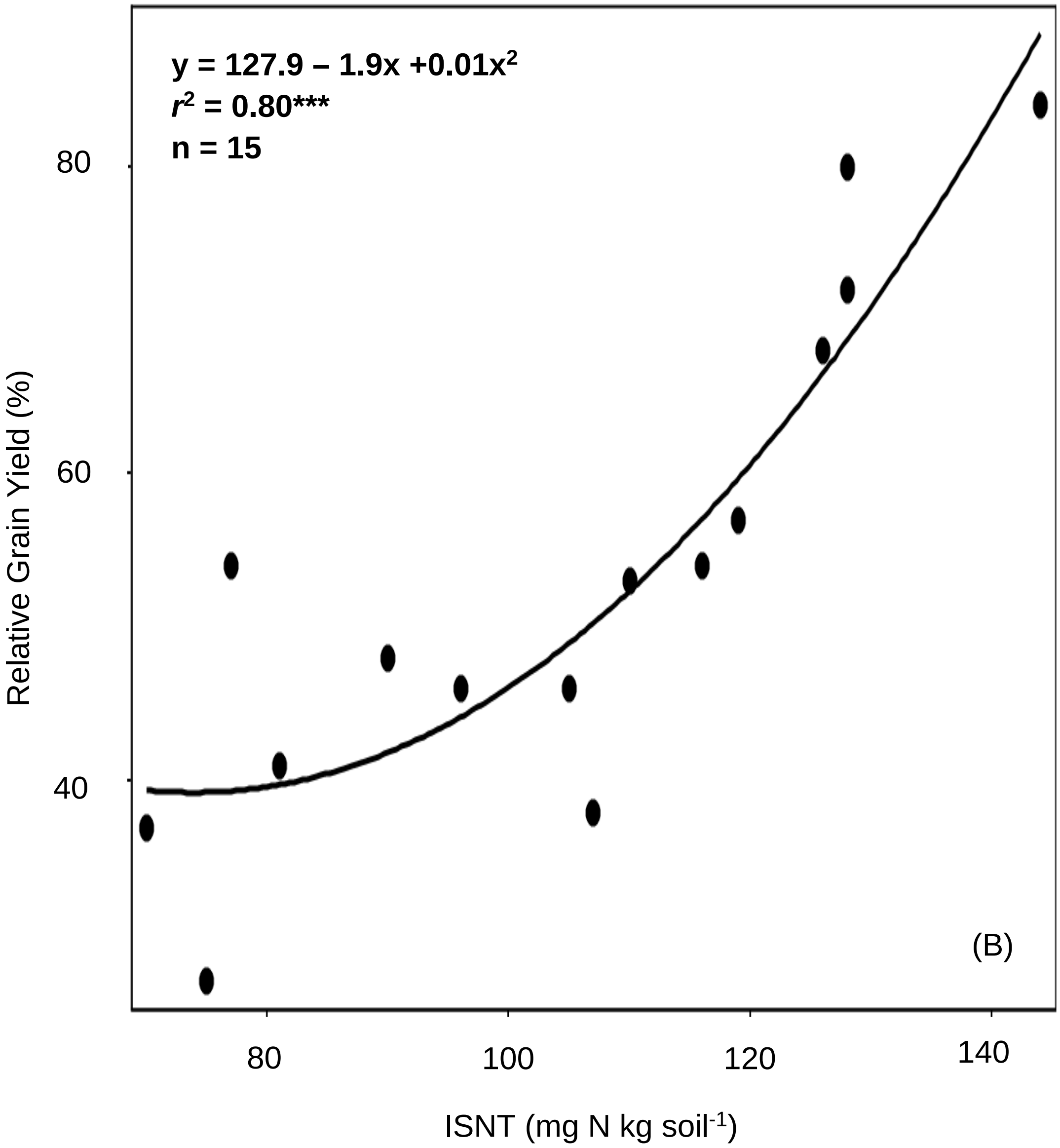

Figure 3.3B. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with the regression equation developed using the soil sampling depth of 0- to 30-cm. *** indicates a significant linear regression at the 0.001 level of probability.

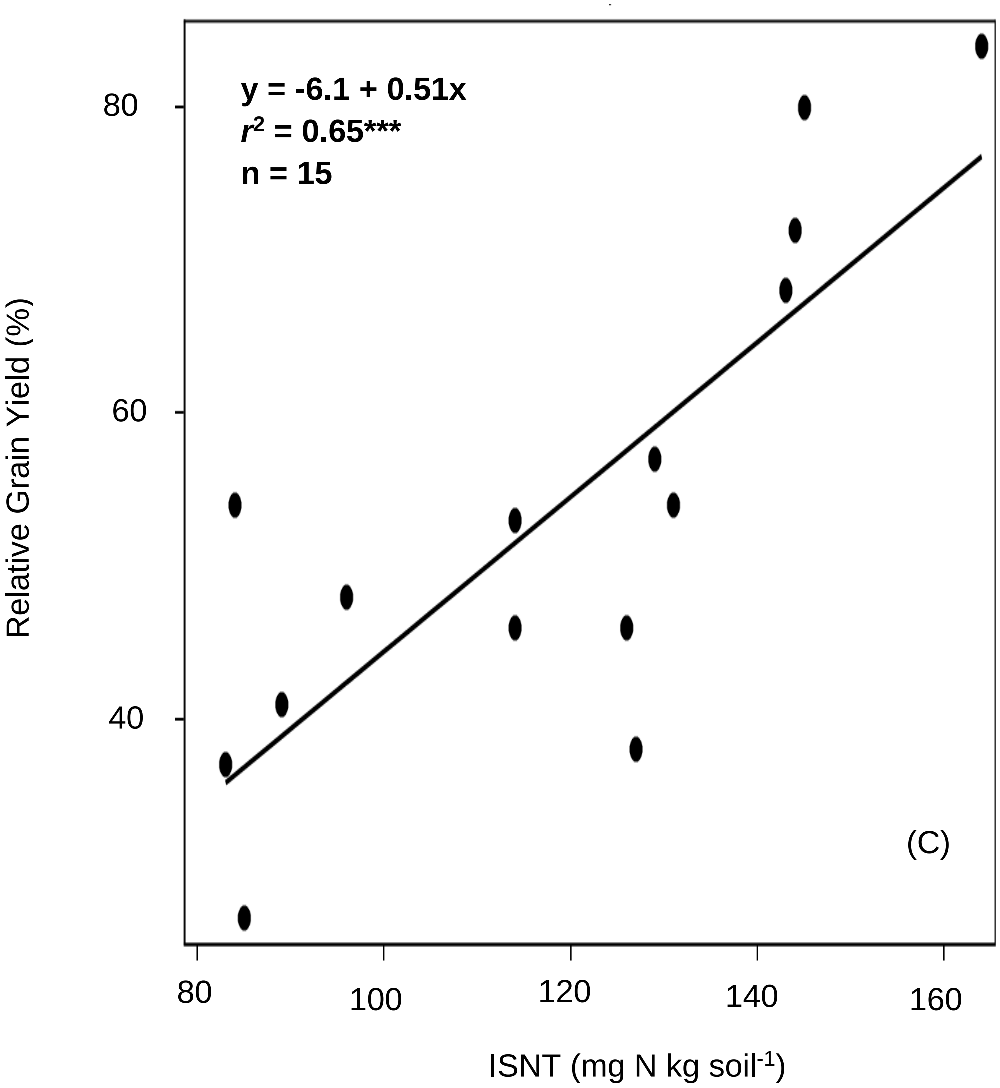

Figure 3.3C. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with the regression equation developed using the soil sampling depth of 0- to 45-cm. *** indicates a significant linear regression at the 0.001 level of probability.

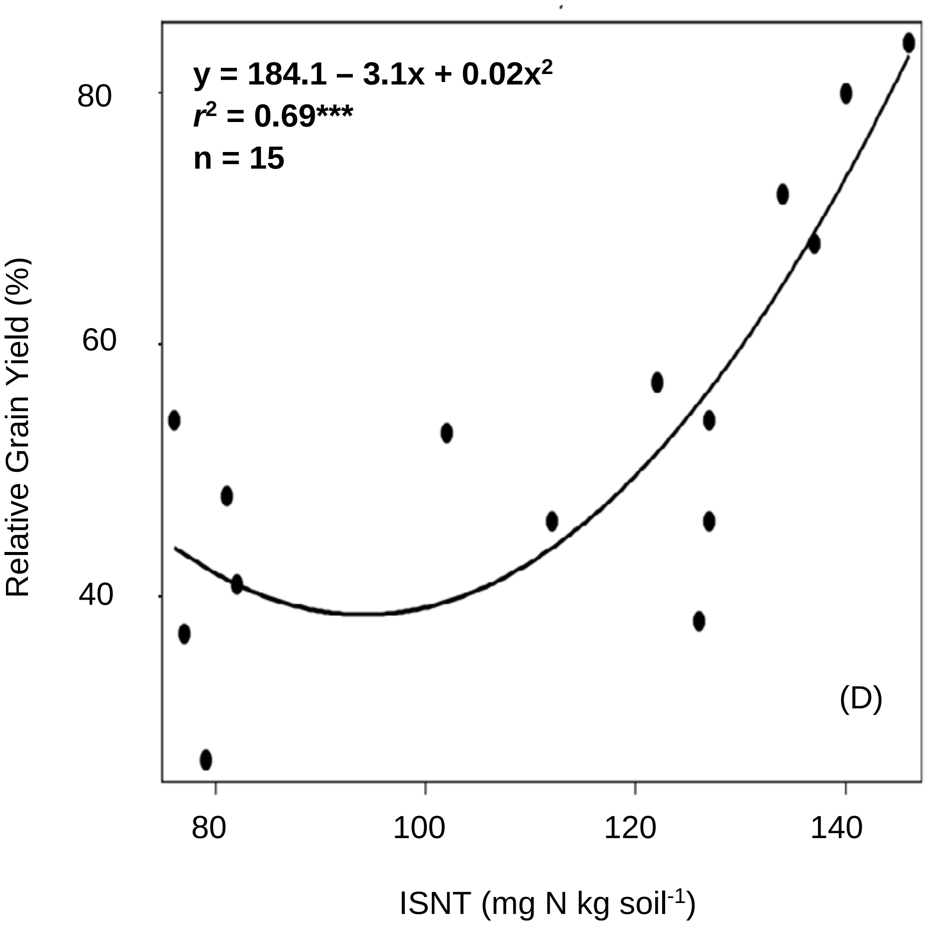

Figure 3.3D. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value with the regression equation developed using the soil sampling depth of 0- to 60-cm. *** indicates a significant linear regression at the 0.001 level of probability.

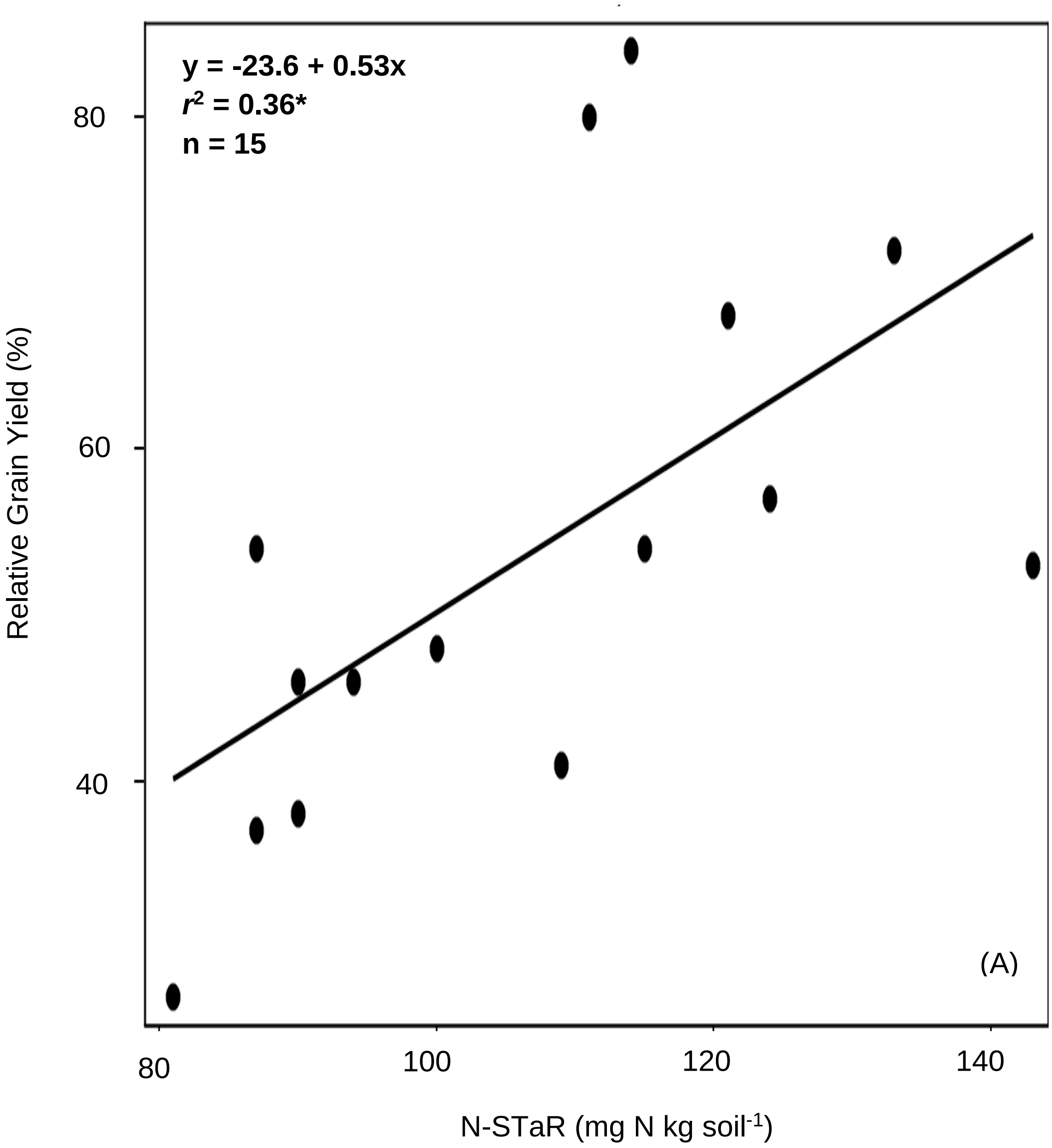

Figure 3.4A. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using the soil sampling depth of 0- to 15-cm. * indicates a significant linear regression at the 0.05 level of probability.

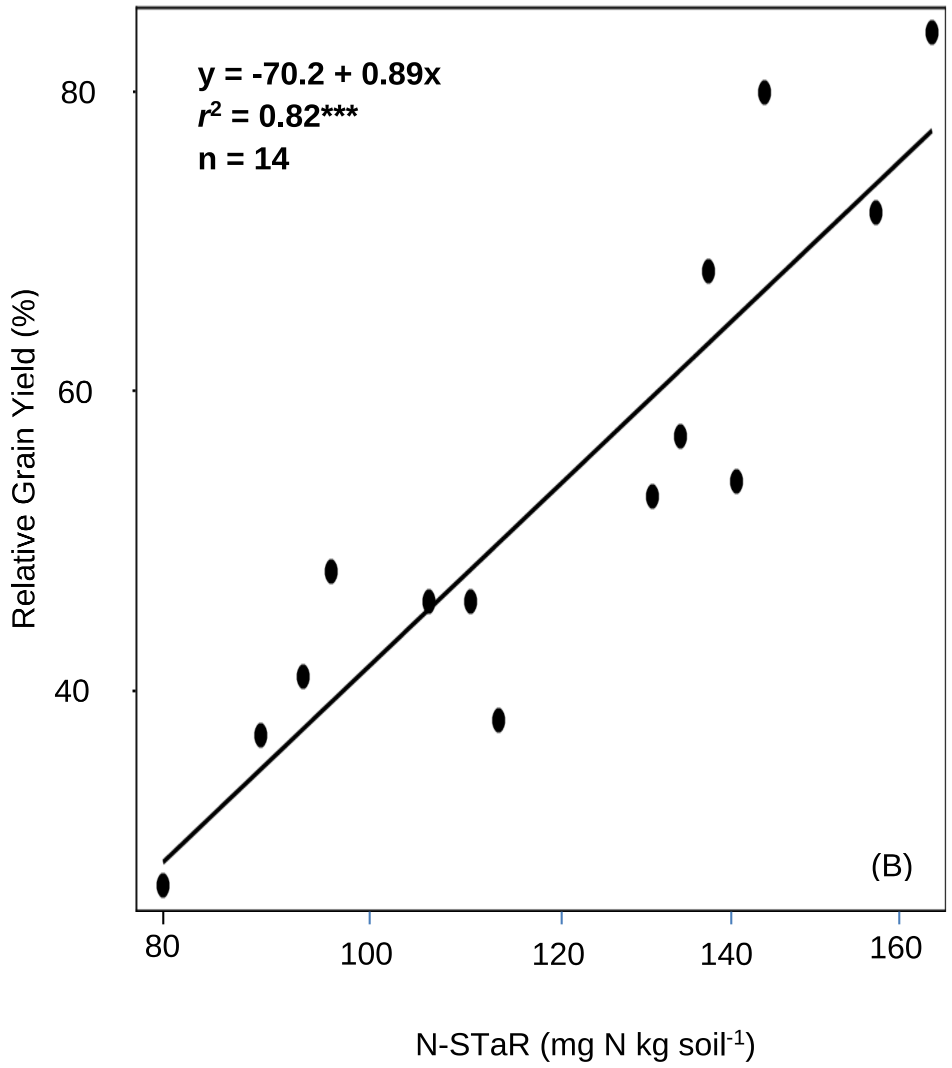

Figure 3.4B. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using the soil sampling depth of 0- to 30-cm. *** indicates a significant linear regression at the 0.001 level of probability.

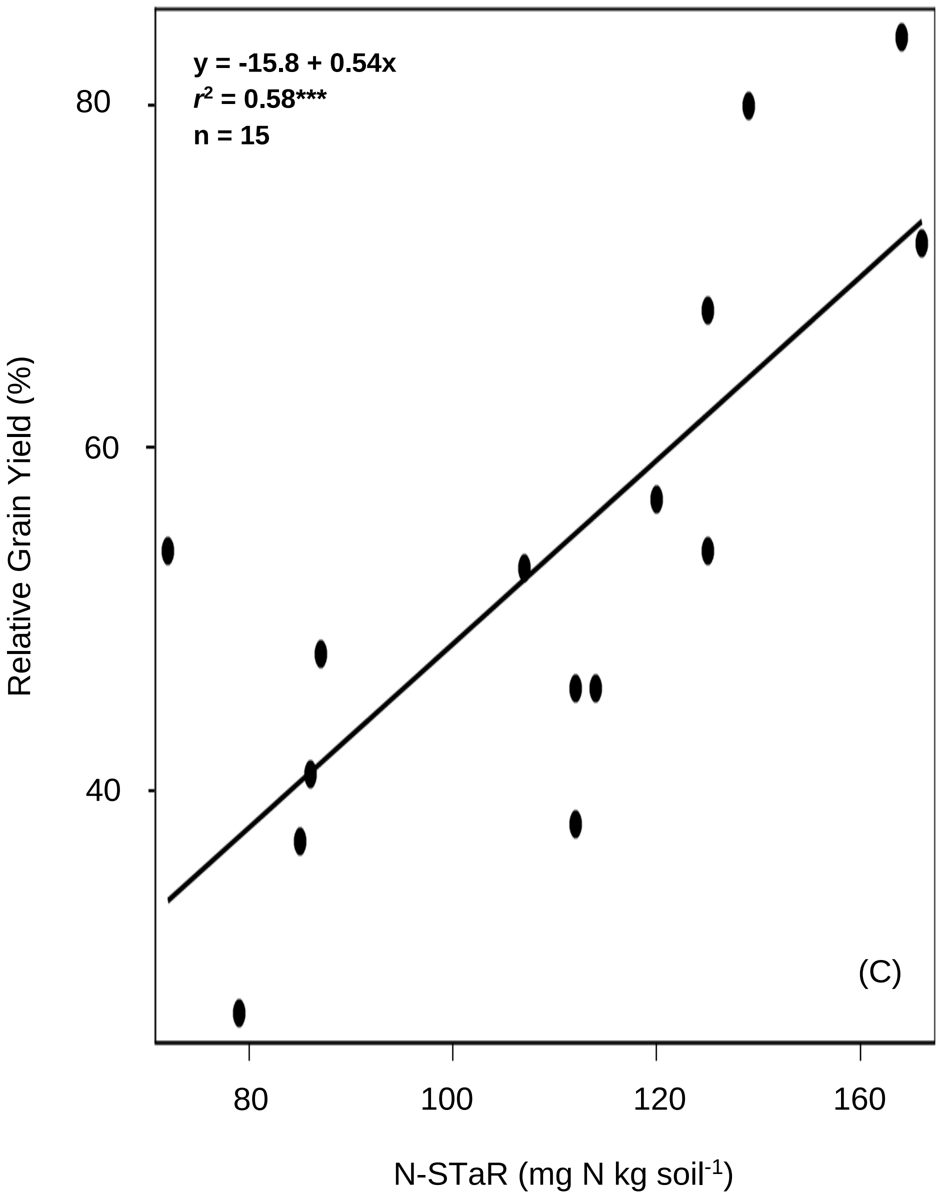

Figure 3.4C. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using the soil sampling depth of 0- to 45-cm. *** indicates a significant linear regression at the 0.001 level of probability.

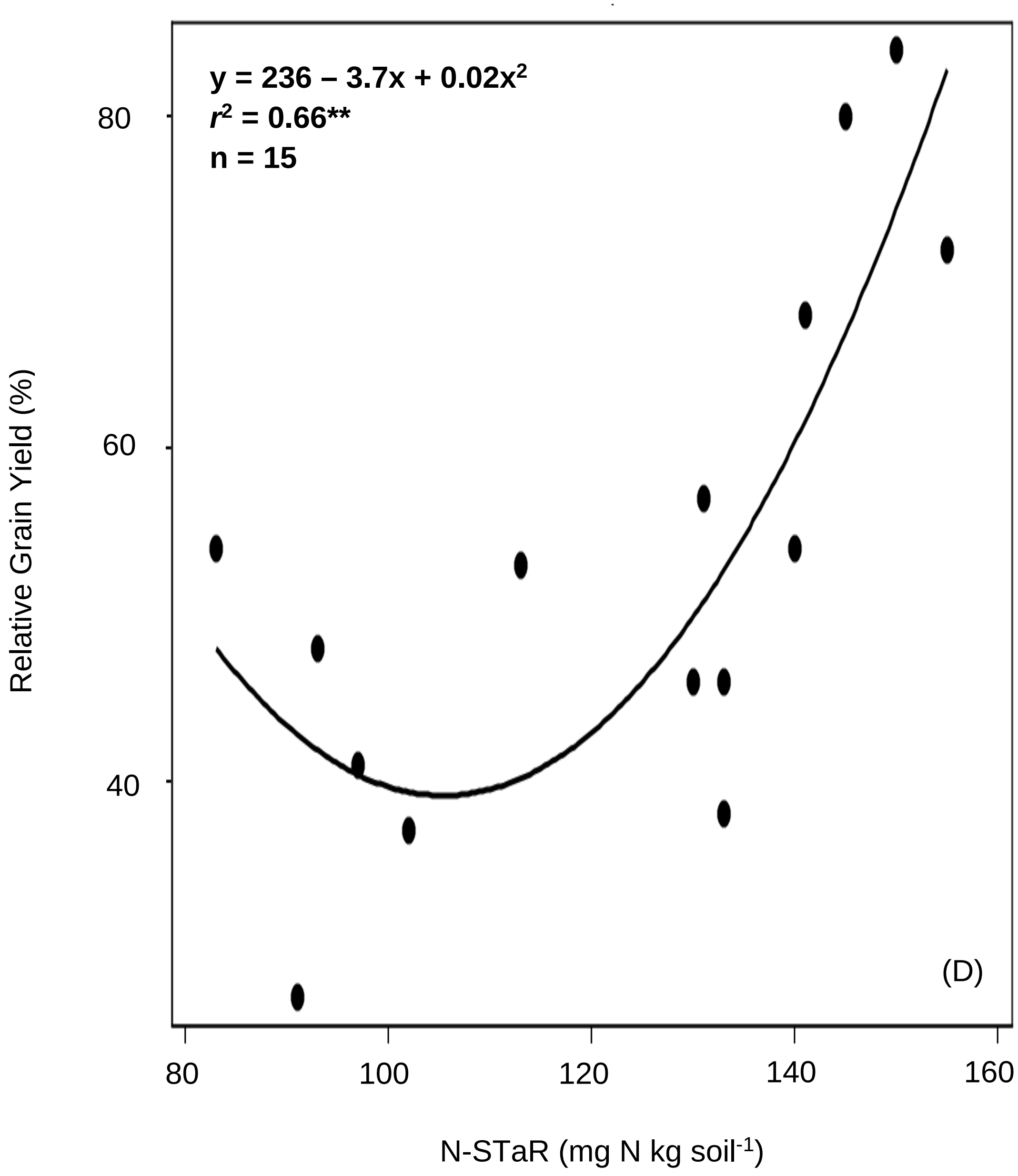

Figure 3.4D. Percent relative grain yield when no N was applied (% RGY_{0N}) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value with the regression equation developed using the soil sampling depth of 0- to 60-cm. ** indicates a significant linear regression at the 0.01 level of probability.

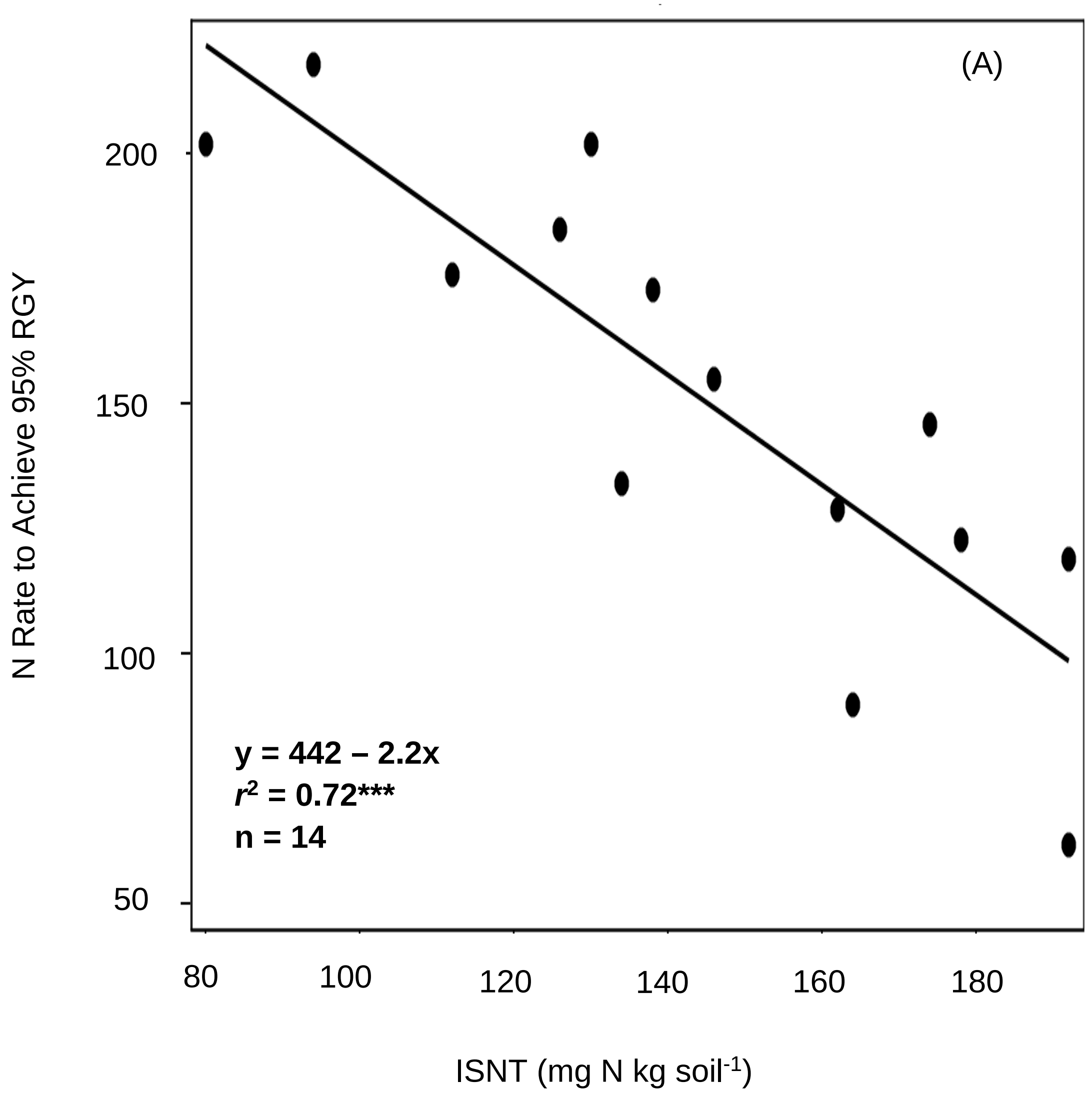

Figure 3.5A. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 15-cm. *** indicates a significant linear regression at the 0.001 level of probability.

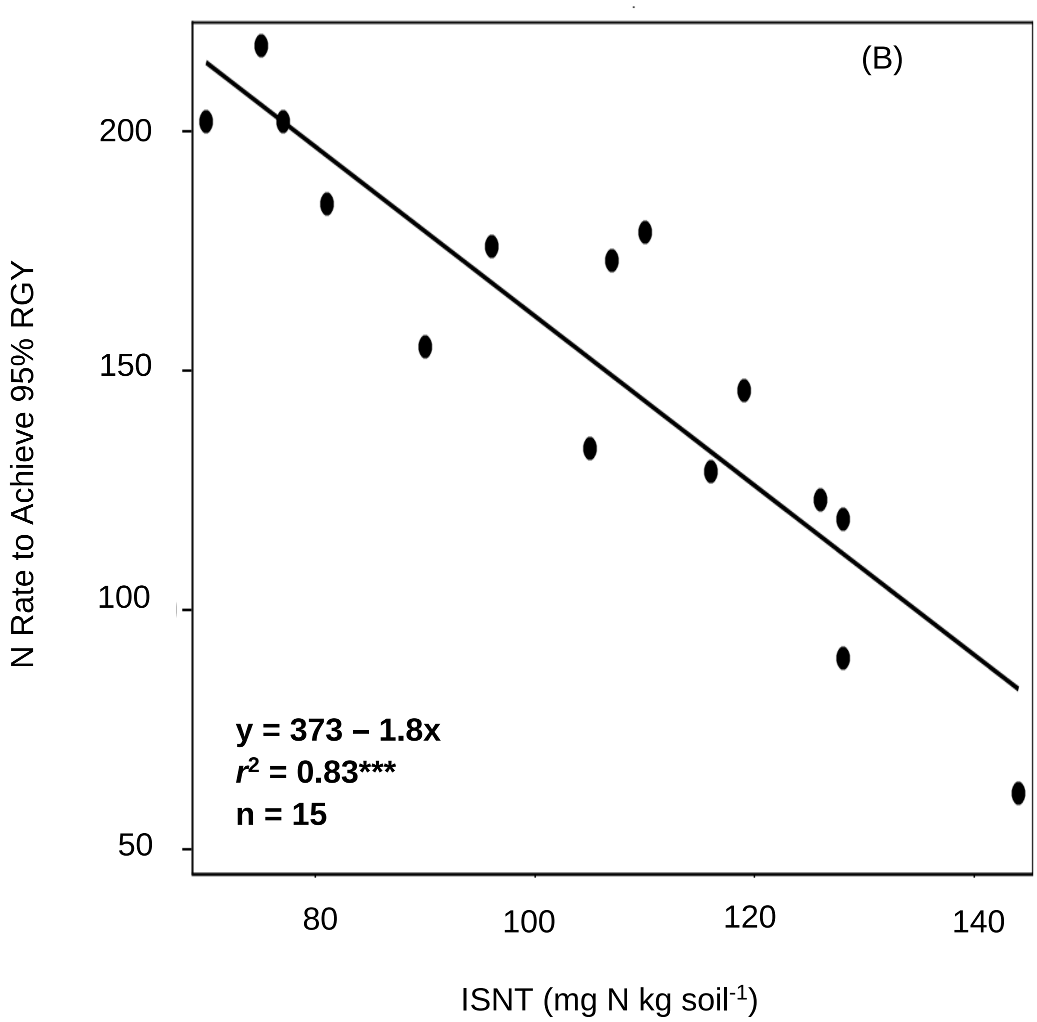

Figure 3.5B. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 30-cm. *** indicates a significant linear regression at the 0.001 level of probability.

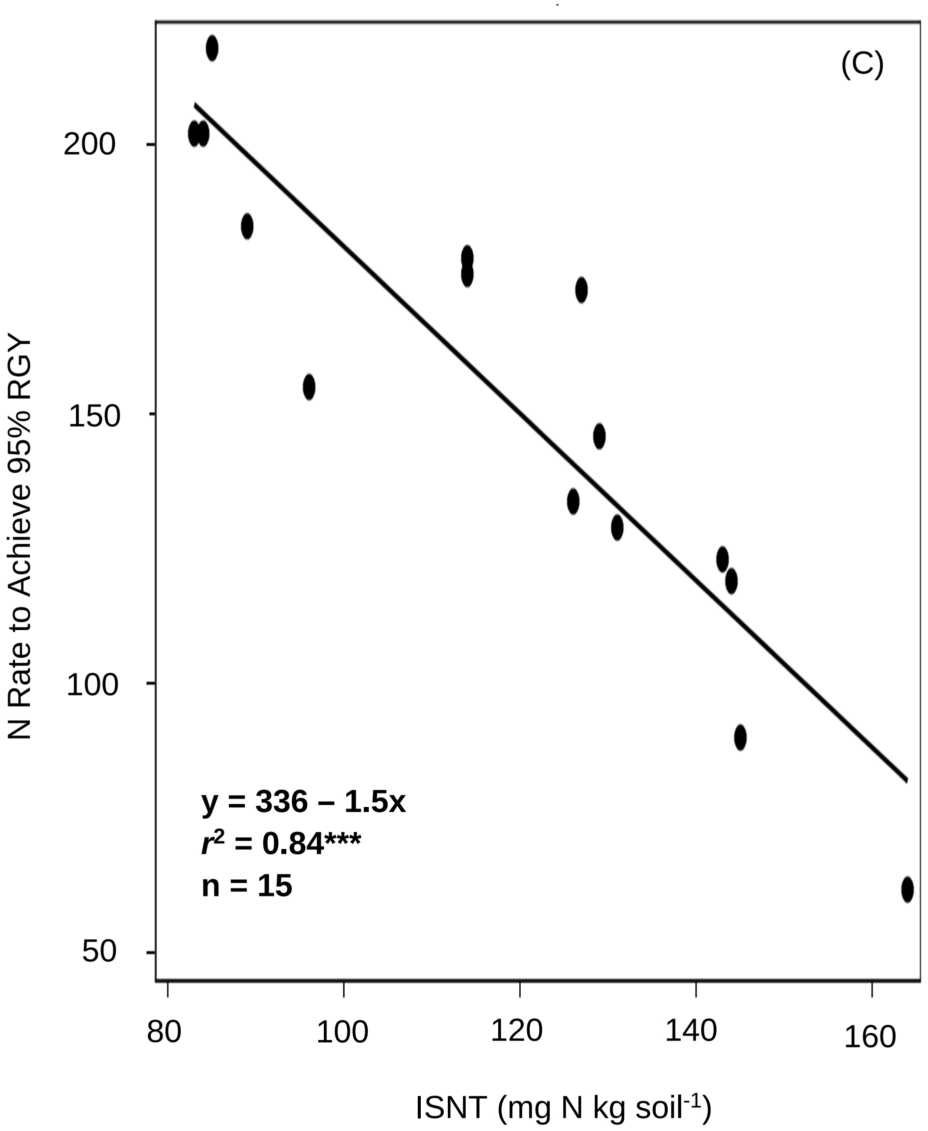

Figure 3.5C. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 45-cm. *** indicates a significant linear regression at the 0.001 level of probability.

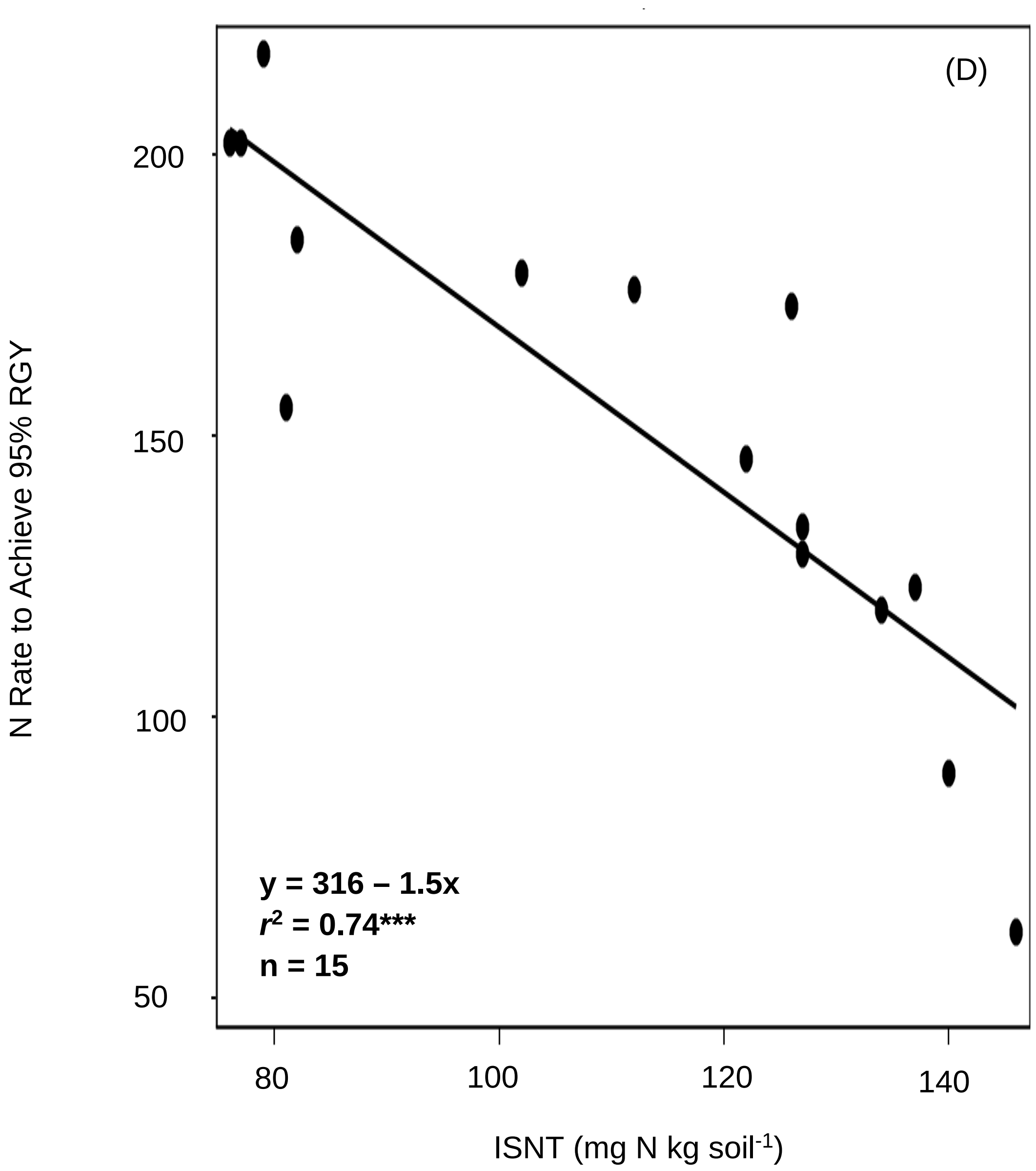

Figure 3.5D. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Illinois Soil Nitrogen Test (ISNT) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 60-cm. *** indicates a significant linear regression at the 0.001 level of probability.

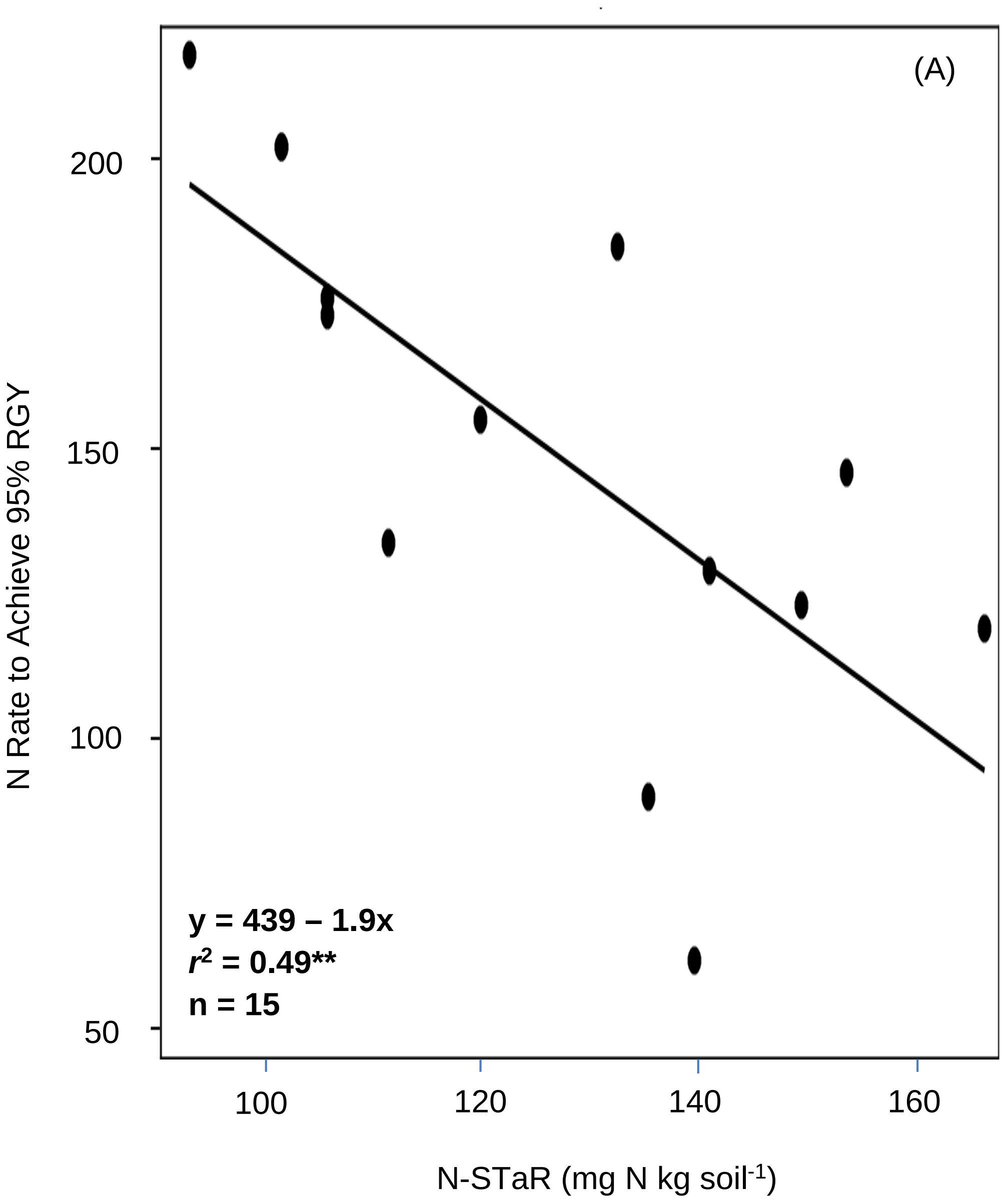

Figure 3.6A. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 15-cm. ** indicates a significant linear regression at the 0.01 level of probability.

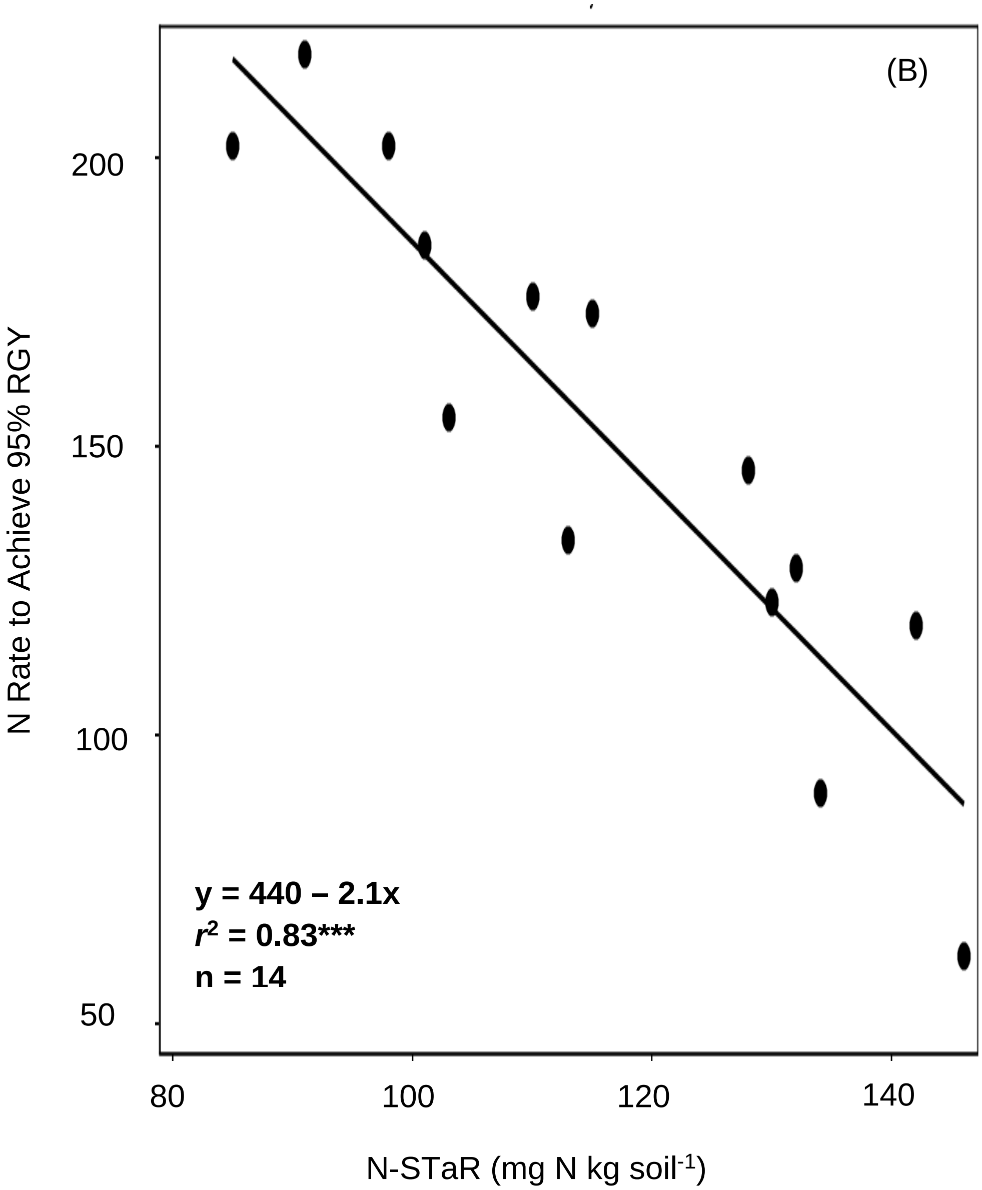

Figure 3.6B. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 30-cm. *** indicates a significant linear regression at the 0.001 level of probability.

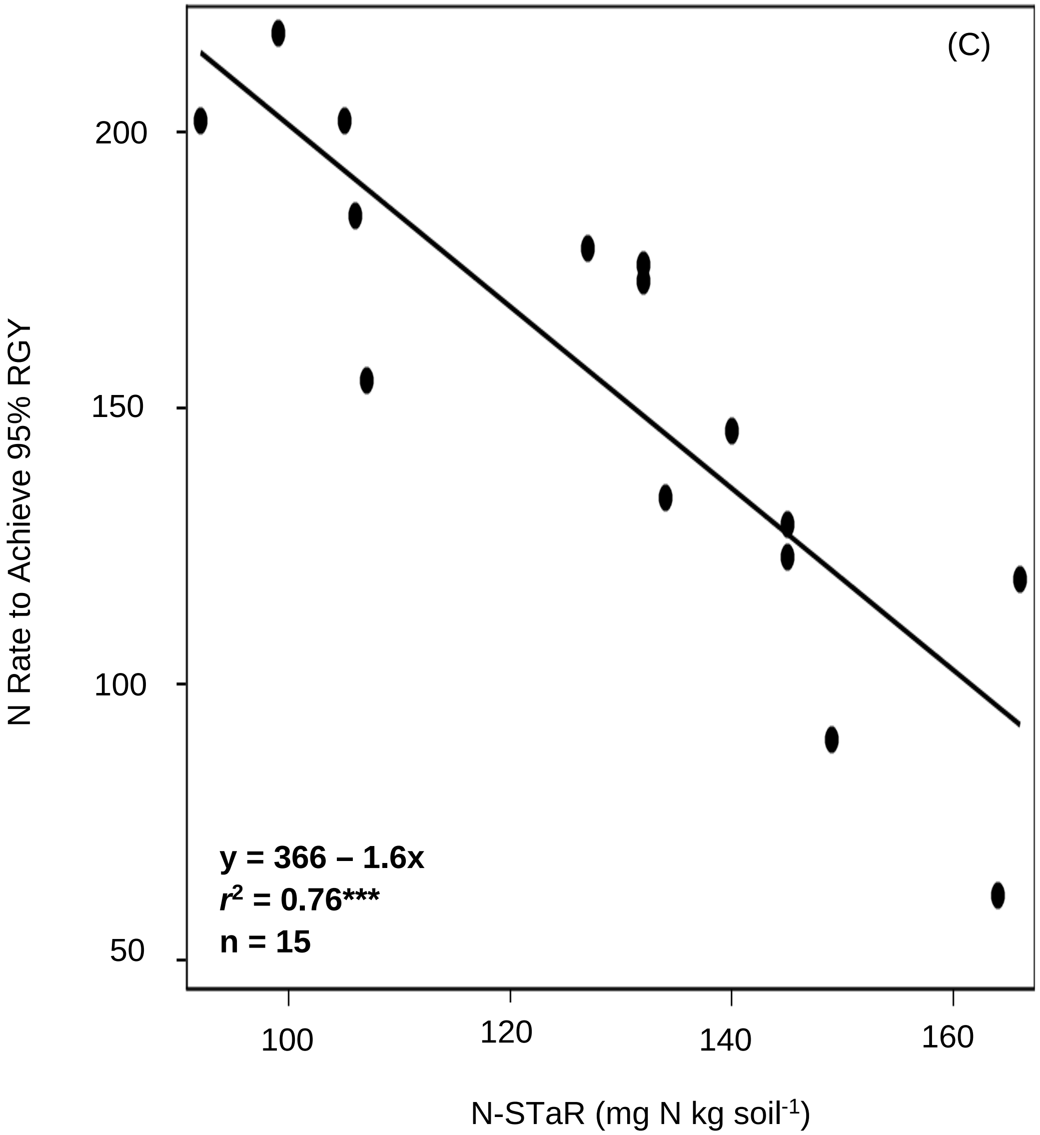

Figure 3.6C. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 45-cm. *** indicates a significant linear regression at the 0.001 level of probability.

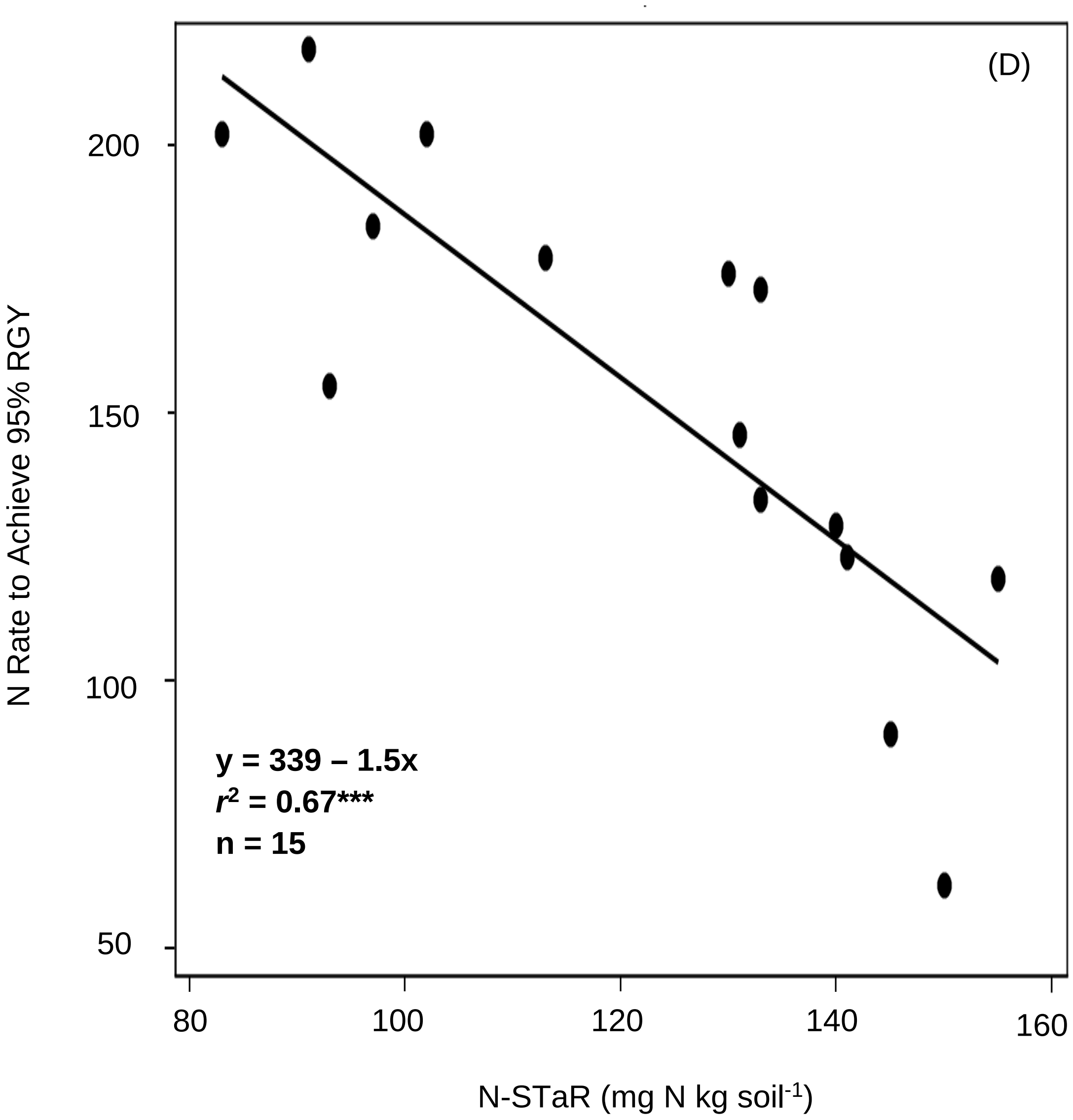

Figure 3.6D. Fertilizer N rate required to achieve 95% relative grain yield (95% RGY) regressed on the Nitrogen Soil Test for Rice (N-STaR) soil test value. Fertilizer N rate calibration curve was developed using a soil sampling depth of 0- to 60-cm. *** indicates a significant linear regression at the 0.001 level of probability.

CHAPTER FOUR

SEEDING DATE EFFECT ON RICE RESPONSE TO NITROGEN FERTILIZATION IN ARKANSAS

ABSTRACT

Rice (*Oryza sativa* L.) response to nitrogen (N) fertilization as influenced by seeding date has not been explored for drill-seeded, delayed flood rice in the mid-South USA. The objective was to characterize the influence of N fertilization on total N (TN) uptake and aboveground dry matter at 50% heading as well as grain yield in response to seeding date. Fertilizer N rates ranged from 0 to 180 kg N ha^{-1} and were applied to rice seeded in April, May, and June at two locations in Arkansas in 2010 and 2012. Total N uptake exhibited a curvilinear (i.e., quadratic) response to fertilizer N rate in 2010 and a linear response to fertilizer N rate in 2012. Aboveground dry matter and rice grain yield exhibited a curvilinear response to N rate in 2010 and 2012. The yield maximizing fertilizer N rate for rice seeded in April ranged from 156 to 165 kg N ha^{-1}, while the yield maximizing fertilizer N rate for rice seeded in May and June ranged from 123 to 170 kg N ha^{-1} in 2010. In 2012, the yield maximizing fertilizer N rate for rice seeded in April ranged from 55 to 137 kg N ha^{-1}, while rice seeded in May and June exhibited a range from 60 to 161 kg N ha^{-1}. Results provided insufficient evidence needed to identify the influence of seeding date on the fertilizer N requirement of rice and for the grain yield advantage of early-seeded rice compared to late-seeded rice. Research is needed to determine if similar or different fertilizer N rate recommendations are required for rice grown after the optimum seeding date period in Arkansas.

INTRODUCTION

Optimal seeding dates provide an indication as to when factors such as climate and soil temperature are suitable for adequate seed germination. Timely planting is a primary annual objective although untimely precipitation can substantially delay planting. When planting is delayed with respect to the optimal seeding date period yield losses have been documented for cereal and leguminous crops. A 15% reduction of corn (*Zea Mays* L.) yield occurred 4 wk following the optimal seeding date cut-off in Minnesota (Van Roekel and Coulter, 2011). Wheat (*Triticum aestivum* L.) yield in Louisiana exhibited an average decrease of 26% when planting was delayed 4 wk (Shah et al., 1994). Rice yield was reduced by 13% as planting was delayed 6 wk in Arkansas despite receiving the same N rate (134 kg N ha^{-1}) across seeding dates (Frizzell et al., 2010). A yield reduction of 27% occurred in Kentucky when soybean [*Glycine max* (L.) Merr.] planting was delayed 8 wk beyond the optimal seeding date period (Lee and Herbek, 2011).

The identification of yield components which are influenced by seeding date could aid in the selection of agronomic practices that might limit or compensate for the delayed seeding date yield penalty. Subedi et al. (2007) compared yield components of early- and late-seeded spring wheat and reported the number of spikes per square meter as well as harvest index declined from April to May in Canada. Reduced kernel weight of corn grown in southern Minnesota was a consequence of late seeding but the influence of seeding date on grain yield response was independent of planting density (Van Roekel and Coulter, 2011). Soybean seeds per square meter were reduced as planting was delayed from April to May in Iowa and was shown to be independent of seeding rate (De Bruin and Pederson, 2008). Jones and Snyder (1987) found that tall and semi-dwarf rice cultivars exhibited a decline in the number of filled grain per panicle in Florida and that changes in row spacing failed to produce a significant yield response for late-seeded rice. Jones and Snyder (1987) subsequently suggested that as seeding was delayed in Florida suboptimal growing conditions (i.e., lower solar radiation, higher air temperature) later in the growing season could have limited the yield response of rice to alterations in row spacing. Manipulating agronomic practices such as seeding rate and row spacing has yet to be successful with regard to increasing or maintaining yield when seeding is delayed beyond the optimum seeding period.

The optimum seeding period changes with geographic region. The end of the optimum seeding period for rice is 20 May, 15 May, and 10 May in southern, central, and northern Arkansas, respectively (Wilson et al., 2013). Planting progress surveys indicated that from 2008 to 2012 an average of 84% of the total rice hectarage in Arkansas had been seeded prior to 20 May with 100% of the rice hectarage seeded prior to 20 May in 2012, but then decreased to 75% in 2013 (National Agricultural Statistics Service, 2013). When seeding is delayed beyond the optimum seeding period it may be necessary to select rice cultivars on the basis of yield stability (Blanche and Linscombe, 2009) or time from seedling emergence to maturity. However, additional indicators of cultivar performance across a wide range of seeding dates may be needed because the selection of short-season or very-short season rice cultivars may not necessarily be the best option when seeding is delayed beyond the optimum seeding period (Norman et al., 1999; Wilson et al., 2013).

The prediction of rice growth stages based on growing degree-days (GDD) has been practiced since the 1970s (Hardke et al., 2013) and may aid cultivar selection by identifying the impact of seeding date on rice growth in Arkansas. The vegetative growth phase (i.e., seedling emergence to panicle initiation) of early-seeded rice requires a greater number of calendar days to achieve a similar number of GDD as late-seeded rice (Frizzell et al., 2010). While the length of the vegetative growth phase is influenced by seeding date, biomass accumulation during reproductive growth is related to grains per square meter (Kropff et al., 1994). Therefore, changes in the length of the reproductive growth phase may have a substantial impact on dry matter accumulation and rice yield. Ying et al. (1998) concluded that increasing the grain yield of modern rice cultivars grown in tropical or sub-tropical environments may depend on increasing biomass accumulation during the growing season.

The influence of seeding date on rice yield was first investigated during the1930s for rice cultivars grown in Arkansas (Adair, 1940). Since then, the results of seeding date studies have led to the identification of optimal seeding dates for modern rice cultivars (Slaton et al., 2003; Linscombe et al., 2004). When rice is planted outside of the optimum seeding period, it becomes even more important to utilize best management practices, including efficient fertilizer management, to optimize rice grain yield. Stand establishment is a concern for rice seeded earlier than the optimum period and an additional 34 to 67 kg N ha^{-1} is necessary to achieve maximum grain yields for low plant populations (Counce et al., 1992). When seeding is delayed, early season monitoring and management interventions may help prevent or correct inadequate plant growth prior to the end of the growing season. However, measurement of the crop N status during vegetative growth may inaccurately predict rice grain yield (Ntamatungiro et al., 1999). Therefore, evaluating the fertilizer N requirement of late-seeded rice early in the growing season would be an ineffective fertilizer management strategy.

Evaluating the influence of seeding date on rice response to N fertilization may enable fertilizer N sources to be managed more effectively after the optimal seeding date period in Arkansas. However, current information regarding the influence of seeding date and N fertilization on rice yield is limited and the seeding date studies that have been conducted (Ghosh et al., 2004; Soleymani and Shahrajabian, 2011) do not adequately represent the cultivars or cultural practices that are currently common to mid-South rice production. The objective of this study was to characterize the influence of N fertilization on TN uptake and aboveground dry matter at 50% heading as well as grain yield of the rice cultivars Wells, Clearfield 151 (CL151), and Clearfield hybrid 745 (CLXL745) in response to seeding date in Arkansas.

MATERIALS AND METHODS

Location Description and Cultivar Characteristics

Seeding date studies were conducted at the University of Arkansas Division of Agriculture Pine Tree Research Station (PTRS) near Colt, AR (35°07'52.8" N, 90°48'39.8" W) in 2010 (PT2010) and 2012 (PT2012) as well as at the University of Arkansas Division of Agriculture Rice Research and Extension Center (RREC) near Stuttgart, AR (34°30'01.0" N, 91°33'22.9" W) in 2010 (RR2010) and 2012 (RR2012). The RREC is located in eastern Arkansas within a region known as the Grand Prairie (Brye and Pirani, 2005) identified as a major land resource area called the Southern Mississippi Valley Loess (i.e., MLRA 134; USDA-NRCS, 2006). The soils at RREC historically supported native prairie vegetation while soils at PTRS historically supported forest vegetation prior to agricultural production. The seeding date studies at RREC were conducted on a Dewitt silt-loam (fine, smectitic, thermic Typic Albaqualfs) soil and at PTRS on a Calloway silt-loam (fine-silty, mixed, active, thermic Aquic Fraglossudalfs) soil in fields that had been in a rice–soybean rotation for at least the past 20 yr. Soil series information is reported using the taxonomic classification system of the Natural Resources Conservation Service (Soil Survey Staff, 2013).

The three rice cultivars included in this study were Wells (Moldenhauer et al., 2007), CLXL745 (RiceTec, Inc., Houston, TX), and CL151 (BASF, Ludwigshafen, Germany). Wells is a short stature, long-grain, early-season rice cultivar with a height of 102-cm and approximately 85 d after planting 50% of the panicles will have exerted from the sheath (50% heading; Hardke et al., 2012). Clearfield 151 (Blanche et al., 2011) is a semidwarf, long-grain, very early-season rice cultivar with a height of 99-cm, achieves 50% heading in 84 d and exhibits resistance to the acetolactase synthase (ALS)-inhibiting herbicide imazethapyr (2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-imidazol-2-yl]-5-ethyl-3-pyridinecarboxylic acid; Hardke et al., 2012). Clearfield 745 is a hybrid, long-grain, very early-season rice cultivar with a height of 109-cm, achieves 50% heading in approximately 79 d and also exhibits resistance to imazethapyr (Hardke et al., 2012). These three cultivars where chosen for the study because they represented the dominant types (i.e., semidwarf, short stature, Clearfield, hybrid) of long-grain rice grown in Arkansas and were either grown on the highest percentage of hectarage prior to the initiation of this study or were projected to be the predominant planted cultivars in the coming years (Wilson et al., 2010a). Currently, 168 kg N ha^{-1} is the fertilizer N rate recommendation for Wells and CLXL745 grown following soybean on silt loam soils, while 134 kg N ha^{-1} is the recommended fertilizer N rate for CL151 grown on silt loam soils following soybean in the crop rotation (Roberts et al., 2013). Grain yields, averaged across the 2010 to 2012 growing seasons, obtained from the Arkansas Rice Performance Trial (ARPT) for rice seeded at an optimal date (i.e., April seeding) were 9.4, 8.9, and 10.1 Mg ha^{-1} for Wells, CL151, and CLXL745, respectively (Hardke et al., 2012).

Seeding Dates and Cultural Practices

Target seeding dates in 2010 and 2012 were selected prior to the initiation of each study and generally occurred in mid-April, mid-May, and mid-June of each year. The exact seeding dates and approximate 50% heading dates at PTRS and RREC are presented in Table 4.1. At each location, the plots were nine rows (18 cm row spacing) wide, 4.8-m in length, and were drill-seeded at a rate of 115 kg ha^{-1} for Wells and CL151, while the hybrid CLXL745 was seeded at a rate of 32 kg ha^{-1}. Weed, insect, and disease control was maintained in accordance with University of Arkansas Cooperative Extension Service guidelines for the production of drill-seeded, delayed flood rice (Hardke, 2013).

Urea (460 g N kg^{-1}) N fertilizer was used for each site-year–seeding date–cultivar combination. Prior to N fertilizer application, urea was treated with the urease inhibitor NBPT (n-butyl-thiophosphoric tiramide) by blending 1 kg of prilled urea with 3.1 mL of Agrotain Ultra (Koch Agronomic Services, LLC., Wichita, KS). Rice was grown under upland conditions until the four- to five-leaf growth stage at which time a single application of N was broadcast applied onto a dry soil surface (i.e., preflood) at a total N rate of: 45, 90, 135, and 180 kg N ha^{-1}. Unfertilized (0 kg N ha^{-1}) rice was also included as a no-N control treatment. Immediately following N fertilization the field was flooded (< 2 d) to a 10-cm depth and the flood maintained until rice achieved physiological maturity.

Plant Sampling and Grain Yield

Plants were sampled for TN uptake estimation by removing the entire aboveground biomass from a 0.91-m linear portion of the first or second inside bordered row at 50% heading; the growth stage when TN uptake of rice is maximized (Guindo et al., 1994a). Plant tissue samples were dried at 60°C to a constant weight and the total aboveground dry matter of rice is reported after scaling to an area basis (i.e., Mg ha^{-1}). Plant tissue was ground in a Wiley Mill (Thomas Scientific, Swedesboro, NJ) to pass a 1-mm sieve and a subsample removed for N analysis by combustion (Campbell, 1992) using an Elementar Rapid N (Elementar Americas, Inc., Mt. Laurel, NJ). Total N uptake was calculated as plant tissue N concentration (g N g tissue^{-1}) multiplied by total aboveground dry matter at 50% heading. Measurements recorded prior to harvest included TN uptake, aboveground dry matter, number of harvest rows, plot length, and a visual estimate of lodging. The harvested grain from five or six center rows of each plot was weighed and a subsample removed to determine moisture content using a Grain Moisture Tester (DICKEY-john Corp., Auburn, IL). Yield is reported after adjusting grain moisture content to 120 g H_2O kg^{-1} and scaling to an area basis (i.e., kg rough rice ha^{-1}).

Statistical Analysis

The seeding date studies in 2010 and 2012 were established using a split-split plot arrangement of treatments in a randomized complete block design with four replications. Site-year was the whole-plot factor, seeding date was the split-plot factor, cultivar was the split-split plot factor, and fertilizer N rate was the numerical covariate measured at the split-split plot level. Total N uptake, aboveground dry matter, or grain yield was initially regressed on fertilizer N rate with the intercept, linear, and quadratic terms of each model depending upon site-year, seeding date, cultivar, and all main effect interactions. The initial regression model identified 36 unique intercept terms for aboveground rice dry matter and 36 unique intercept, linear, and quadratic terms for TN uptake and rice grain yield. Therefore, analyses of TN uptake, aboveground dry matter, and grain yield of rice were conducted by site-year. Nonsignificant ($P > 0.10$) model terms were removed in sequence until a satisfactory final model was identified. Treatment means for TN uptake, aboveground dry matter, and grain yield were calculated across four replicate N rate treatments for each site-year–seeding date–cultivar combination. Least square means of TN uptake, aboveground dry matter, or grain yield were separated using Fisher's protected LSD and statistical significance interpreted at the 0.10 probability level. All statistical analyses were conducted using PROC MIXED of SAS v9.2 (SAS Institute, Inc., Cary, NC).

RESULTS AND DISCUSSION

Total Nitrogen Uptake of Rice in 2010

Total N uptake of rice measured at 50% heading was a quadratic function of fertilizer N rate in 2010; the intercept term was nonsignificant and did not depend on seeding date or cultivar for PT2010. However, the linear and quadratic coefficient depended on seeding date and cultivar for PT2010 while only the intercept term was dependent on seeding date and cultivar for RR2010 (Table 4.2). Total N uptake was similar across N rates for CL151 and Wells seeded in April and May and increased as fertilizer N rate increased from 0 to 90 kg N ha^{-1} before plateauing or decreasing at N rates > 90 kg N ha^{-1} for PT2010 (Table 4.3; Figure 4.1A and 4.1C). Total N uptake of CLXL745 was also similar when seeded in April and May but did not exhibit a plateau or decrease at N rates > 90 kg N ha^{-1}, as was observed for CL151 and Wells; instead, TN uptake increased as N rate increased from 0 to 180 kg N ha^{-1} (Figure 4.1B). Total N uptake of all three cultivars seeded in June was characterized by concave up, increasing curves that were numerically lower compared to when these cultivars were seeded in April and May, using N rates < 90 kg N ha^{-1}, but tended to increase as N fertilizer rate increased becoming numerically greater using N rates > 135 kg N ha^{-1} for PT2010. Predicted TN uptake of CL151 fertilized with the currently recommended N rate (i.e., 134 kg N ha^{-1}) was significantly greater when the cultivar was seeded in June compared to when seeded in April. The predicted TN uptake of CLXL745 and Wells fertilized with the currently recommended N rate (i.e., 168 kg N ha^{-1}) was significantly greater when seeded in June compared to when seeded in April or May for PT2010.

The common linear and quadratic coefficients among seeding dates and cultivars resulted in three parallel regression curves for each cultivar for RR2010 (Table 4.3; Figure 4.1D–F). Total N uptake of all three cultivars increased as N rate increased from 0 to 180 kg N ha^{-1} for all three seeding dates for RR2010. Predicted TN uptake of all three cultivars was significantly greater when seeded in May compared to April or June for RR2010.

Total Nitrogen Uptake of Rice in 2012

Total N uptake of rice was a linear function of fertilizer N rate in 2012; the intercept term depended on seeding date and cultivar while the linear portion of the function did not depend on seeding date or cultivar for PT2012 and RR2012 (Table 4.2). The linear slope coefficient for TN uptake in response to fertilizer N rate indicated that, when averaged over seeding dates and cultivars, TN uptake of preflood applied fertilizer N was 97 and 98% for PT2012 and RR2012, respectively (Table 4.3). Proper application and management of preflood applied fertilizer N can result in N uptake efficiency ranging from 65 to 75% utilizing the ^{15}N isotopic tracer method (Norman et al., 2003) which is noticeably lower than the N uptake efficiency measured in this study utilizing the difference method. The difference method typically results in higher N uptake efficiencies compared to the ^{15}N isotopic tracer method (Reddy and Patrick, 1976). Similar to the results of the current study, previous research has shown TN uptake of rice to be a linear function of fertilizer N rate with fertilizer N use efficiency based on the difference method as high as 98% (Reiter et al., 2008).

The common linear coefficients among seeding dates indicated TN uptake differences remained constant across fertilizer N rates and resulted in three parallel regression curves for each cultivar for PT2012 and RR2012 (Table 4.3; Figure 4.2A–F). The influence of seeding date on TN uptake was similar between site-years for all three cultivars in 2012 with TN uptake increasing as fertilizer N rate increased to the highest N rate applied of 180 kg N ha^{-1}. Total N uptake of CL151 was significantly greater when seeded in June compared to April or May for RR2012 and significantly greater in June compared to April, with May and June being similar for PT2012. Total N uptake of CLXL745 seeded in June was significantly greater compared to when seeded in April or May for each site-year in 2012. Total N uptake of Wells was significantly greater when seeded in June compared to May or April for RR2012 and significantly greater in May and June compared to April for PT2012, with May and June being similar.

Rice grown using the same daytime temperature (30ºC) but different nighttime temperature (22 or 27ºC) revealed that exposure to the higher nighttime temperature significantly increased N concentration of the aboveground dry matter during the vegetative growth phase of rice (Chen et al., 2013). Kurimoto et al. (2004) reported net N uptake rate of rice during the vegetative growth phase was 60% lower when grown at 15ºC compared to 25ºC, indicating exposure to a consistently higher temperature during vegetative growth positively stimulated N uptake. The tendency for TN uptake (measured at 50% heading) to increase as seeding date was delayed suggests a higher mean air temperature (i.e., faster GDD accumulation rate) may have contributed to the greater TN uptake of late-seeded rice observed in this study. However, the author can only speculate regarding the relative influence of temperature on TN uptake of rice grown under field conditions.

Overall, TN uptake of all three rice cultivars tended to be numerically greater when seeded in May or June compared to when seeded in April, regardless of fertilizer N rate for RR2010, PT2012, and RR2012. For PT2010, TN uptake of all three cultivars exhibited a tendency to be numerically greater when seeded in June compared to when seeded in April or May at only the highest fertilizer N rates. In general, these results suggest that rice was able to more efficiently take up applied N later in the growing season as temperatures become warmer and/or N uptake increased as a result of increased native soil N mineralization in response to warmer temperatures later in the growing season.

Influence of Seeding Date on Aboveground Dry Matter of Rice

The rice cultivars utilized in this study were commonly grown across the rice producing region of Arkansas in 2012 (Hardke and Wilson, 2013). Modern rice cultivars require less time to develop from emergence to maturity and have optimum seeding date windows that are shorter and occur earlier in the year compared to cultivars commonly grown prior to 1984 (Slaton et al., 2003). The vegetative growth phase of rice is partially influenced by the amount of N available for plant uptake and excessive N fertilization can delay maturity extending the number of days required to achieve 50% heading (Harrell et al., 2011). The shorter optimum seeding date window of modern rice cultivars suggests that seeding date may influence plant growth and development in addition to grain yield.

Aboveground Dry Matter of Rice in 2010

Aboveground dry matter of rice measured at 50% heading was a quadratic function of N fertilizer rate in 2010; the intercept term as well as the linear and quadratic coefficient was dependent on seeding date for PT2010, while RR2010 exhibited a common linear and quadratic coefficient and only the intercept term depended on seeding date and cultivar (Table 4.4). For PT2010, aboveground dry matter increased as fertilizer N rate increased from 0 to 135 kg N ha^{-1} but reached a plateau and eventually decreased at fertilizer N rates > 135 kg N ha^{-1} when rice was seeded in April or May (Figure 4.3). The quadratic coefficient in June was nonsignificant and suggests that, when averaged across cultivars, aboveground dry matter increased linearly as N rate increased from 0 to 180 kg N ha^{-1} (Table 4.5). Predicted aboveground dry matter was significantly greater when rice was seeded in May or June compared to April as fertilizer N rate increased from 0 to 180 kg N ha^{-1}. Also, predicted aboveground dry matter was significantly greater when rice was seeded in May compared to June at a N fertilizer rate of 134 kg N ha^{-1}, but was similar when seeded in May compared to June at a N rate of 168 kg N ha^{-1} for PT2010.

The common linear and quadratic coefficient for RR2010 indicated that differences in aboveground dry matter among seeding dates for all three cultivars remained constant regardless of fertilizer N rate, which resulted in three parallel regression curves for each cultivar for RR2010 (Table 4.6; Figure 4.4A–C). In general, aboveground dry matter of all three cultivars tended to be significantly greater when rice was seeded in May compared to when seeded in April or June. However, aboveground dry matter of CL151 seeded in May was similar and not significantly different compared to when seeded in June for RR2010.

Exposure to the same daytime temperature (27ºC) but different nighttime temperature (17 or 27ºC) revealed that higher nighttime temperature significantly increased aboveground dry matter of rice during the vegetative growth phase (Kanno et al., 2009). The greater aboveground dry matter of rice seeded later in the growing season (May or June seeding dates) compared to rice seeded early in the growing season (April seeding dates), observed for both site-years in 2010 for the present study, appeared to support the belief of a beneficial effect of high temperature on aboveground dry matter at a physiologically important growth phase of rice (i.e., 50% heading). However, the positive influence of high temperature on aboveground dry matter may not extend beyond the vegetative growth phase. Previous research conducted under field conditions documented a tendency for total dry matter to decrease as seeding date was delayed (Faw and Johnston, 1975; Dawadi and Chaudhary, 2013) which is contrary to the aboveground dry matter trend presented in this study.

Aboveground Dry Matter of Rice in 2012

In 2012, aboveground dry matter was a quadratic function of fertilizer N rate; the intercept term depended on seeding date and cultivar for PT2012 and RR2012 (Table 4.4). For PT2012, the linear and quadratic coefficient depended on seeding date, while RR2012 exhibited a common linear and quadratic coefficient. A comparison of the aboveground dry matter when no N fertilizer was applied, an intercept term within each site-year, indicated that CLXL745 achieved numerically greater aboveground dry matter at each seeding date compared to CL151 and Wells in 2012 (Table 4.7). Aboveground dry matter of CLXL745 ranged from 7.4 to 15.1 Mg ha^{-1} and from 11.6 to 16.6 Mg ha^{-1} for PT2012 and RR2012, respectively. The results of this study suggested the numerically greater dry matter of CLXL745 compared to CL151 and Wells was a consistent trend across seeding dates for each site-year in 2012. The yield advantage of hybrid rice has been attributed to greater dry matter accumulation (Peng et al., 1999); however, previous research in Asia indicated that cultivars which produce a large number of unproductive tillers and excessive leaf area may cause mutual shading which reduces sink size as well as canopy photosynthesis and may ultimately limit rice grain yield (Dingkuhn et al., 1991).

Aboveground dry matter of all three cultivars seeded in April and May increased as fertilizer N rate increased from 0 to 135 kg N ha^{-1} and then exhibited a tendency to plateau at fertilizer N rates > 135 kg N ha^{-1} for PT2012 (Figure 4.5A–C). The quadratic coefficient for all three cultivars was nonsignificant for the June seeding date and suggests that aboveground dry matter was a linear function of N rate, although, aboveground dry matter did not exhibit a tendency to increase as a linear function of fertilizer N rate in June for PT2012 (Table 4.7; Figure 4.5A–C). One possible explanation for the different fertilizer N response trends among seeding dates could be a result of the numerically greater aboveground dry matter of all three cultivars without fertilizer N when seeded in June compared to April and May for PT2012. Rice seeded in June did not exhibit a positive response to fertilizer N which is in apparent contrast to the positive, curvilinear response of aboveground dry matter to N fertilizer observed for rice seeded in April and May. The predicted aboveground dry matter of all three cultivars seeded in June without fertilizer N was 7.7 Mg ha^{-1} greater compared to rice seeded in April. The predicted aboveground dry matter of CL151 without fertilizer N was 4.6 Mg ha^{-1} greater when seeded in June compared to May. Aboveground dry matter without fertilizer N was 4.5 Mg ha^{-1} greater when seeded in June compared to May for CLXL745 and Wells for PT2012. The numerically greater dry matter of all three cultivars without fertilizer N, when seeded in June compared to April or May, potentially contributed to the lack of a positive response of dry matter to added fertilizer N for PT2012. The aboveground dry matter of CL151 was significantly greater when seeded in May and fertilized with the recommended N rate of 134 kg N ha^{-1} compared to when seeded in April or June, while aboveground dry matter was significantly greater for CLXL745 and Wells when seeded in May and fertilized with the recommended N rate of 168 kg N ha^{-1} compared to when seeded in April or June for PT2012.

The common linear and quadratic coefficients used to describe aboveground dry matter response to N fertilization resulted in three parallel regression curves for each cultivar for RR2012 (Table 4.7; Figure 4.5D–F). Aboveground dry matter of all three cultivars tended to increase as fertilizer N rate increased from 0 to 90 kg N ha^{-1}, plateaued and then slightly decreased at fertilizer N rates > 135 kg N ha^{-1}, regardless of seeding date for RR2012. The aboveground dry matter of all three cultivars was significantly greater when seeded in May or June compared to when seeded in April for RR2012.

Overall, aboveground dry matter of rice was a quadratic function of fertilizer N rate for all four site-years evaluated in this study. Aboveground dry matter of rice seeded in May or June, without fertilizer N, was numerically greater compared to rice seeded in April in 2010 and 2012. In general, these results suggest native soil N mineralized during the growing season was utilized more effectively by late-seeded rice (May or June seeding dates), and as a result greater aboveground dry matter accumulation occurred compared to rice seeded earlier in the growing season (April seeding dates) in 2010 and 2012. The numerically greater TN uptake of late-seeded rice without fertilizer N, compared to rice without fertilizer N and seeded earlier in the growing season, also indicated that native soil N was utilized more effectively for all three cultivars when seeded later in the growing season in 2010 and 2012. However, aboveground dry matter was also used to calculate TN uptake for each seeding date–cultivar combination. Therefore, the general agreement between aboveground dry matter and TN uptake fertilizer N response trends for each seeding date evaluated in 2010 and 2012 was not an unexpected result.

Rice Grain Yield in 2010

The influence of seeding date on rice grain yield has been well documented by previous research (Slaton et al., 2003; Linscombe et al., 2004; Sha and Linscombe 2007; Slaton et al., 2007; Blanche and Linscombe, 2009). However, the response of rice grain yield to N fertilization when seeding date is delayed is not as well understood. Rice grain yield was a quadratic function of fertilizer N rate in 2010; the intercept term as well as the linear and quadratic coefficient depended on seeding date and cultivar which resulted in nine unique regression curves for PT2010, whereas for RR2010, the intercept term and the linear coefficient depended on seeding date and cultivar while the quadratic coefficient depended only on seeding date (Tables 4.8 and 4.9). With the exception of CLXL745, rice grain yield was numerically greater when seeded in April compared to when seeded in May or June for PT2010. Seven of the nine grain yield regression curves developed for PT2010 did not exhibit a yield peak within the range of fertilizer N rates applied in this study (Figure 4.6A–C). This suggests that maximum rice grain yield was not obtained for a majority of the seeding date–cultivar combinations for PT2010. However, CL151 and Wells seeded in June exhibited a grain yield plateau and the yield-maximizing fertilizer N rates for CL151 and Wells were 170 and 123 kg N ha^{-1}, respectively, when seeded in June for PT2010.

The predicted grain yield of CL151 fertilized with the current recommended N rate of 134 kg N ha^{-1} was significantly greater when seeded in April (9255 kg ha^{-1}) compared to when seeded in May (7434 kg ha^{-1}) or June (4744 kg ha^{-1}) for PT2010 (Figure 4.6A). Application of the recommended fertilizer N rate for CL151 increased grain yield by 4836, 4717, and 3842 kg ha^{-1} compared to CL151 which did not receive fertilizer N and seeded in April, May, or June, respectively. The predicted grain yield of CLXL745 fertilized with the current fertilizer N rate recommendation of 168 kg N ha^{-1} was significantly greater when seeded in April (9894 kg ha^{-1}) compared to when seeded in May (8955 kg ha^{-1}) or June (4600 kg ha^{-1}) for PT2010 (Figure 4.6B). Application of the recommended fertilizer N rate for CLXL745 increased grain yield by 4187, 3319, and 3170 kg ha^{-1} compared to CLXL745 which did not receive fertilizer N and seeded in April, May, or June, respectively. The predicted grain yield of Wells fertilized with the current recommended N rate of 168 kg N ha^{-1} was also significantly greater when seeded in April (9534 kg ha^{-1})

compared to when seeded in May (8537 kg ha^{-1}) or June (4717 kg ha^{-1}) for PT2010 (Figure 4.6C). Application of the currently recommended fertilizer N rate for Wells increased grain yield by 4998, 5252, and 1853 kg ha^{-1} compared to Wells which did not receive fertilizer N and seeded in April, May, or June, respectively.

Rice grain yield of all three cultivars seeded in April tended to increase as fertilizer N rate increased from 0 to 135 kg N ha^{-1} and plateaued at N rates > 135 kg N ha^{-1} for RR2010 (Figure 4.6D–F). Whereas, grain yield of all three cultivars seeded in May or June tended to reach a plateau at a fertilizer N rate of 90 kg N ha^{-1}. Grain yield was significantly greater when rice was seeded in April compared to May or June across all of the fertilizer N application rates for all three cultivars for RR2010. Similar to the results of this study, previous research reported grain yield was significantly affected by seeding date and tended to be greatest when seeded in April or earlier and generally decreased when rice was seeded in May or June in Arkansas (Slaton et al., 2007).

The influence of N fertilization on rice grain yield in response to seeding date appeared to be similar when rice was seeded in May or June across all fertilizer N rates applied for RR2010 (Figure 4.6D–F). Application of the currently recommended fertilizer N rate of 134 kg N ha^{-1} for CL151 seeded in April resulted in a yield increase of 4843 kg ha^{-1} compared to the yield of CL151 without fertilizer N, while CL151 fertilized with the recommended N rate only increased grain yield 1200 kg ha^{-1} when seeded in May and 1751 kg ha^{-1} when seeded in June. Similarly, CLXL745 seeded in April and fertilized with the recommended fertilizer N rate of 168 kg N ha^{-1} resulted in a yield increase of 5511 kg ha^{-1} compared to the yield of CLXL745 without fertilizer N, while application of the recommended fertilizer N rate increased yield by only 1673 kg ha^{-1} when seeded in May and 2158 kg ha^{-1} when seeded in June. Grain yield of Wells seeded in April and fertilized with the recommended N rate exhibited an increase of 5144 kg ha^{-1} compared to Wells which did not receive fertilizer N, while application of the recommended N rate increased yield by only 1305 kg ha^{-1} when seeded in May and 1791 kg ha^{-1} when seeded in June.

Wilson et al. (2010b) reported atypical grain yields of early-seeded as well as late-seeded rice and documented a substantial grain yield reduction ranging from 18 to 64% for late-seeded rice, despite application of 134 kg N ha^{-1}, for ARPT at RREC in 2010. Abnormally high air temperatures could have contributed to the low grain yield of early-seeded rice and the combination of high air temperature and a shorter vegetative growth phase was intimated as being particularly detrimental to the grain yield of late-seeded rice in 2010 (Wilson et al., 2010b). In the present study, application of currently recommended fertilizer N rates increased grain yield for each seeding date–cultivar combination compared to unfertilized rice for RR2010; although the grain yield increase of rice seeded in May or June was < 50% of the yield increase of rice seeded in April for RR2010. The results of this study are in agreement with the rice grain yield results reported by Wilson et al. (2010b) and indicated application of current fertilizer N rate recommendations failed to increase grain yield of late-seeded rice for RR2010.

Rice grain yield exhibited a plateau for each cultivar when seeded in April, May, or June indicating that maximum grain yield was achieved within the range of fertilizer N rates applied in this study for RR2010 (Figure 4.6D–F). The yield maximizing fertilizer N rate for all three cultivars seeded in April, May, or June was estimated using the grain yield regression curves developed for RR2010 (Table 4.9). Yield-maximizing fertilizer N rates were always greatest in April and declined with each successive seeding date for all three cultivars. The predicted fertilizer N rates required to achieve maximum grain yield were 156, 127, and 126 kg N ha^{-1} for CL151 when seeded in April, May, or June, respectively. The predicted fertilizer N rates required to achieve maximum grain yield were 165, 151, and 142 kg N ha^{-1} for CLXL745 when seeded in April, May, or June, respectively. The predicted fertilizer N rates required to achieve maximum grain yield were 160, 134, and 132 kg N ha^{-1} for Wells seeded in April, May, or June, respectively. This result suggests that as seeding date was delayed the fertilizer N rate required to achieve maximum grain yield decreased as a result of a yield plateau which occurred at a lower fertilizer N rate for rice seeded in May or June compared to when rice was seeded in April for RR2010.

Rice Grain Yield in 2012

Rice grain yield was a quadratic function of fertilizer N rate in 2012 (Table 4.8). The intercept term and linear coefficient depended on seeding date and cultivar, while the quadratic coefficient depended only on cultivar for PT2012. The intercept term as well as the linear and quadratic coefficient depended on seeding date and cultivar resulting in nine unique regression curves for RR2012 (Table 4.9). The results of this study are in agreement with previous research which indicated rice grain yield increased as a curvilinear function of N rate in Arkansas (Golden et al., 2006; Golden et al., 2009). Grain yield of CL151 and CLXL745 seeded in May increased as fertilizer N rate increased from 0 to 180 kg N ha^{-1}, while grain yield of Wells seeded in May increased as fertilizer N rate increased from 0 to 135 kg N ha^{-1} and plateaued at N rates > 135 kg N ha^{-1} for PT2012 (Figure 4.7A–C). Six of the nine regression curves either did not exhibit a positive response to fertilizer N or did not exhibit a grain yield plateau within the range of fertilizer N rates applied in this study. Clearfield 151 seeded in April achieved a yield plateau, however, the predicted yield-maximizing N rate (197 kg N ha^{-1}) exceeded the highest fertilizer N rate applied (180 kg N ha^{-1}) suggesting that maximum grain yield was not achieved for PT2012. Wells achieved a grain yield plateau within the range of fertilizer N rates applied and the predicted yield-maximizing N rate was 137, 161, and 109 kg N ha^{-1} when seeded in April, May, or June, respectively for PT2012.

Clearfield 151 and CLXL745 seeded in June exhibited linear and quadratic coefficients that were not significantly different from zero for PT2012 (Table 4.9; Figure 4.7A and 4.7B). Grain yield of CL151 without fertilizer N, an intercept term, seeded in June was 6030 kg ha^{-1} greater for PT2012 compared to PT2010, while grain yield of CLXL745 without fertilizer N and seeded in June was 6150 kg ha^{-1} greater for PT2012 compared to PT2010. The numerically greater grain yield of CL151 and CLXL745 without fertilizer N and seeded in June for PT2012, compared to PT2010, suggests that grain yield did not respond positively to N fertilization as a result of the much greater grain yield of unfertilized rice (i.e., 0 kg N ha^{-1}) for PT2012. The grain yield of Wells without fertilizer N was 869, 894, and 2610 kg ha^{-1} greater when seeded in April, May, or June respectively, for PT2012 compared to PT2010. However, all three regression curves for Wells exhibited linear and quadratic coefficients that were statistically different from zero indicating that Wells exhibited a positive, curvilinear response to fertilizer N for all three seeding dates for PT2012.

Grain yield was similar for all three cultivars when seeded in April or May compared to June across a majority of fertilizer N rates evaluated for RR2012 (Figure 4.7D–F). There was a tendency for rice grain yield to decline when fertilizer N rate was > 45 kg N ha^{-1} for CL151 when seeded in April and May. Whereas, rice grain yield tended to decline when fertilizer N rate was > 90 kg N ha^{-1} for CLXL745 seeded in May and for Wells seeded in April for RR2012. However, grain yield of Wells seeded in May increased as N rate increased from 0 to 135 kg N ha^{-1}, plateaued and eventually decreased at fertilizer N rates > 135 kg N ha^{-1}. Grain yield of all three cultivars exhibited a plateau and eventually decreased at fertilizer N rates > 45 kg N ha^{-1} when seeded in June for RR2012.

Grain yield of rice seeded in June was noticeably lower compared to the grain yield of rice seeded in April or May for RR2012 (Figure 4.7D–F). Hardke et al. (2012) also noted reduced grain yield for late-seeded rice and suggested that exposure to high average air temperatures resulted in rapid plant growth and development which likely contributed to the reduced grain yield obtained for late-seeded rice compared to early-seeded rice for ARPT at RREC in 2012. While the underlying factors contributing to the yield advantage of early-seeded rice were not elucidated by the results of this study, the apparent reduction in grain yield observed for rice seeded in June compared to rice seeded in April or May, regardless of fertilizer N rate, suggests that late-seeded rice also responds much differently to fertilizer N compared to early-seeded rice for RR2012.

Grain yield of all three cultivars without fertilizer N was numerically greater for RR2012 compared to RR2010 when seeded in April, May, or June (Table 4.9). Results of ARPT seeding date studies (Wilson et al., 2010b; Hardke et al., 2012) were similar to the results of this study and ARPT reported a noticeable difference in grain yield of Wells in 2010 compared to 2012 for RREC. Wells grain yield at RREC was 1714 and 6300 kg ha^{-1} greater when seeded in April and May, respectively, in 2012 compared to 2010 despite receiving 134 kg N ha^{-1} in both years (Wilson et al., 2010b; Hardke et al., 2012). Clearfield 151 and CLXL745 were not included in the seeding date studies conducted in 2012. However, grain yields of CL151 and CLXL745 seeded in May was 5242 and 3881 kg ha^{-1}, respectively, for ARPT in 2010 for RREC (Wilson et al., 2010b) and were noticeably lower compared to the grain yield of CL151 (8417 kg ha^{-1}) and CLXL745 (8165 kg ha^{-1}) seeded in May at RREC in 2009 despite receiving 134 kg N ha^{-1} in both years (Wilson et al., 2009).

Similar to the grain yield trend observed for PT2012, grain yield was not a quadratic function of fertilizer N rate for each seeding date–cultivar combination evaluated for RR2012 (Table 4.9). The linear and quadratic coefficients of the regression curves for CL151 and Wells seeded in June and for CLXL745 seeded in April were not significantly different from zero. Grain yield of CL151 and Wells without fertilizer N and seeded in June was 2049 and 1607 kg ha^{-1} greater respectively, for RR2012 compared to RR2010. Grain yield of CLXL745 without fertilizer N and seeded in April was 986 kg ha^{-1} greater for RR2012 compared to RR2010. The numerically greater grain yield for CL151 and Wells without fertilizer N and seeded in June and CLXL745 without fertilizer N and seeded in April indicated that a greater amount of native soil N uptake contributed to the lack of a positive response of rice grain yield to the addition of N fertilizer observed for RR2012 compared to RR2010. Five of the nine grain yield regression curves exhibited a positive N fertilizer response within the range of N rates applied in this study and the yield maximizing N rate was calculated for CL151 and Wells seeded in April and May as well as CLXL745 seeded in May for RR2012 (Figure 2.7D–F). The yield maximizing N rate for CL151 seeded in April and May was 55 and 60 kg N ha^{-1}, respectively, and was 87 kg N ha^{-1} for CLXL745 seeded in May. The yield maximizing N rate for Wells seeded in April and May was 96 and 141 kg N ha^{-1}, respectively for RR2012.

Overall, rice grain yield was a quadratic function of fertilizer N rate for all four site-years in 2010 and 2012. Rice seeded in April tended to achieve greater grain yield compared to when rice was seeded in May and June regardless of fertilizer N rate in 2010. Yield-maximizing fertilizer N rates were greatest for rice seeded early in the growing season (April seeding dates) and were reduced as seeding was delayed (May or June seeding dates) for each seeding date–cultivar combination for RR2010. Clearfield 151 exhibited the greatest reduction in yield-maximizing fertilizer N rates when seeded in May or June compared to April, while the yield maximizing fertilizer N rates for CLXL745 were reduced the least when seeded in May or June compared to April for RR2010. Grain yield of unfertilized rice (i.e., 0 kg N ha^{-1}) was numerically greater in 2012 compared to 2010 and could have potentially contributed to the different fertilizer N response, particularly the lack of a positive grain yield response to fertilizer N, of the seeding date–cultivar combinations evaluated in this study.

CONCLUSIONS

This research was conducted to characterize the influence of N fertilization and seeding date on TN uptake at 50% heading, aboveground dry matter at 50% heading, and grain yield of rice grown on silt loam soils. Four site-years of fertilizer N rate response trials indicated, in general, TN uptake and aboveground dry matter tended to be numerically greater for rice seeded in May or June compared to rice seeded in April in 2010 and 2012. Rice grain yield was numerically greater when seeded in April compared to when seeded in May or June in 2010, while grain yield tended to be similar when seeded in April or May compared to when seeded in June in 2012. Maximum grain yield in Arkansas is typically achieved when rice accumulates 150 to 200 kg N ha^{-1} during the growing season (Norman et al., 2003). The cultivars utilized in this study achieved > 150 kg N ha^{-1} at 50% heading regardless of seeding date in 2010 and 2012. Although there was a different trend for TN uptake of rice in response to fertilizer N rate in 2010 and 2012, seeding date did not appear to pose any limitation to the TN uptake of CL151, CLXL745, or Wells when fertilized with the current University of Arkansas N rate recommendation.

One of four site-years (i.e., RR2010) was used to predict the yield maximizing N fertilizer rate for each seeding date–cultivar combination. Three of four site-years (i.e., PT2010, PT2012, and RR2012) exhibited a grain yield trend in response to fertilizer N rate which prevented the identification of a yield maximizing fertilizer N rate for each seeding date–cultivar combination, potentially as a result of greater grain yield variability and/or the lack of a grain yield plateau within the range of fertilizer N rates applied. Predicted yield-maximizing fertilizer N rates for RR2010 were greatest when rice was seeded in April. As seeding date was delayed the yield maximizing fertilizer N rate, averaged across cultivars, was reduced by 14 and 17% when rice was seeded in May or June, respectively. In contrast to the yield trend observed in 2010, grain yield did not responded positively to N fertilization for each seeding date–cultivar combination in 2012 and five of the 18 regression curves exhibited nonsignificant linear and quadratic coefficients in 2012. This result reflected the different fertilizer N response of grain yield among seeding dates for rice seeded in April, May, or June in 2010 compared to 2012.

The results presented in this manuscript do not provide a clear explanation as to why rice grain yield tended to achieve a maximum when seeded in April and a decreased N response when seeded in May or June in Arkansas. However, the results of this study provided insight into TN uptake, aboveground dry matter, and grain yield in response to N fertilization for rice seeded in April, May, or June. The greater TN uptake and aboveground dry matter of rice seeded in May or June compared to rice seeded in April suggested that the yield advantage exhibited by rice seeded in April was not a result of more efficient N uptake or greater dry matter production. One possible explanation for the yield advantage of rice seeded in April is that the greater aboveground dry matter of rice seeded in May or June was the result of a greater number of unproductive tillers which would contribute to dry matter accumulation but not grain yield. The trend among yield maximizing fertilizer N rates for each cultivar as seeding date was delayed remained unclear following the completion of this study in 2012. Therefore, continued research aimed at identifying the similarity or difference among yield-maximizing fertilizer N rates for early-seeded rice compared to late-seeded rice is needed for modern cultivars grown using the drill-seeded, delayed flood production system in Arkansas.

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Table 4.1. Site-year, seeding date, and approximate date of 50% heading for drill-seeded, delayed flood rice grown in Arkansas during 2010 and 2012. Number shown in parentheses is day of calendar year.

Site-year	Seeding Date	50% Heading Date†
PT2010	15 April (105 d)	23 July (204 d)
	13 May (133 d)	4 August (216 d)
	15 June (166 d)	2 September (245 d)
RR2010	13 April (103 d)	21 July (202 d)
	12 May (132 d)	3 August (215 d)
	10 June (161 d)	31 August (243 d)
PT2012	10 April (101 d)	20 July (202 d)
	11 May (132 d)	1 August (214 d)
	5 June (157 d)	23 August (236 d)
RR2012	13 April (104 d)	23 July (205 d)
	11 May (132 d)	2 August (215 d)
	19 June (171 d)	7 September (251 d)

†Estimated emergence date and plant growth phases were based on seeding date and calculated using the degree-day 10 (DD10) of Wells.

Table 4.2. Analysis of variance *P* values for total nitrogen (TN) uptake of rice determined at 50% heading and affected by seeding date (D) and cultivar (C) as well as all main effect interactions at Pine Tree Research Station (PTRS) in 2010 (PT2010) and 2012 (PT2012), and at Rice Research and Extension Center (RREC) in 2010 (RR2010) and 2012 (RR2012).

Source of Variation		Site-year			
	df	PT2010	RR2010	PT2012	RR2012
		----------------------------*P* value------------------------------------			
Seeding Date (D)	2	0.4746	0.0008	0.0005	< 0.0001
Cultivar (C)	2	0.6850	0.1137	< 0.0001	< 0.0001
D x C	4	0.6441	0.0732	0.0191	0.1819
Linear N rate	1	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Linear N rate x D	2	0.0015	0.3530	0.4658	0.6914
Linear N rate x C	2	0.1040	0.9992	0.7405	0.5708
Linear N rate x D x C	4	0.6903	0.8566	0.1433	0.9682
Quadratic N rate	1	0.0687	0.0255	0.7928	0.8919
Quadratic N rate x D	2	< 0.0001	0.1993	0.4739	0.1141
Quadratic N rate x C	2	0.0270	0.8548	0.4045	0.7696
Quadratic N rate x D x C	4	0.5509	0.5580	0.1782	0.8702

Table 4.3. Intercept terms and regression coefficients for total nitrogen (TN) uptake determined at 50% heading and affected by seeding date (April, May or June) and cultivar (CL151, CLXL745 and Wells) at Pine Tree Research Station (PTRS) in 2010 (PT2010) and 2012 (PT2012) as well as at Rice Research and Extension Center in 2010 (RR2010) and 2012 (RR2012). Standard errors are shown in parentheses.

		CL151			CLXL745			Wells		
Site-year	Seeding Date	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic
PT2010	April	56.4	1.80	-0.0075	64.8	1.04	-0.0024	55.0	1.54	-0.0056
		(12.5)	(0.32)	(0.0017)	(12.6)	(0.33)	(0.0018)†	(12.7)	(0.32)	(0.0017)
	May	55.1	1.79	-0.0069	47.3	1.03	-0.0017	39.4	1.53	-0.0049
		(12.3)	(0.34)	(0.0018)	(13.7)	(0.33)	(0.0017)†	(13.7)	(0.34)	(0.0018)
	June	66.8	0.57	0.0024	76.2	-0.19	0.0075	52.1	0.32	0.0043
		(12.4)	(0.32)	(0.0017)†	(13.6)	(0.34)†	(0.0018)	(13.5)	(0.34)†	(0.0018)
RR2010	April	71.5	0.57	0.0014	67.8	0.57	0.0014	44.5	0.57	0.0014
		(7.7)	(0.12)	(0.0006)	(7.9)	(0.12)	(0.0006)	(7.8)	(0.12)	(0.0006)
	May	95.8	0.57	0.0014	97.4	0.57	0.0014	99.2	0.57	0.0014
		(7.7)	(0.12)	(0.0006)	(7.8)	(0.12)	(0.0006)	(7.8)	(0.12)	(0.0006)
	June	65.7	0.57	0.0014	77.2	0.57	0.0014	66.3	0.57	0.0014
		(7.9)	(0.12)	(0.0006)	(8.4)	(0.12)	(0.0006)	(8.0)	(0.12)	(0.0006)
PT2012	April	64.4	0.97	-‡	84.4	0.97	-‡	35.3	0.97	-‡
		(8.7)	(0.04)		(9.0)	(0.04)		(8.7)	(0.04)	
	May	78.7	0.97	-‡	97.2	0.97	-‡	91.2	0.97	-‡
		(8.9)	(0.04)		(9.0)	(0.04)		(8.9)	(0.04)	
	June	93.3	0.97	-‡	129.9	0.97	-‡	83.6	0.97	-‡
		(9.0)	(0.04)		(9.0)	(0.04)		(9.0)	(0.04)	
RR2012	April	80.7	0.98	-§	121.6	0.98	-§	66.8	0.98	-§
		(7.9)	(0.05)		(7.8)	(0.05)		(7.9)	(0.05)	
	May	129.6	0.98	-§	170.5	0.98	-§	115.7	0.98	-§
		(7.8)	(0.05)		(8.1)	(0.05)		(7.9)	(0.05)	
	June	167.3	0.98	-§	208.2	0.98	-§	153.3	0.98	-§
		(8.3)	(0.05)		(8.4)	(0.05)		(8.2)	(0.05)	

†Coefficient is not significantly different than zero. §Quadratic coefficient not significant (*P* = 0.89) in the final model.

‡Quadratic coefficient not significant (*P* = 0.79) in the final model.

Table 4.4. Analysis of variance *P* values for aboveground dry matter of rice determined at 50% heading and affected by seeding date (D) and cultivar (C) as well as all main effect interactions for Pine Tree Research Station (PTRS) in 2010 (PT2010) and 2012 (PT2012) and at Rice Research and Extension Center (RREC) in 2010 (RR2010) and 2012 (RR2012).

Source of Variation		Site-year			
	df	PT2010	RR2010	PT2012	RR2012
		----------------------------*P* value--------------------------------			
Seeding Date (D)	2	0.0265	< 0.0001	< 0.0001	< 0.0001
Cultivar (C)	2	0.1271	< 0.0001	< 0.0001	< 0.0001
D x C	4	0.2788	0.0303	0.7715	0.8346
Linear N rate	1	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Linear N rate x D	2	0.0632	0.6982	< 0.0001	0.4549
Linear N rate x C	2	0.9004	0.2515	0.9870	0.9094
Linear N rate x D x C	4	0.2492	0.4812	0.1139	0.6502
Quadratic N rate	1	0.0001	< 0.0001	< 0.0001	< 0.0001
Quadratic N rate x D	2	0.0260	0.5575	0.0046	0.9716
Quadratic N rate x C	2	0.8283	0.3854	0.7764	0.6984
Quadratic N rate x D x C	4	0.2118	0.7582	0.1127	0.9189

Table 4.5. Intercept terms and regression coefficients for aboveground dry matter of rice determined at 50% heading, averaged across cultivar, as affected by seeding date (April, May, or June) at Pine Tree Research Station (PTRS) in 2010 (PT2010). Standard errors are shown in parentheses.

Site-year	Seeding Date	Intercept	Linear	Quadratic
	April	5.5	0.09	-0.00031
PT2010		(0.9)	(0.02)	(0.00012)
	May	8.8	0.13	-0.00050
		(0.9)	(0.02)	(0.00013)
	June	8.1	0.06	-0.00003
		(0.9)	(0.02)	(0.00012)†

†Coefficient is not significantly different than zero.

Table 4.6. Intercept terms and regression coefficients for aboveground dry matter of rice determined at 50% heading and affected by seeding date (April, May or June) and cultivar (CL151, CLXL745 and Wells) at Rice Research and Extension Center (RREC) in 2010 (RR2010). Standard errors are shown in parentheses.

		CL151			CLXL745			Wells		
Site-year	Seeding Date	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic
RR2010	April	7.4	0.07	-0.00020	7.6	0.07	-0.00020	5.7	0.07	-0.00020
		(0.6)	(0.01)	(0.00005)	(0.6)	(0.01)	(0.00005)	(0.6)	(0.01)	(0.00005)
	May	11.3	0.07	-0.00020	14.2	0.07	-0.00020	11.8	0.07	-0.00020
		(0.6)	(0.01)	(0.00005)	(0.6)	(0.01)	(0.00005)	(0.6)	(0.01)	(0.00005)
	June	10.1	0.07	-0.00020	12.7	0.07	-0.00020	9.3	0.07	-0.00020
		(0.6)	(0.01)	(0.00005)	(0.7)	(0.01)	(0.00005)	(0.6)	(0.01)	(0.00005)

Table 4.7. Intercept terms and regression coefficients for aboveground dry matter of rice determined at 50% heading and affected by seeding date (April, May or June) and cultivar (CL151, CLXL745 and Wells) at Pine Tree Research Station (PTRS) in 2012 (PT2012) and at Rice Research and Extension Center in 2012 (RR2012). Standard errors are shown in parentheses.

		CL151			CLXL745			Wells		
Site-year	Seeding Date	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic
PT2012	April	5.0	0.11	-0.00031	7.4	0.11	-0.00031	4.7	0.11	-0.00031
		(0.5)	(0.01)	(0.00007)	(0.5)	(0.01)	(0.00007)	(0.5)	(0.01)	(0.00007)
	May	8.1	0.13	-0.00043	10.6	0.13	-0.00043	7.9	0.13	-0.00043
		(0.5)	(0.01)	(0.00007)	(0.5)	(0.01)	(0.00007)	(0.5)	(0.01)	(0.00007)
	June	12.7	0.02	-0.00009	15.1	0.02	-0.00009	12.4	0.02	-0.00009
		(0.5)	(0.01)	(0.00007)†	(0.5)	(0.01)	(0.00007)†	(0.5)	(0.01)	(0.00007)†
RR2012	April	8.2	0.07	-0.00027	11.6	0.07	-0.00027	8.5	0.07	-0.00027
		(0.6)	(0.01)	(0.00006)	(0.6)	(0.01)	(0.00006)	(0.6)	(0.01)	(0.00006)
	May	13.2	0.07	-0.00027	16.6	0.07	-0.00027	13.5	0.07	-0.00027
		(0.6)	(0.01)	(0.00006)	(0.6)	(0.01)	(0.00006)	(0.6)	(0.01)	(0.00006)
	June	12.0	0.07	-0.00027	15.4	0.07	-0.00027	12.3	0.07	-0.00027
		(0.6)	(0.01)	(0.00006)	(0.6)	(0.01)	(0.00006)	(0.6)	(0.01)	(0.00006)

†Coefficient is not significantly different than zero.

Table 4.8. Analysis of variance *P* values for rice grain yield as affected by seeding date (D), cultivar (C) as well as all main effect interactions for Pine Tree Research Station (PTRS) in 2010 (PT2010) and 2012 (PT2012) and at Rice Research and Extension Center (RREC) in 2010 (RR2010) and 2012 (RR2012).

Source of Variation		Site-year			
	df	PT2010	RR2010	PT2012	RR2012
		----------------------------*P* value--------------------------------			
Seeding Date (D)	2	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Cultivar (C)	2	< 0.0001	< 0.0001	< 0.0001	0.0677
D x C	4	< 0.0001	< 0.0001	0.0009	0.3699
Linear N rate	1	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Linear N rate x D	2	0.0050	< 0.0001	0.0080	0.0656
Linear N rate x C	2	< 0.0001	0.0183	< 0.0001	0.1559
Linear N rate x D x C	4	0.0757	0.2822	0.0012	0.1037
Quadratic N rate	1	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Quadratic N rate x D	2	0.5053	< 0.0001	0.1052	0.1294
Quadratic N rate x C	2	< 0.0001	0.2360	< 0.0001	0.3705
Quadratic N rate x D x C	4	0.0009	0.4572	0.0038	0.0245

Table 4.9. Intercept terms and regression coefficients for grain yield of rice as affected by seeding date (April, May or June) and cultivar (CL151, CLXL745 and Wells) at Pine Tree Research Station (PTRS) in 2010 (PT2010) and at Rice Research and Extension Center (RREC) in 2010 (RR2010). Standard errors are shown in parentheses.

		CL151			CLXL745			Wells		
Site-year	Seeding Date	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic	Intercept	Linear	Quadratic
PT2010	April	4419	56.5	-0.1532	5707	11.0	0.0829	4536	50.2	-0.1220
		(250)	(6.2)	(0.0332)	(250)	(6.2)	(0.0332)	(250)	(6.2)	(0.0332)
	May	2717	46.8	-0.0864	5636	27.0	-0.0431	3285	46.2	-0.0888
		(250)	(6.2)	(0.0332)	(250)	(6.2)	(0.0332)†	(250)	(6.2)	(0.0332)
	June	902	47.3	-0.1392	1430	-6.8	0.1526	2864	34.4	-0.1394
		(250)	(6.2)	(0.0332)	(251)	(6.3)†	(0.0036)	(250)	(6.2)	(0.0332)
RR2010	April	5597	63.3	-0.2023	6621	66.8	-0.2023	3630	64.6	-0.2023
		(176)	(3.2)	(0.0165)	(175)	(3.2)	(0.0165)	(176)	(3.2)	(0.0165)
	May	1791	19.0	-0.0747	3044	22.5	-0.0747	1822	20.3	-0.0747
		(175)	(3.1)	(0.0164)	(175)	(3.1)	(0.0164)	(175)	(3.1)	(0.0164)
	June	3242	27.9	-0.1107	3840	31.4	-0.1107	3048	29.3	-0.1107
		(175)	(3.2)	(0.0165)	(175)	(3.2)	(0.0165)	(175)	(3.2)	(0.0165)
PT2012	April	9084	-18.3	0.0862	8730	34.9	-0.1842	4397	94.7	-0.4248
		(498)	(13.1)†	(0.0694)†	(444)	(12.4)	(0.0659)	(506)	(13.6)	(0.0755)
	May	5152	43.3	-0.1309	5597	34.9	-0.0344	4202	48.4	-0.1237
		(436)	(11.4)	(0.0606)	(436)	(11.6)	(0.0634)†	(436)	(11.4)	(0.0606)
	June	6998	-1.8	0.0376	7662	1.1	-0.0047	5805	43.4	-0.1963
		(436)	(11.4)†	(0.0606)†	(439)	(11.8)†	(0.0620)†	(436)	(11.4)	(0.0606)
RR2012	April	7258	20.5	-0.1875	7607	7.9	-0.0220	6622	55.5	-0.2899
		(388)	(11.5)	(0.0642)	(413)	(11.8)†	(0.0651)†	(413)	(11.9)	(0.0677)
	May	6840	33.5	-0.2813	7189	31.7	-0.1818	6203	39.4	-0.1396
		(381)	(11.2)	(0.0634)	(387)	(11.8)	(0.0664)	(387)	(11.3)	(0.0636)
	June	5291	8.7	-0.0727	5640	7.3	-0.1055	4655	13.4	-0.0820
		(387)	(11.3)†	(0.0636)†	(412)	(11.6)†	(0.0646)	(412)	(11.6)†	(0.0646)†

†Coefficient is not significantly different than zero.

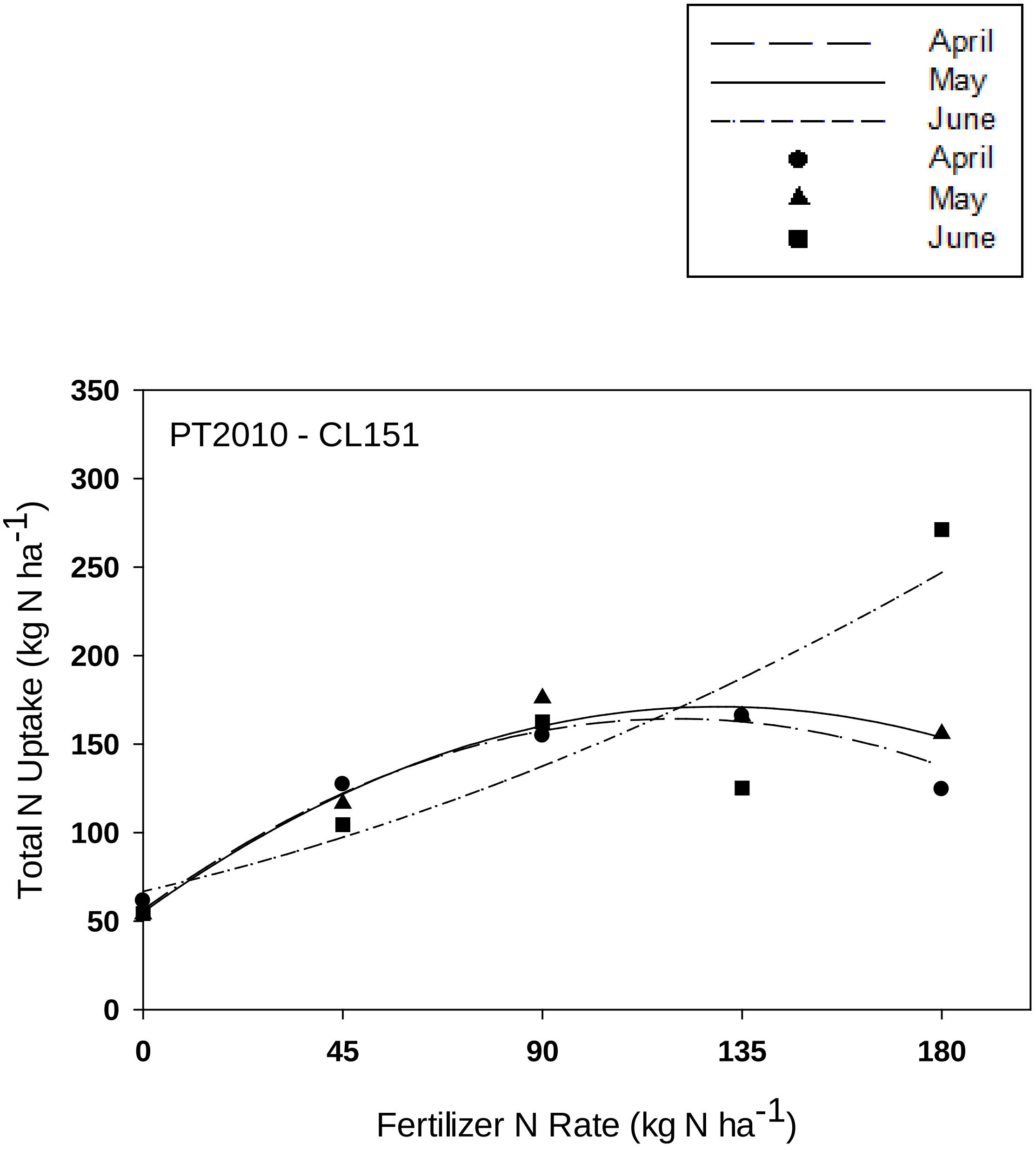

Figure 4.1A. Total nitrogen (TN) uptake of CL151 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2010 (PT2010).

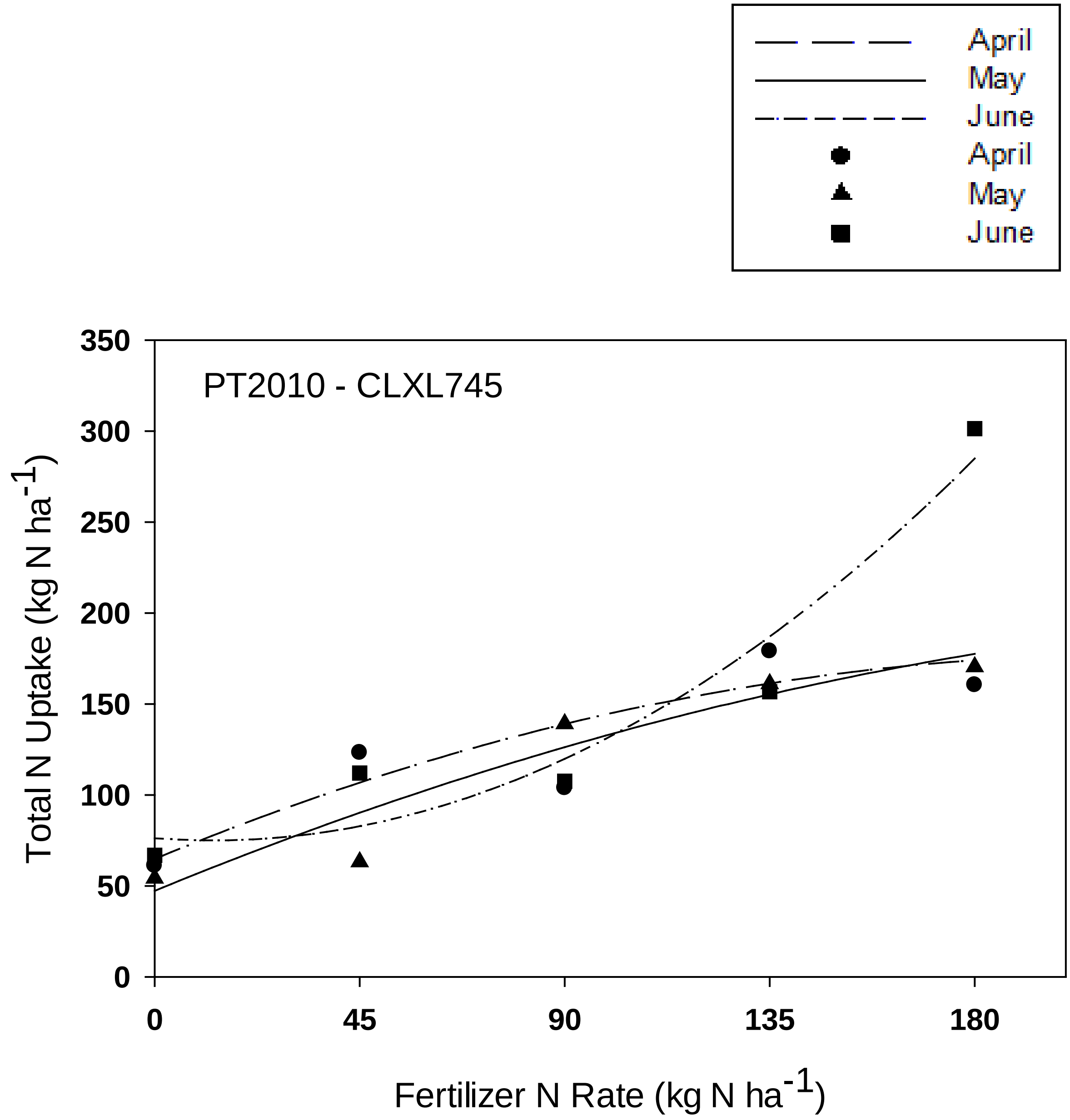

Figure 4.1B. Total nitrogen (TN) uptake of CLXL745 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2010 (PT2010).

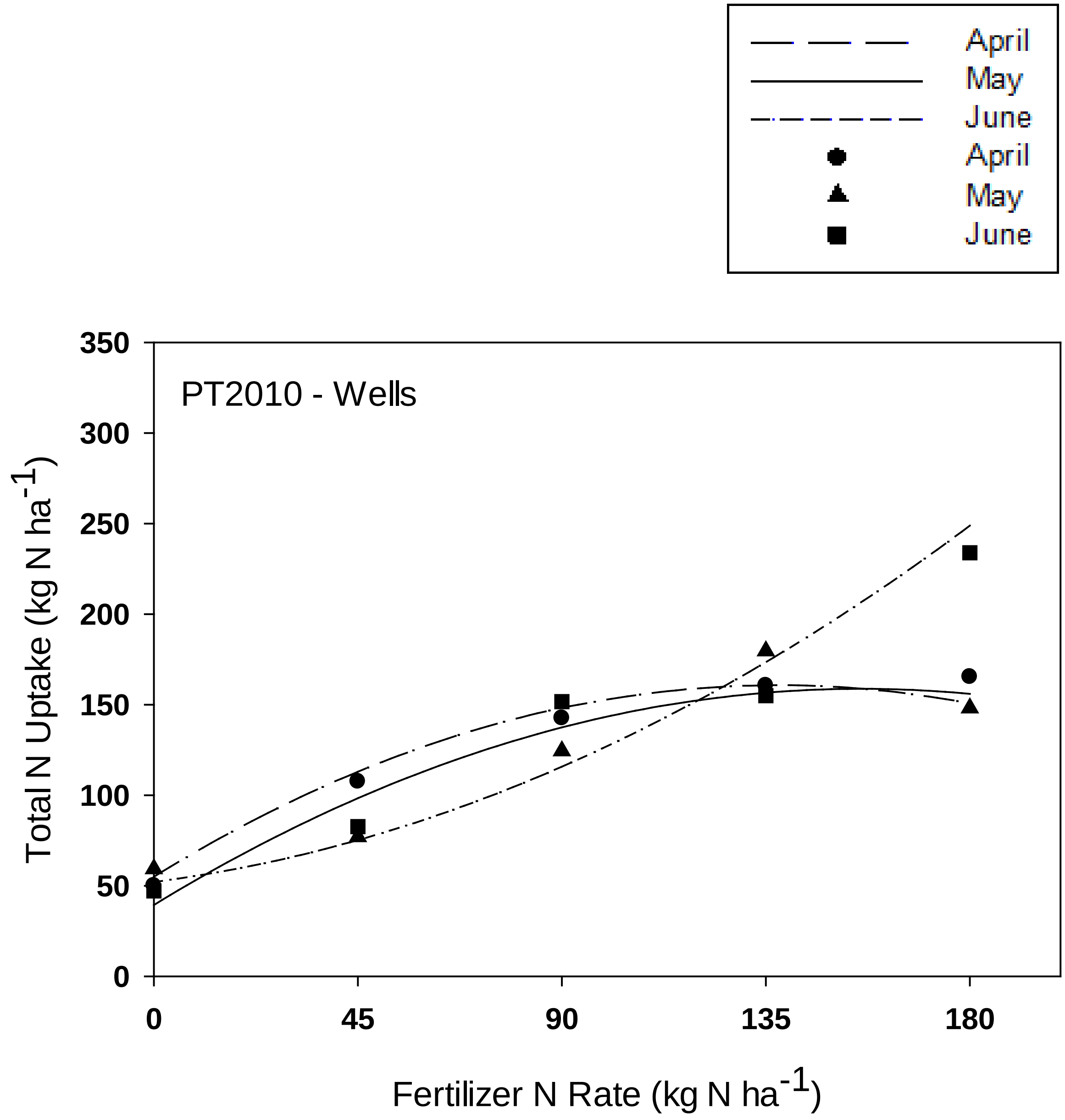

Figure 4.1C. Total nitrogen (TN) uptake of Wells at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2010 (PT2010).

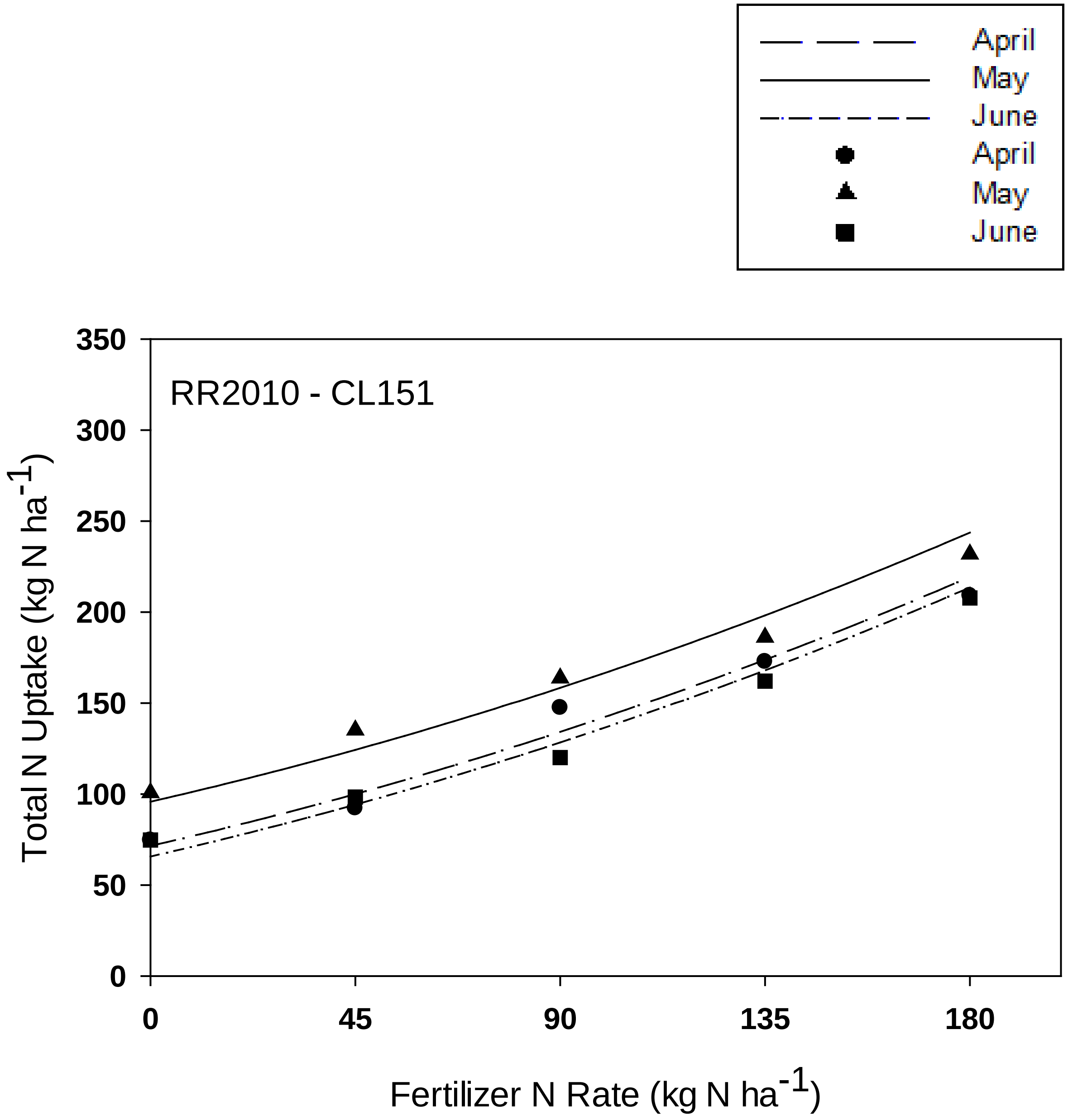

Figure 4.1D. Total nitrogen (TN) uptake of CL151 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

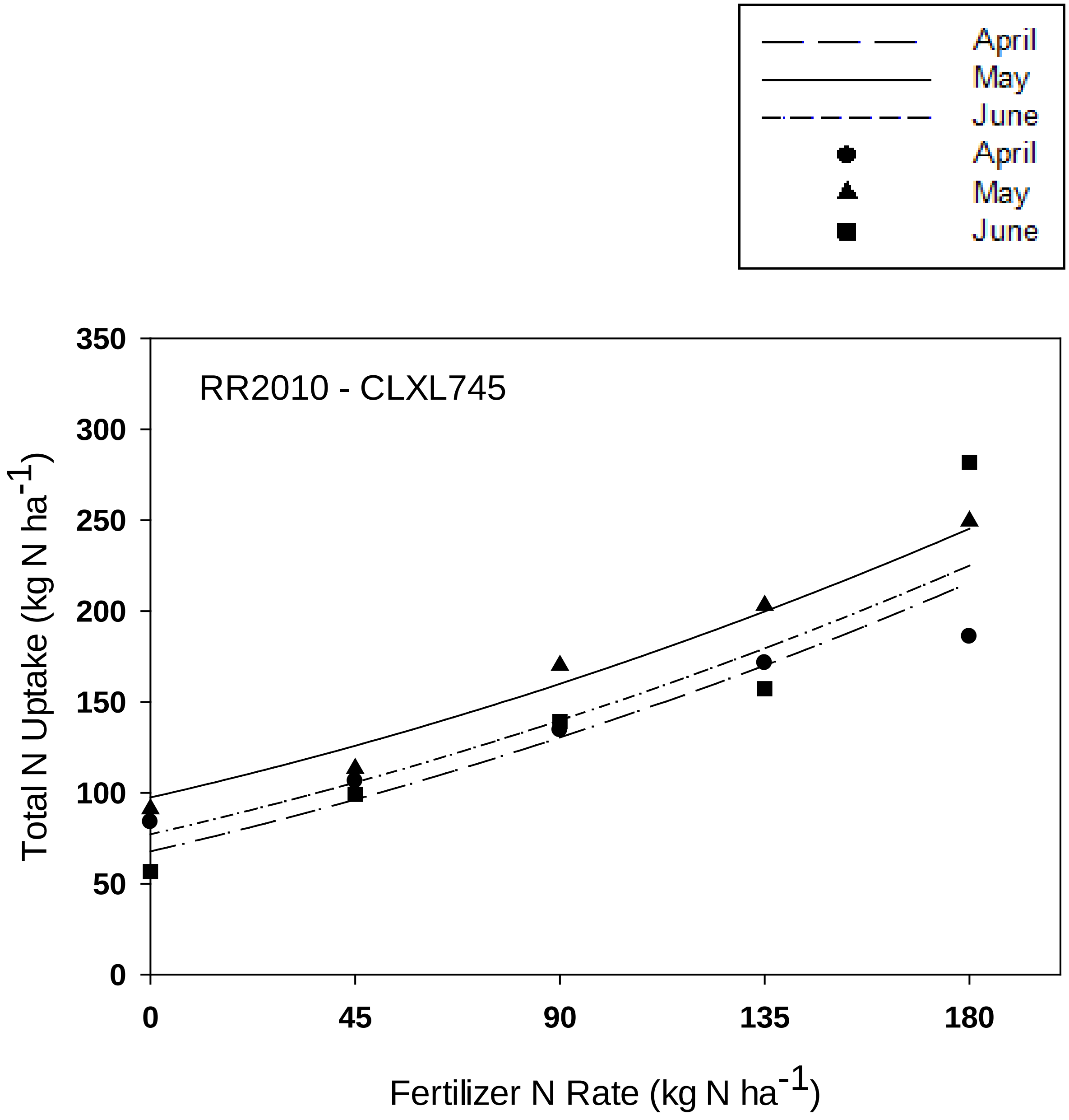

Figure 4.1E. Total nitrogen (TN) uptake of CLXL745 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

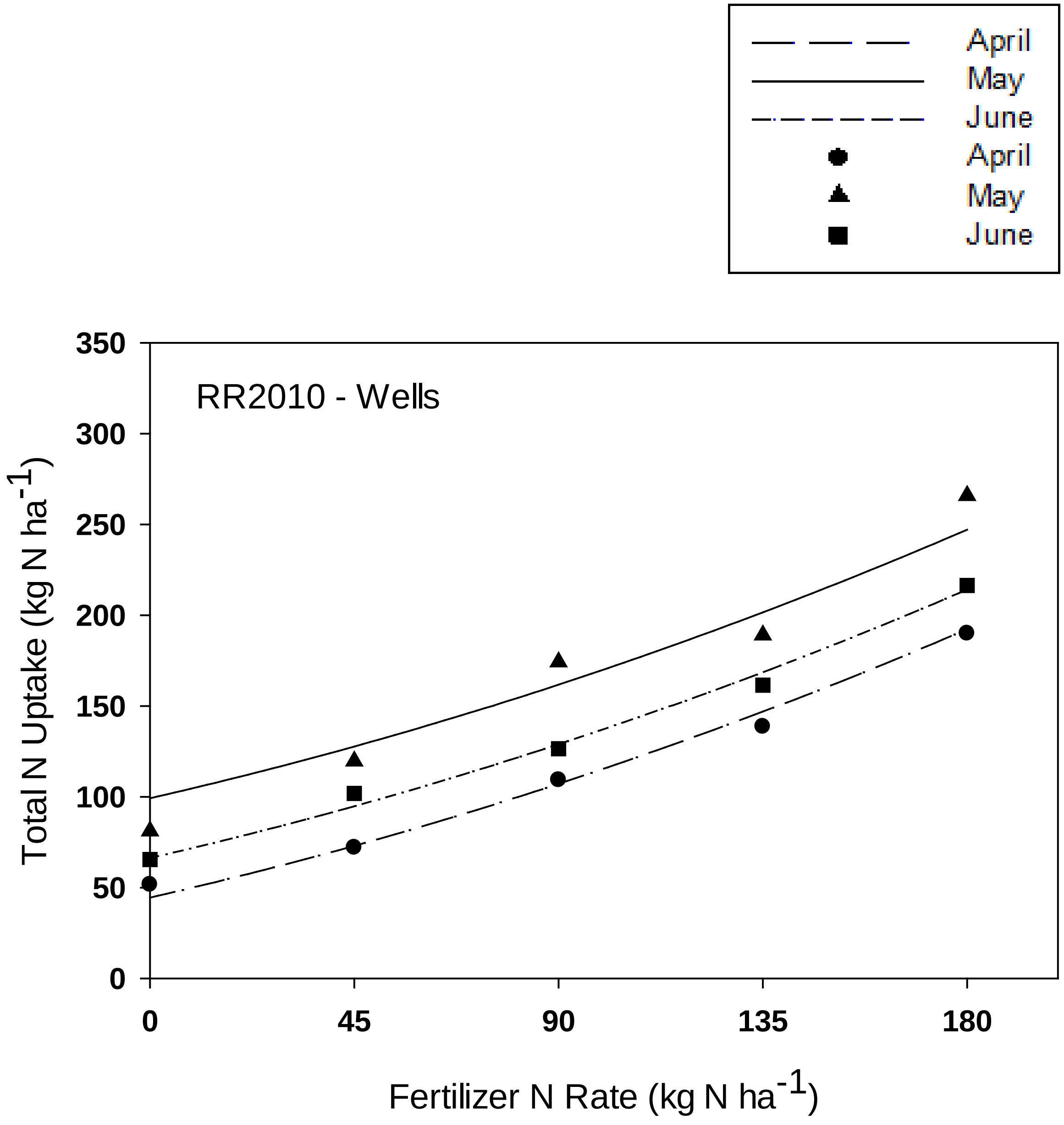

Figure 4.1F. Total nitrogen (TN) uptake of Wells at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

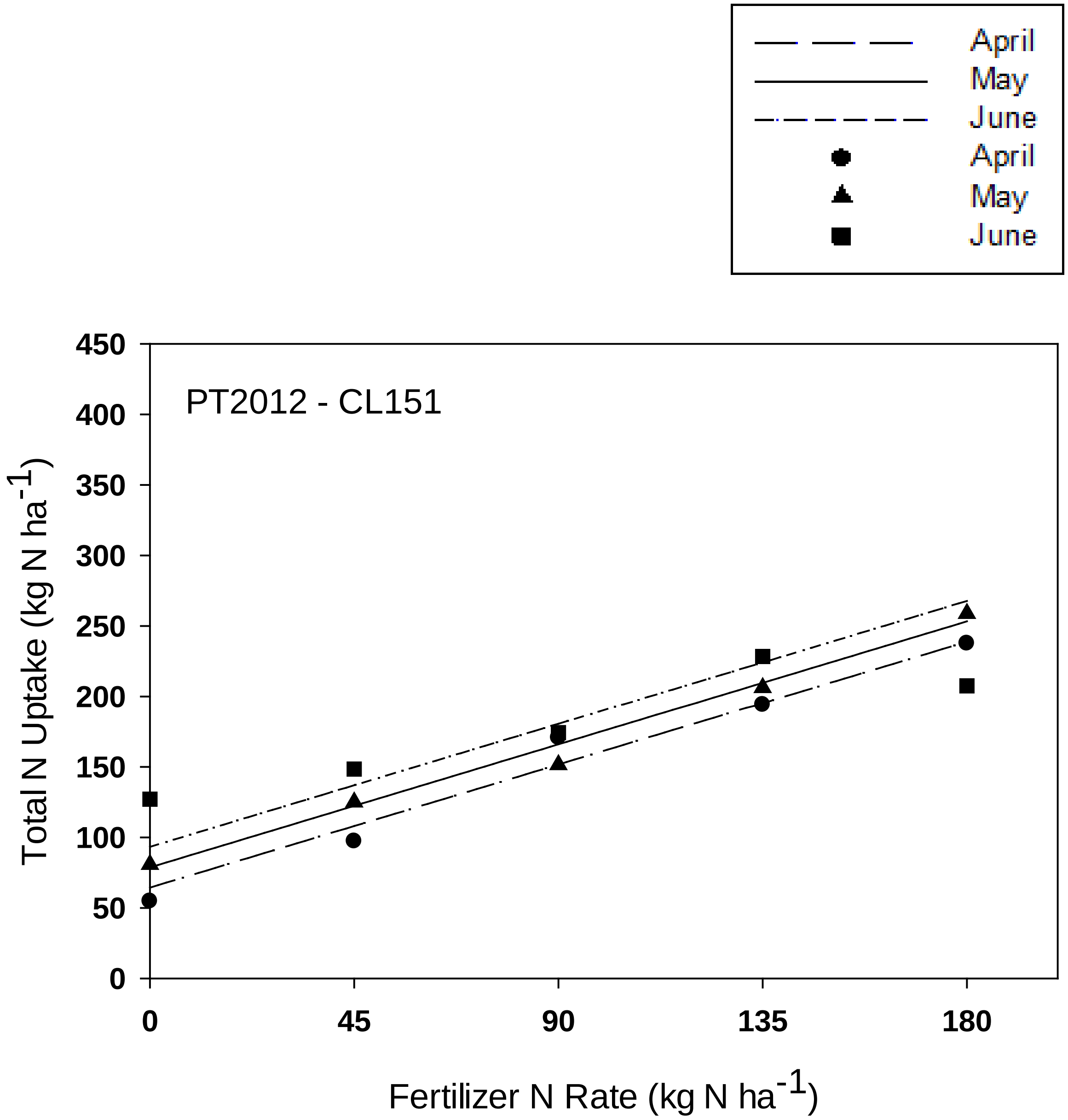

Figure 4.2A. Total nitrogen (TN) uptake of CL151 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

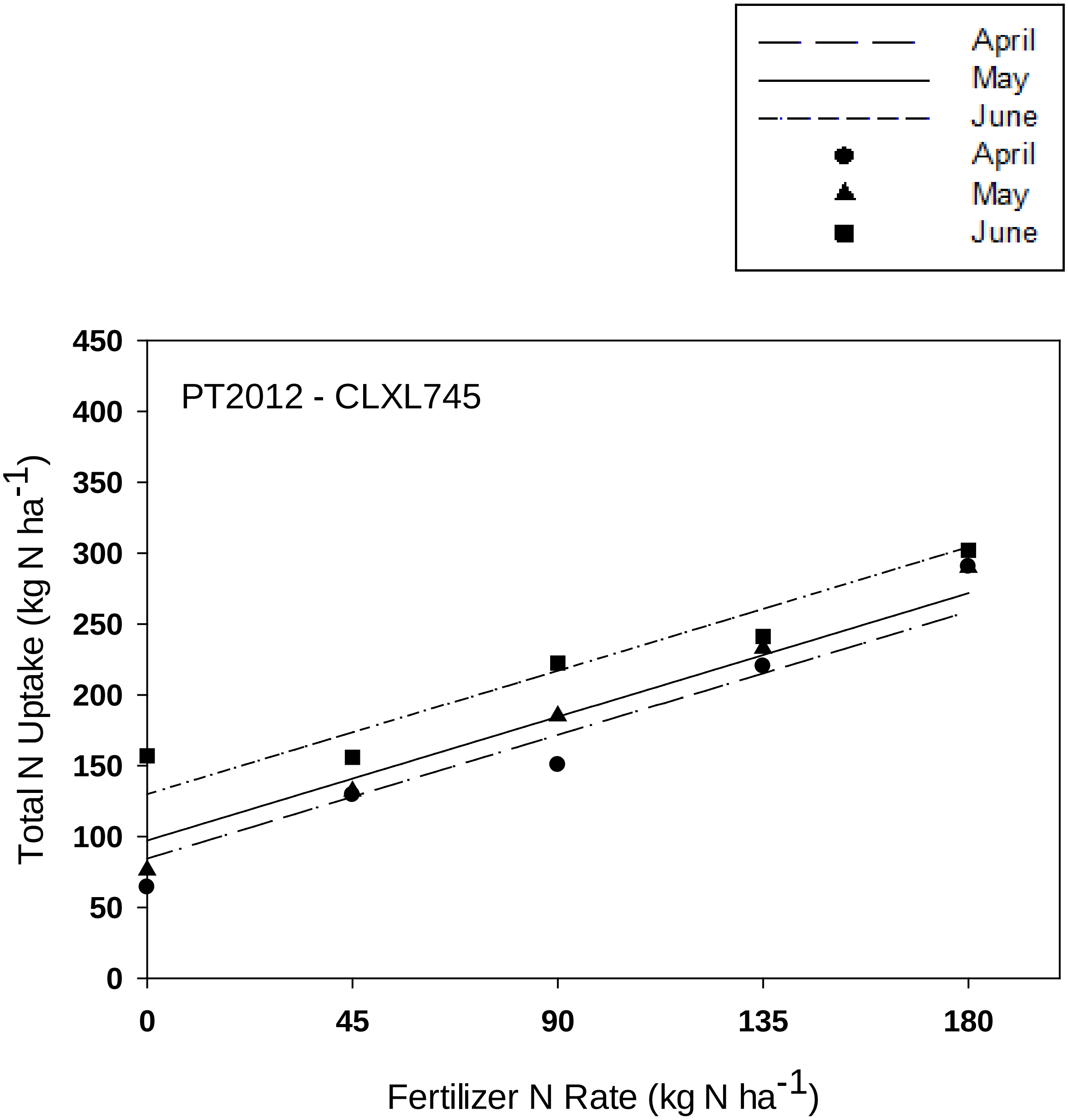

Figure 4.2B. Total nitrogen (TN) uptake of CLXL745 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

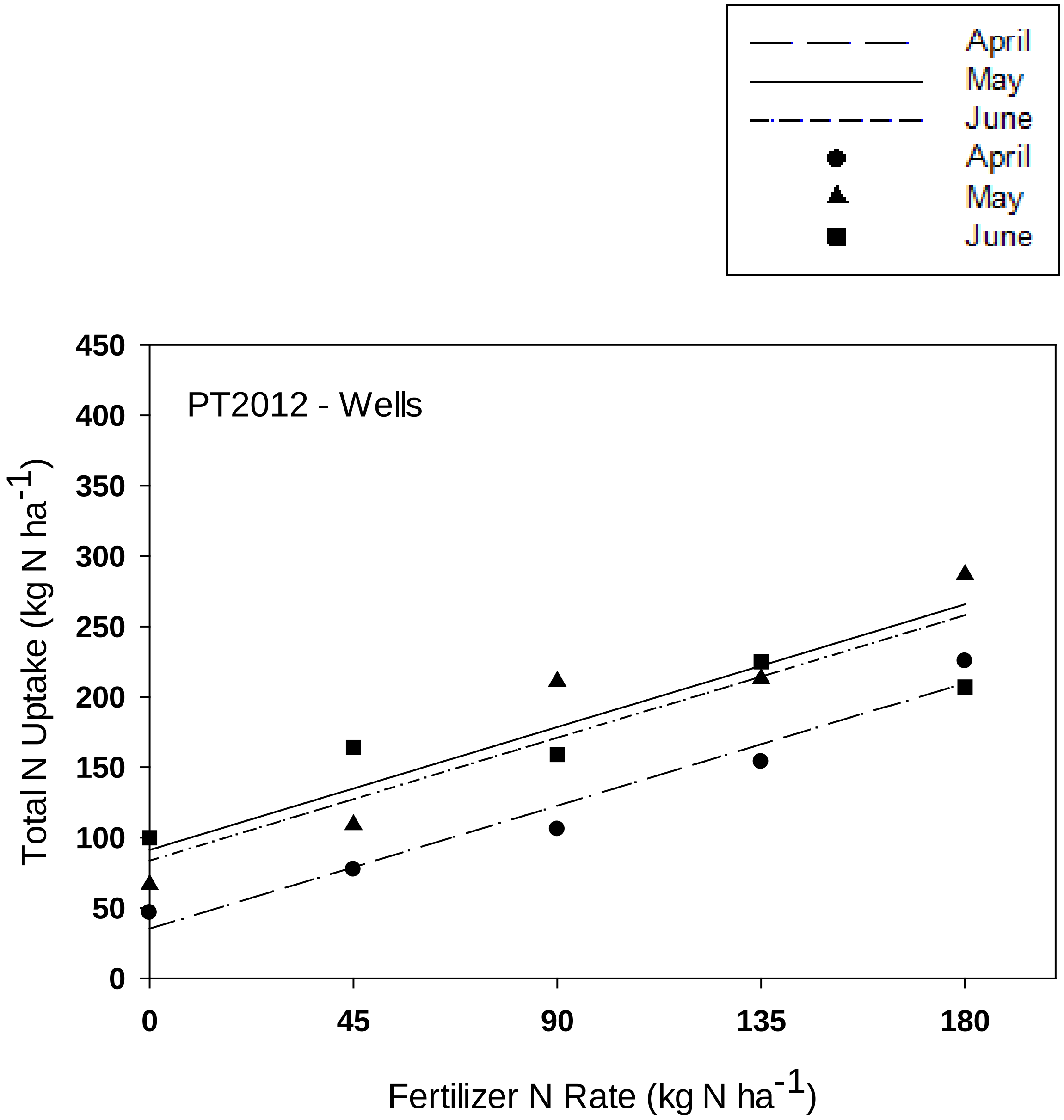

Figure 4.2C. Total nitrogen (TN) uptake of Wells at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

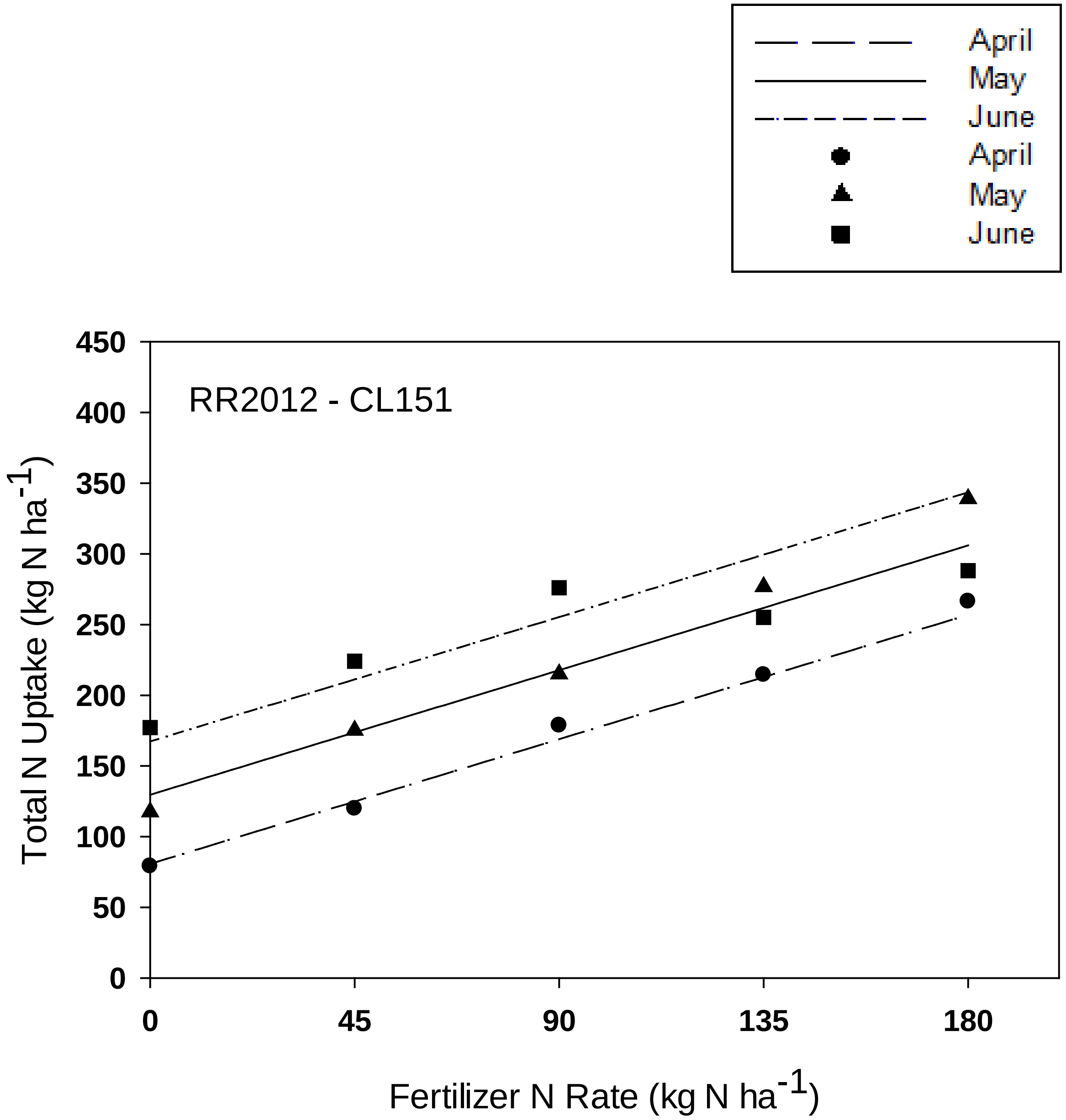

Figure 4.2D. Total nitrogen (TN) uptake of CL151 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

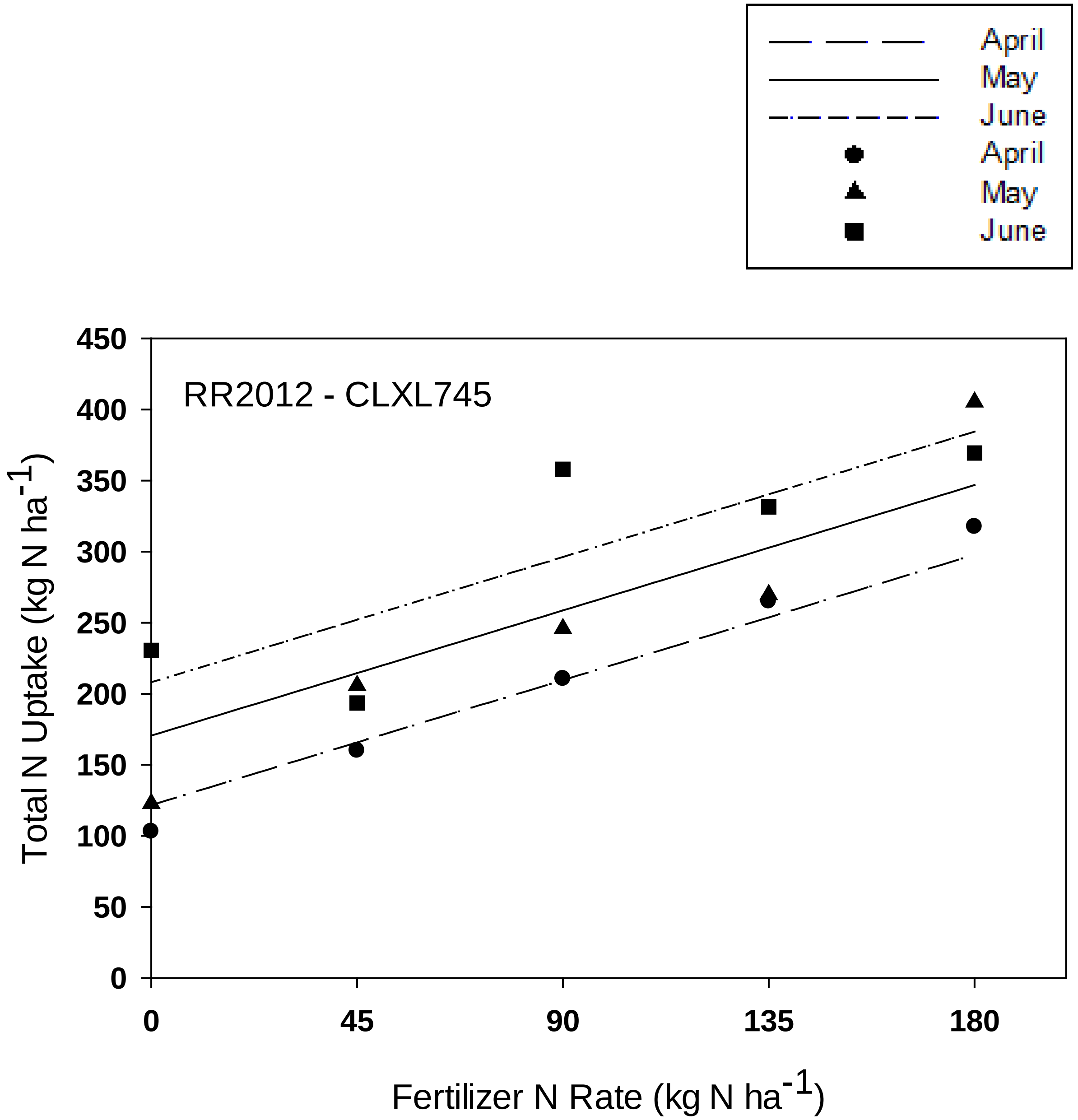

Figure 4.2E. Total nitrogen (TN) uptake of CLXL745 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

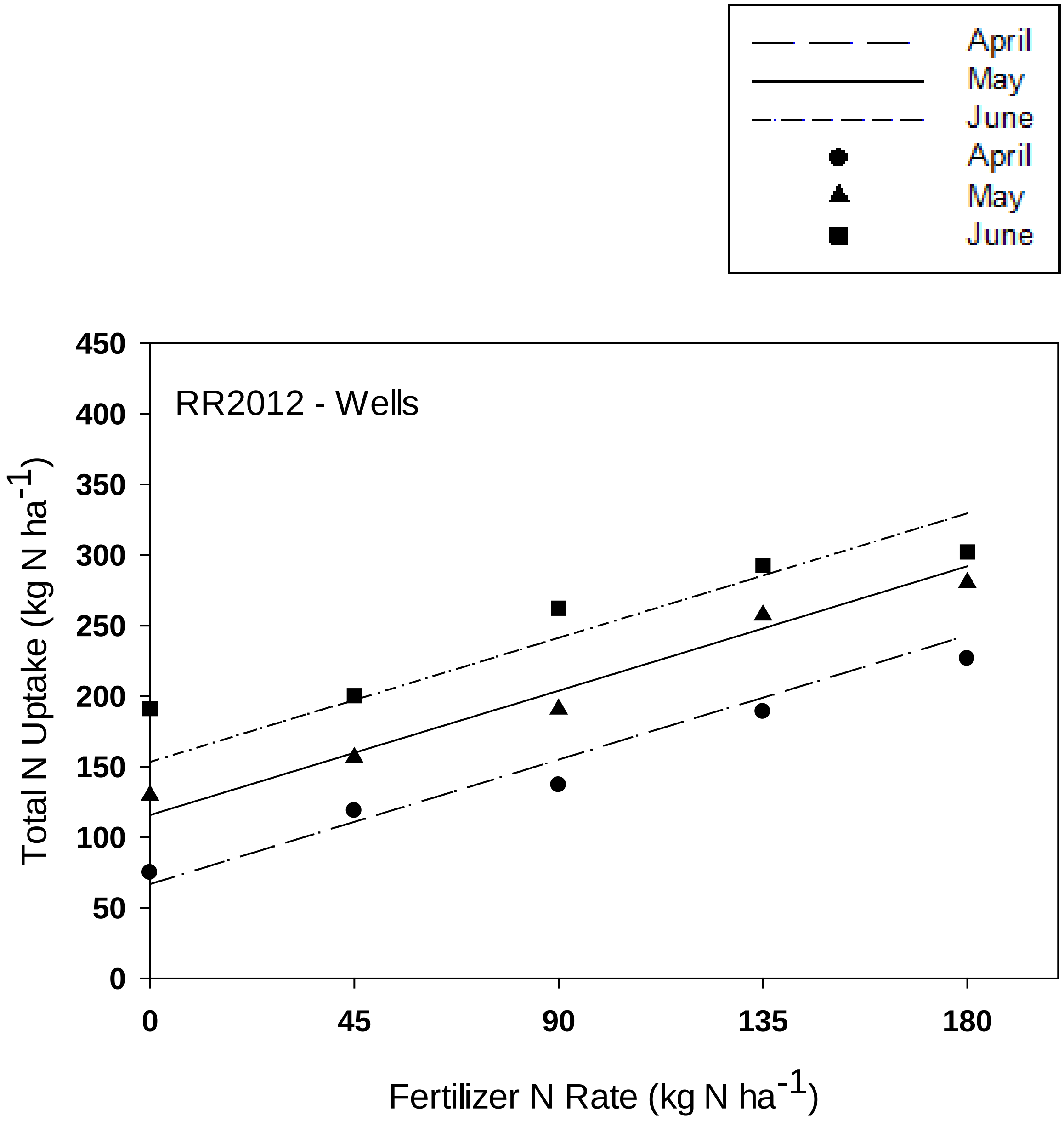

Figure 4.2F. Total nitrogen (TN) uptake of Wells at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

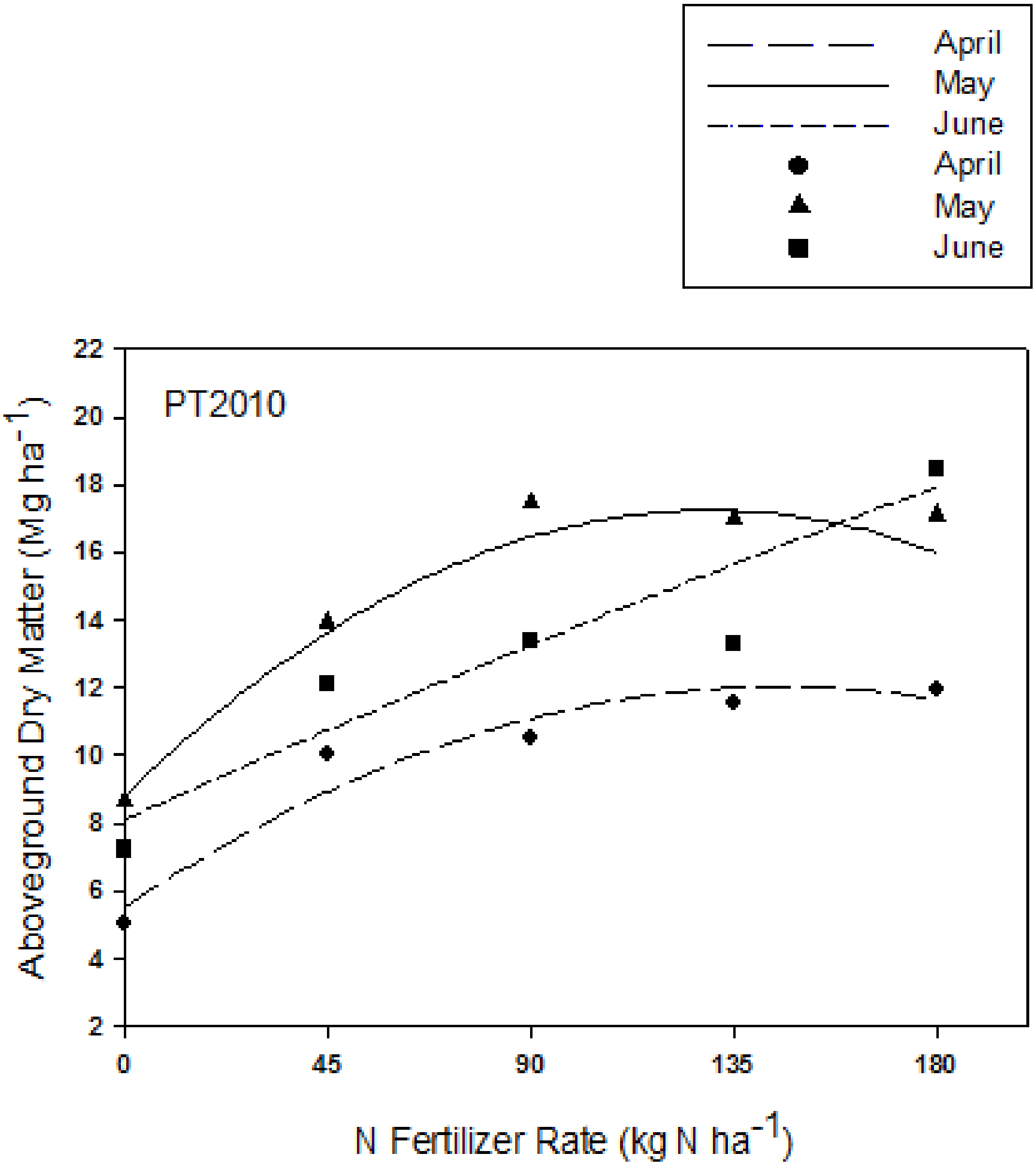

Figure 4.3. Aboveground dry matter of rice at 50% heading in response to fertilizer N rate as affected by seeding date (April, May or June) at Pine Tree Research Station (PTRS) in 2010 (PT2010).

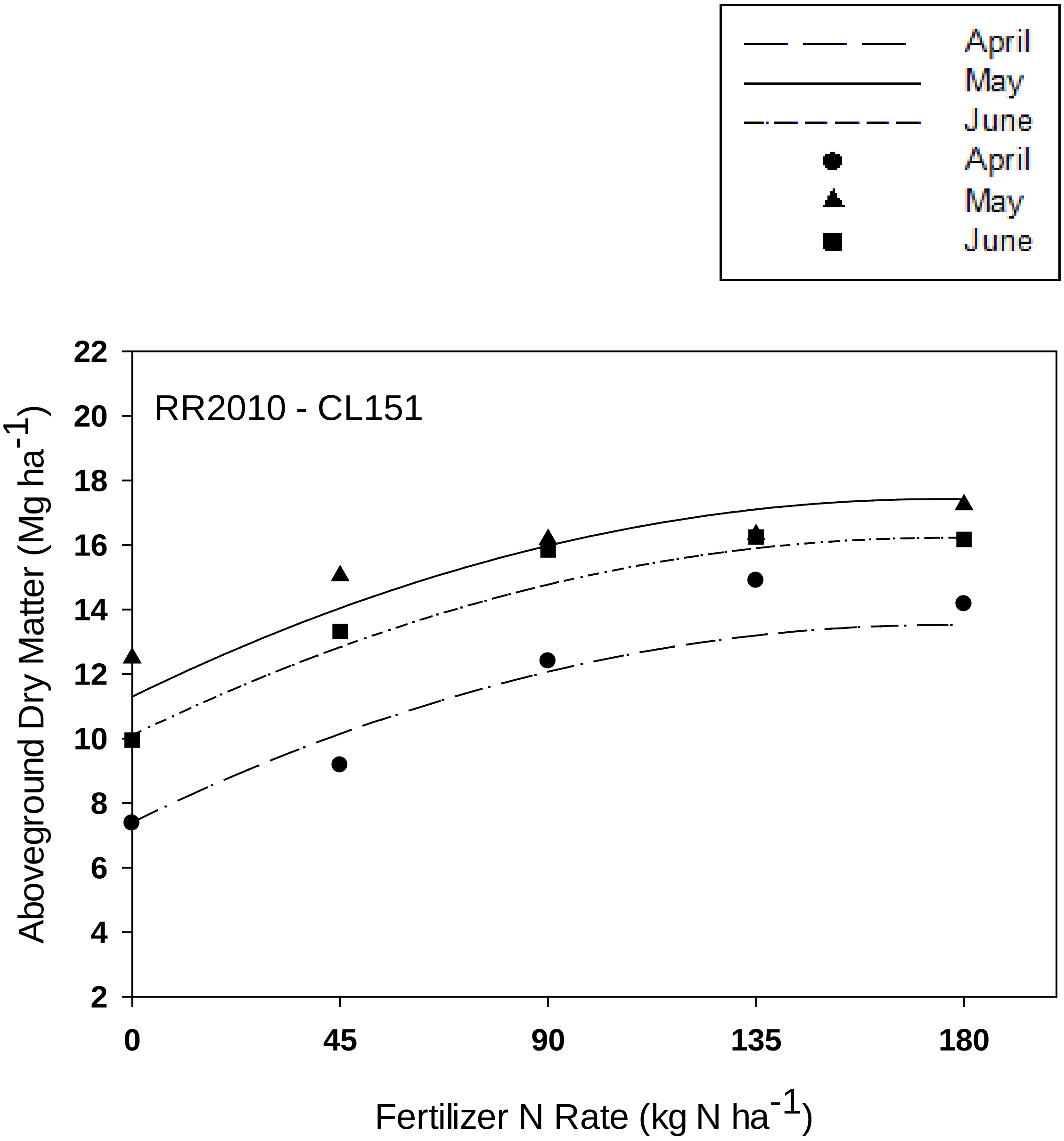

Figure 4.4A. Aboveground dry matter of CL151 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

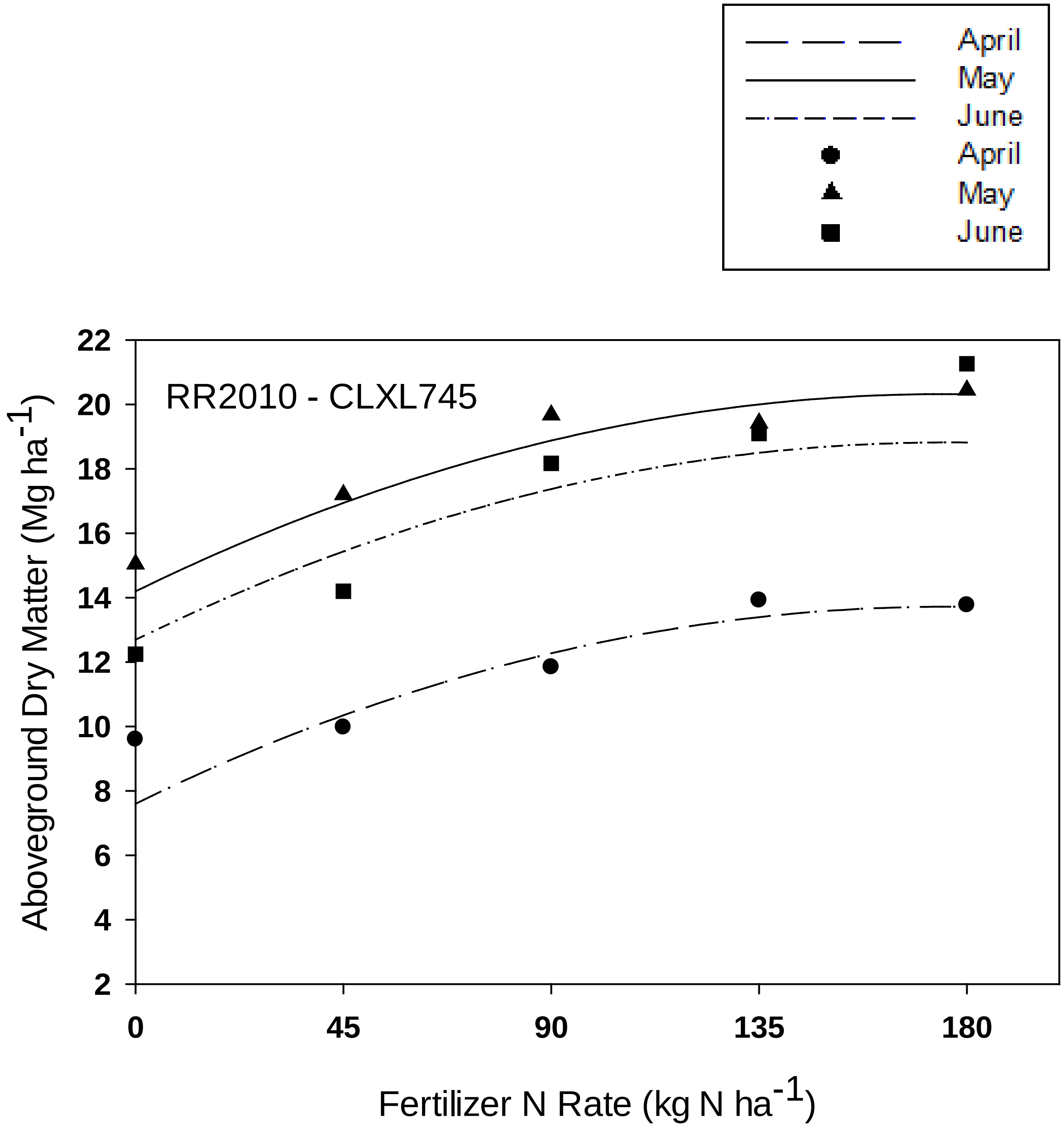

Figure 4.4B. Aboveground dry matter of CLXL745 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

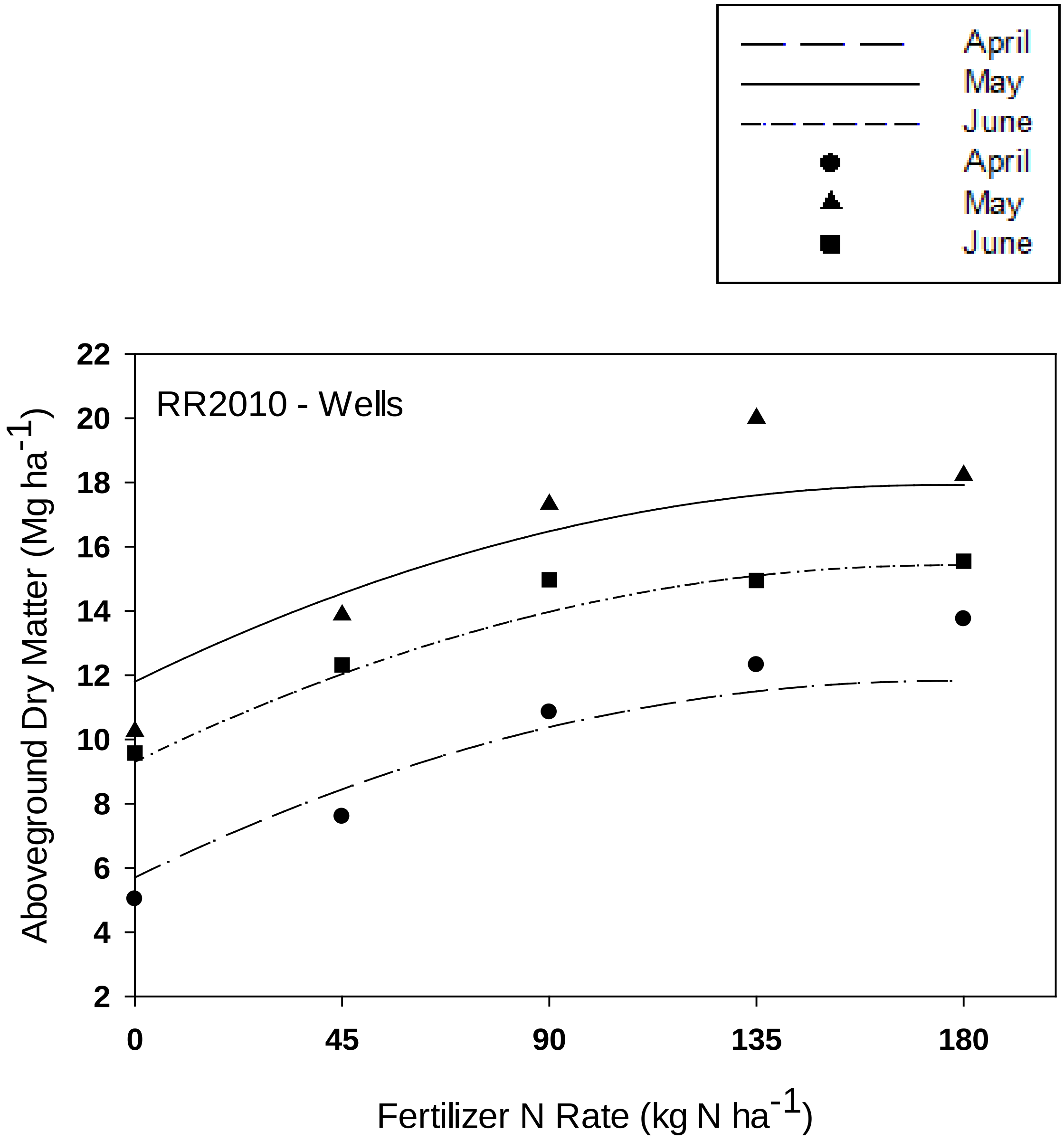

Figure 4.4C. Aboveground dry matter of Wells at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

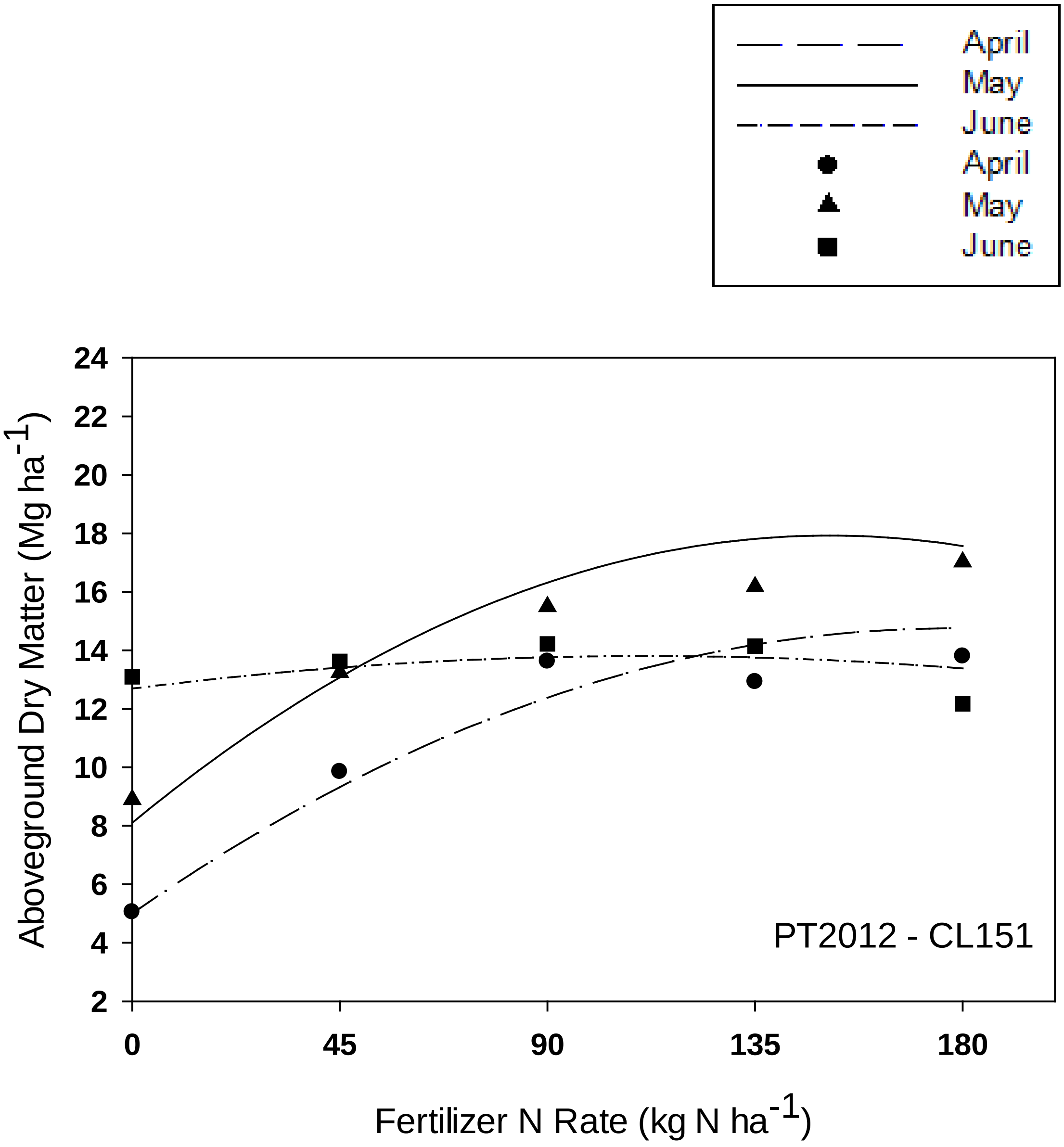

Figure 4.5A. Aboveground dry matter of CL151 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

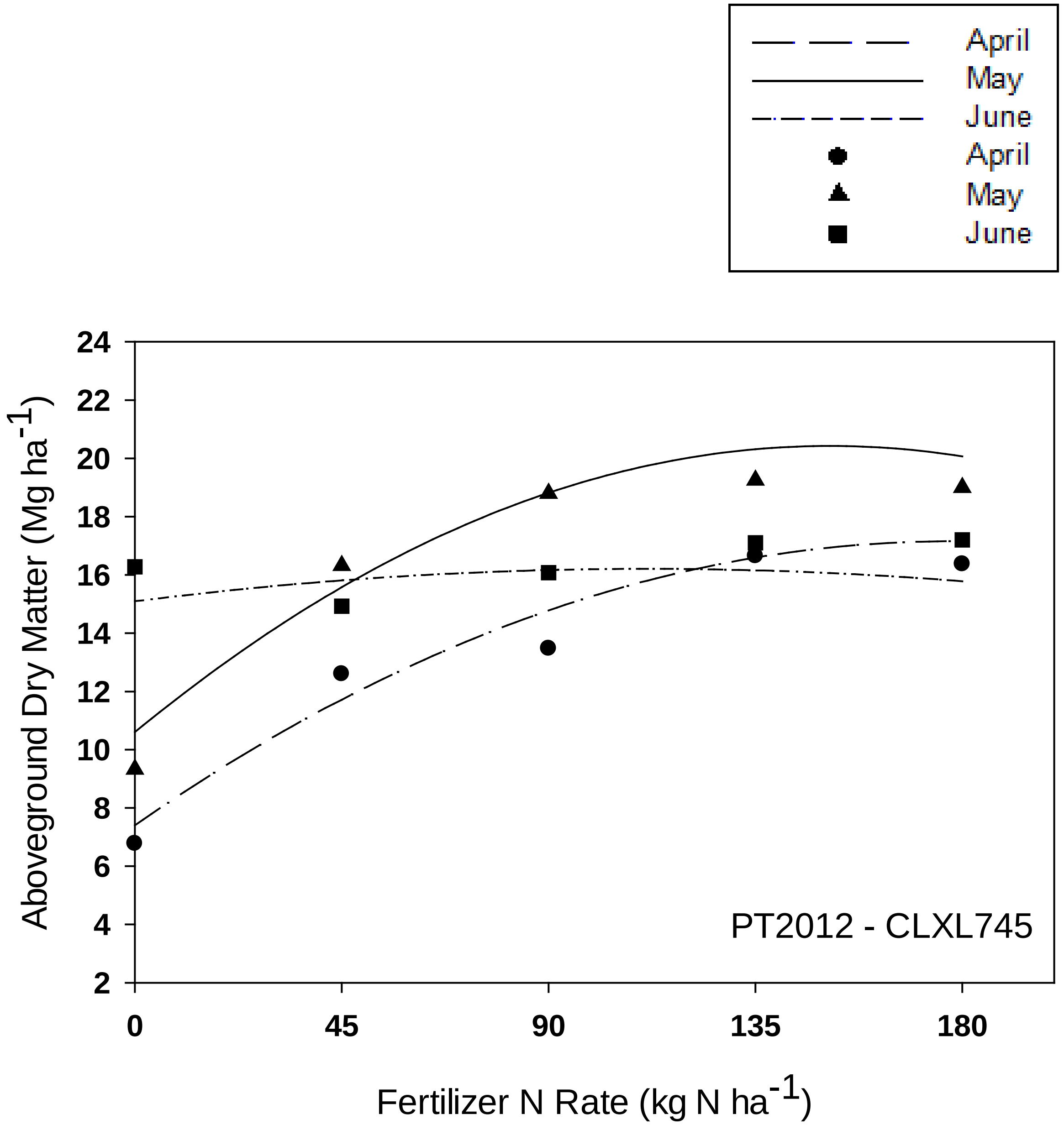

Figure 4.5B. Aboveground dry matter of CLXL745 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

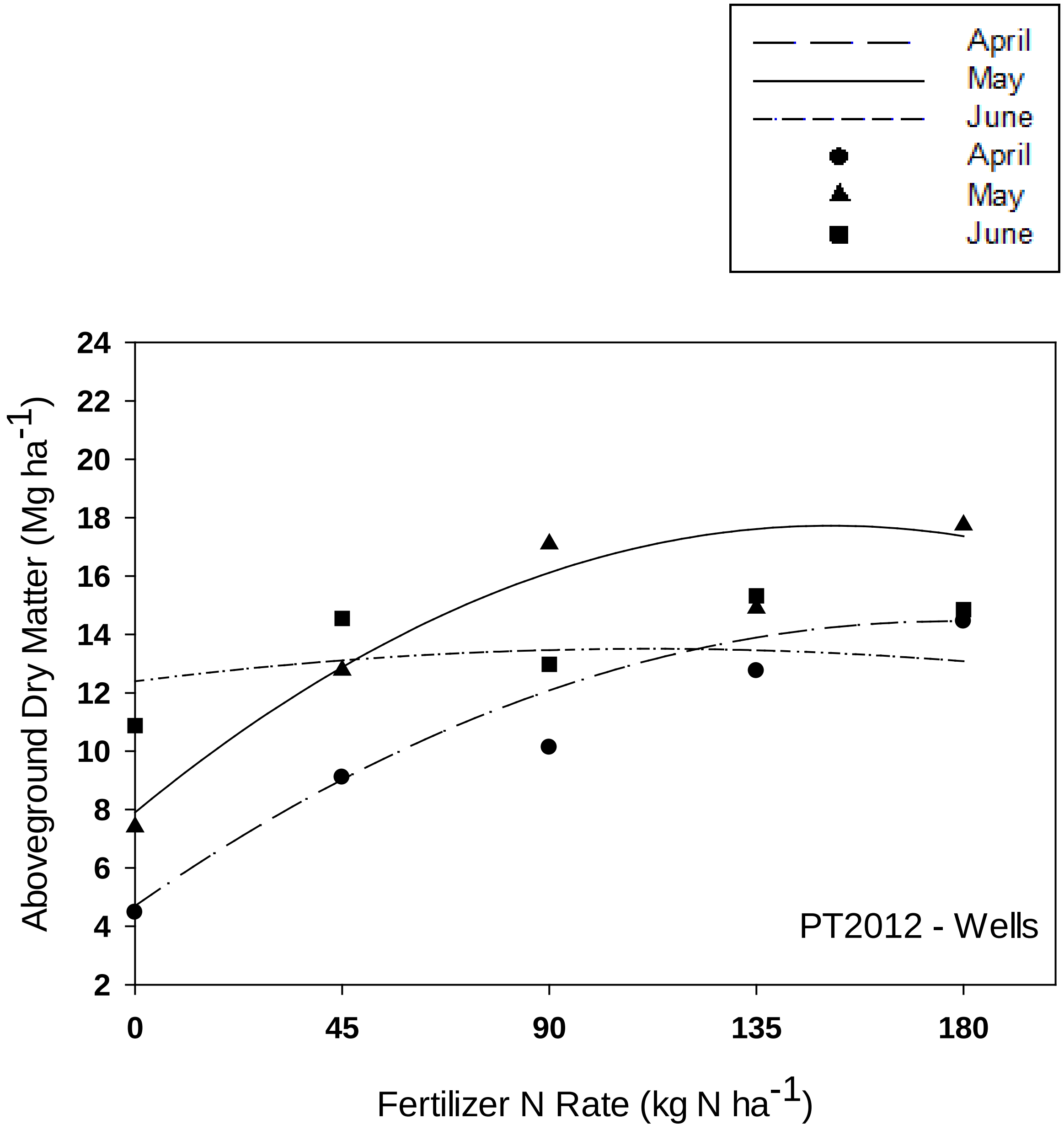

Figure 4.5C. Aboveground dry matter of Wells at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

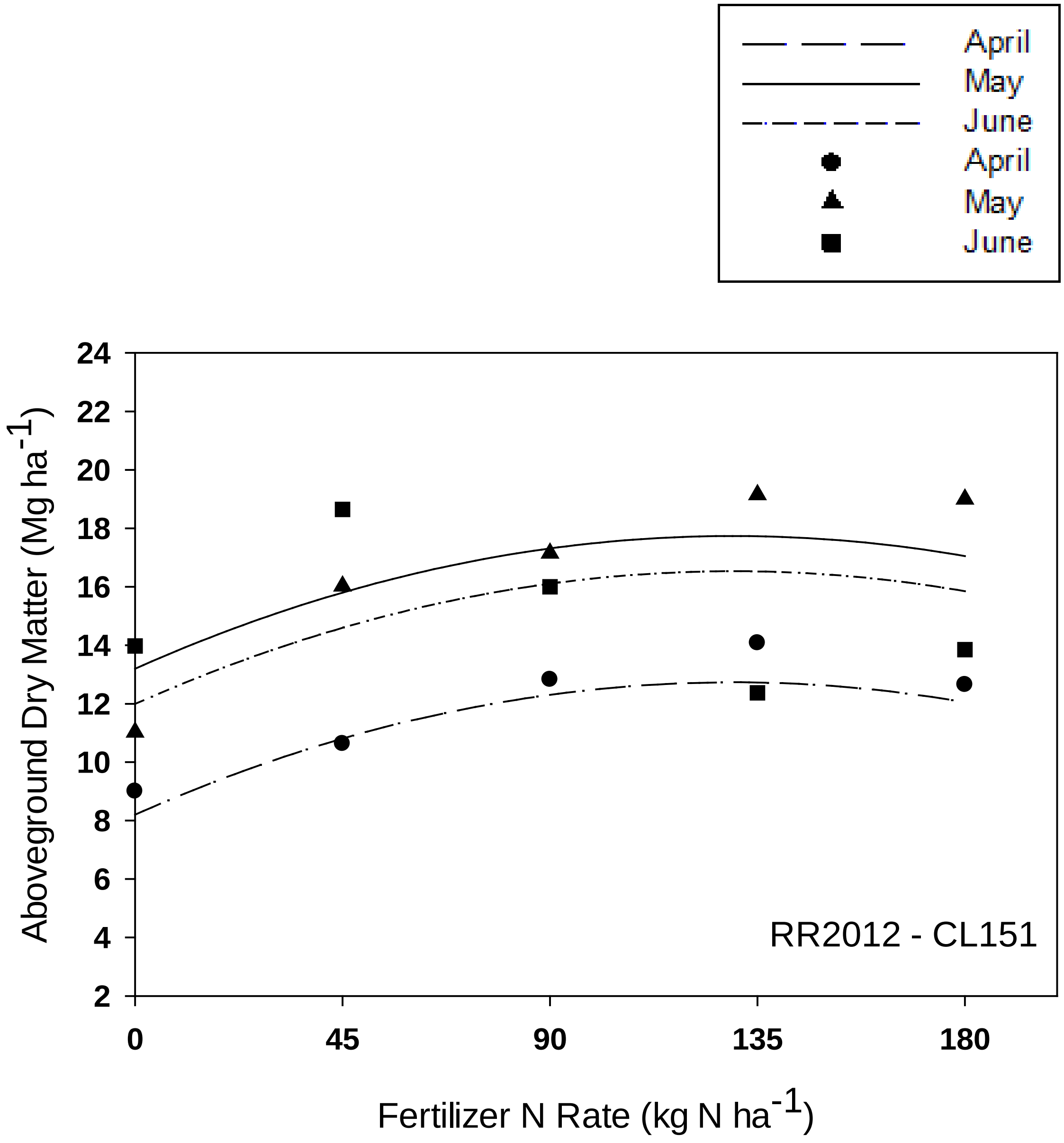

Figure 4.5D. Aboveground dry matter of CL151 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

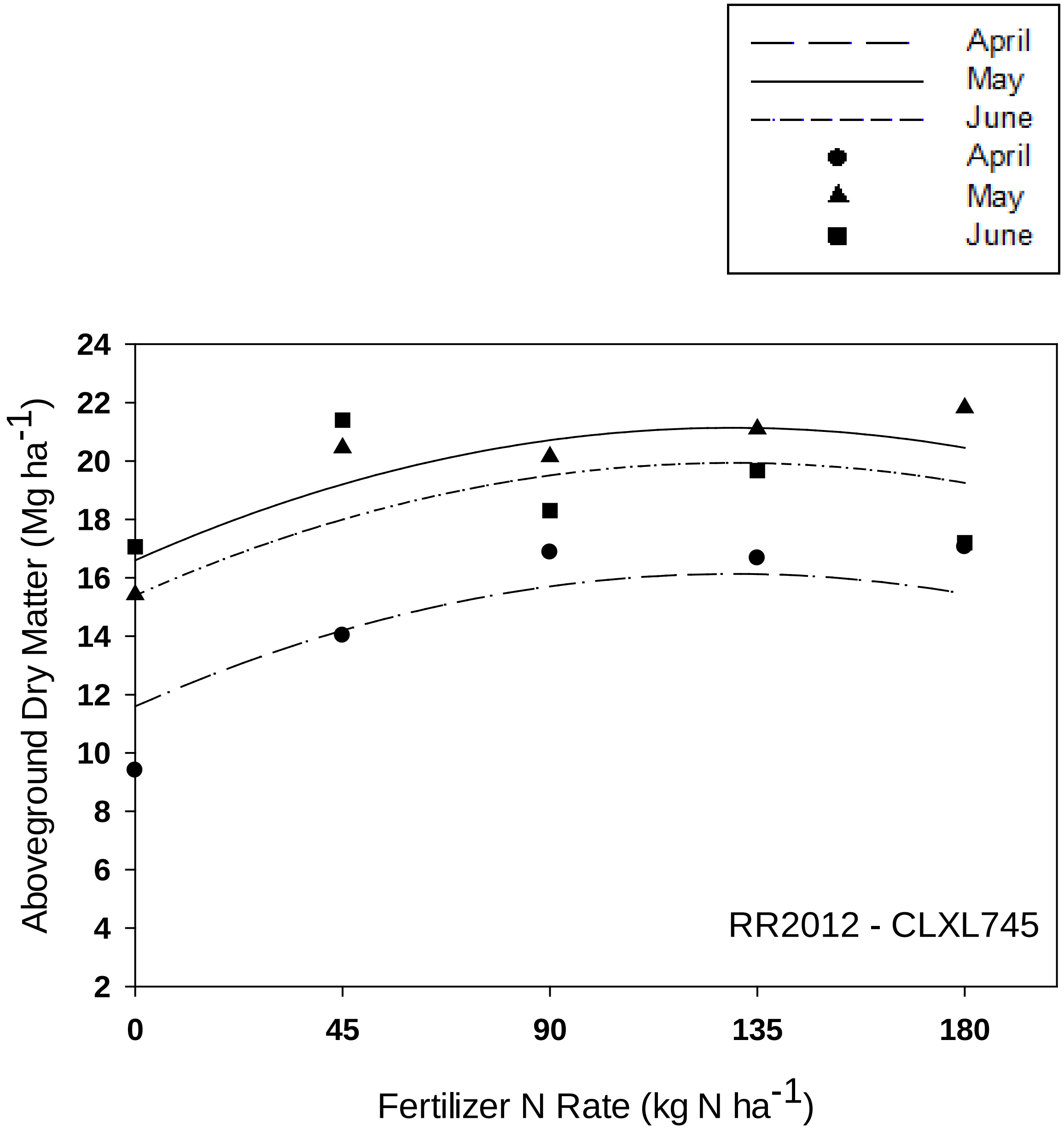

Figure 4.5E. Aboveground dry matter of CLXL745 at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

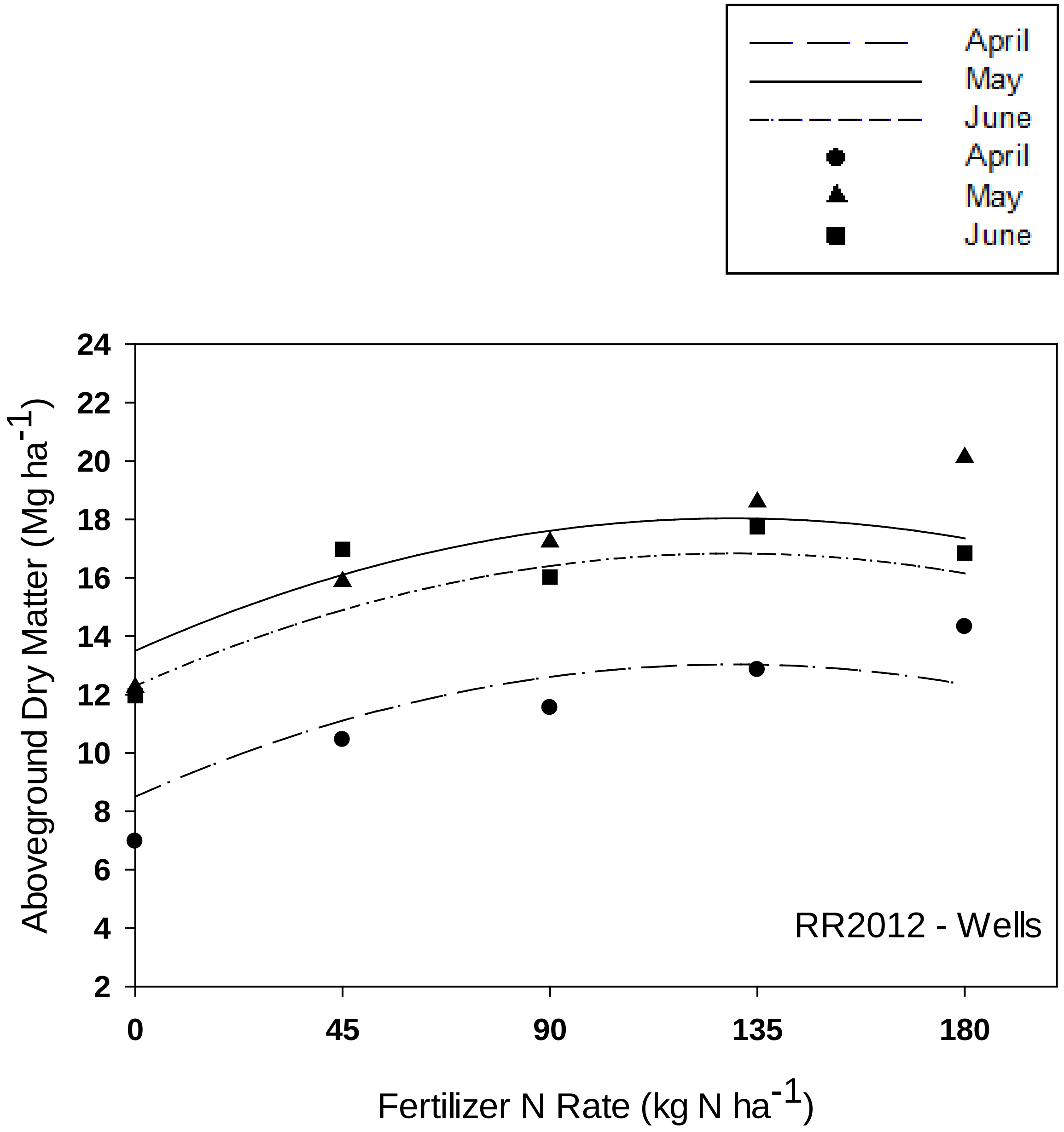

Figure 4.5F. Aboveground dry matter of Wells at 50% heading in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

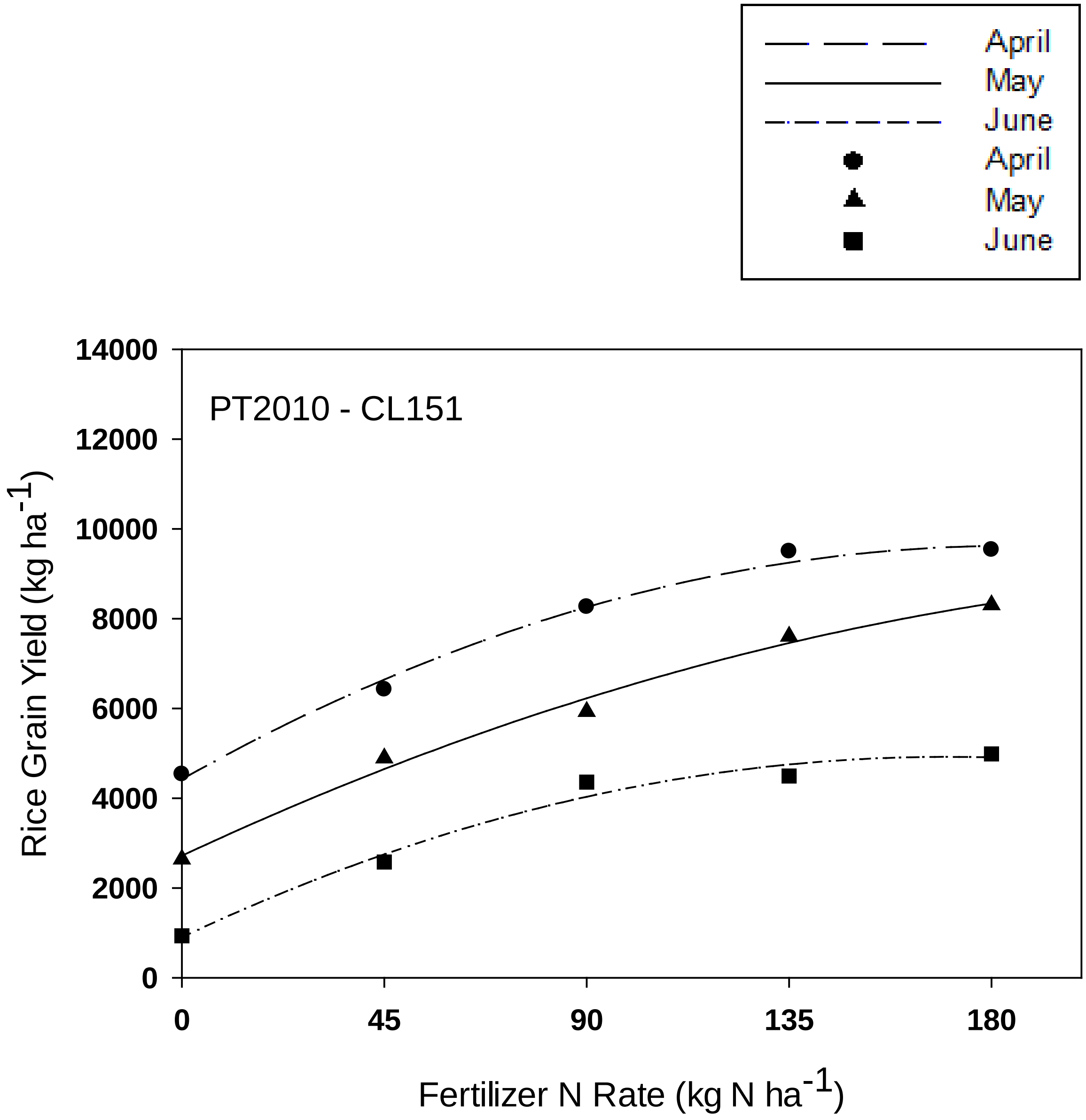

Figure 4.6A. Grain yield of CL151 in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2010 (PT2010).

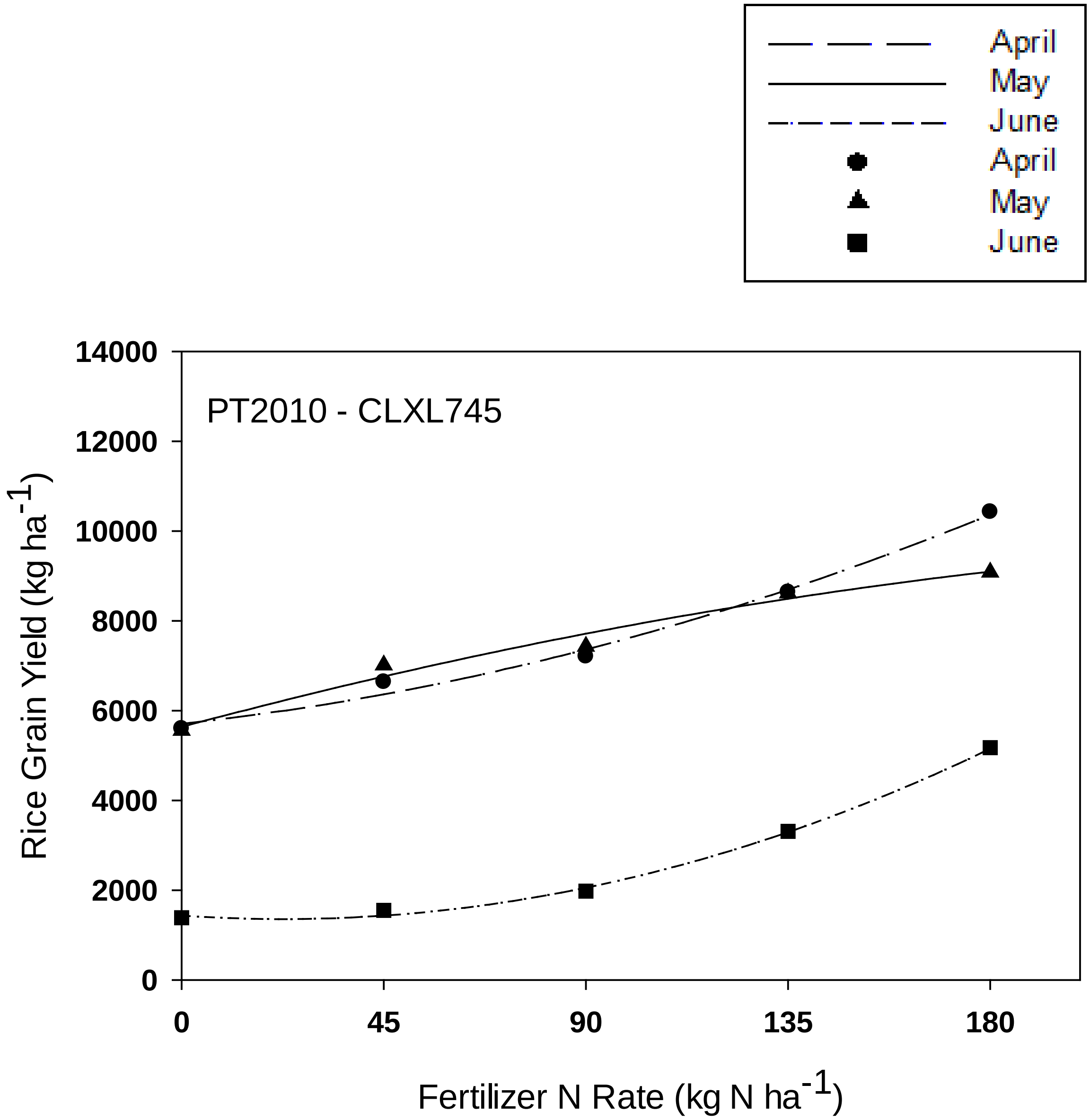

Figure 4.6B. Grain yield of CLXL745 in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2010 (PT2010).

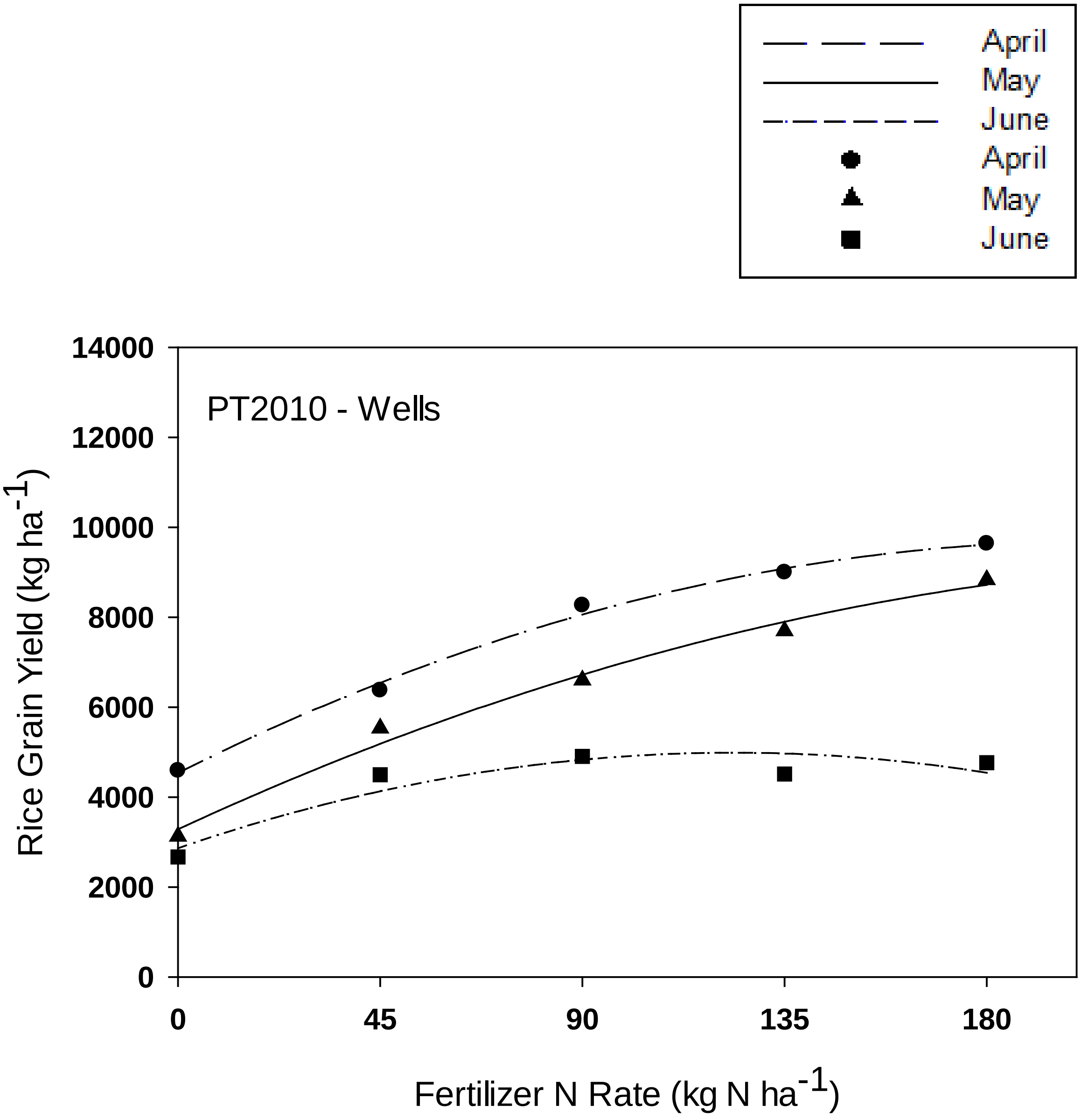

Figure 4.6C. Grain yield of Wells in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2010 (PT2010).

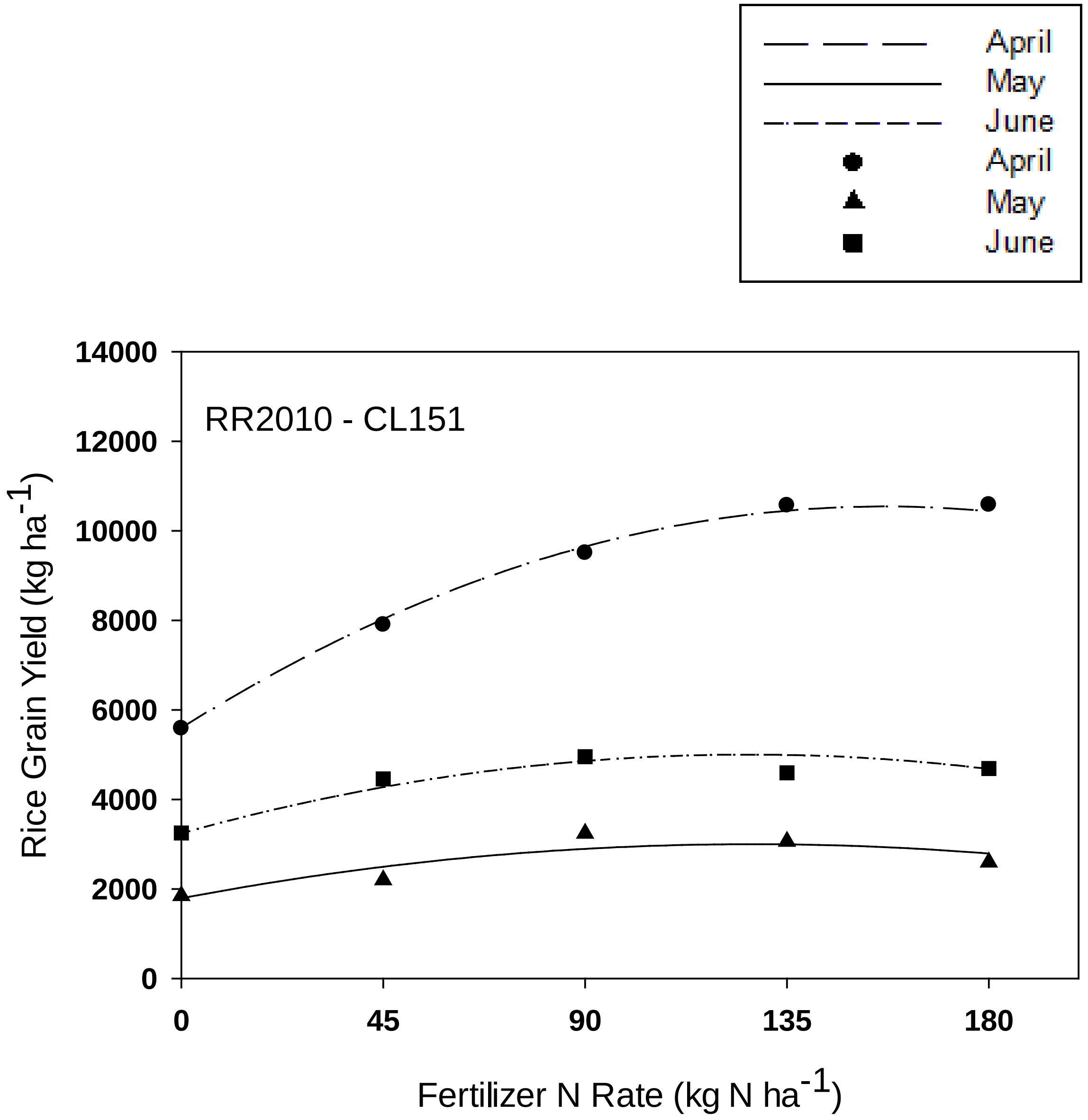

Figure 4.6D. Grain yield of CL151 in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

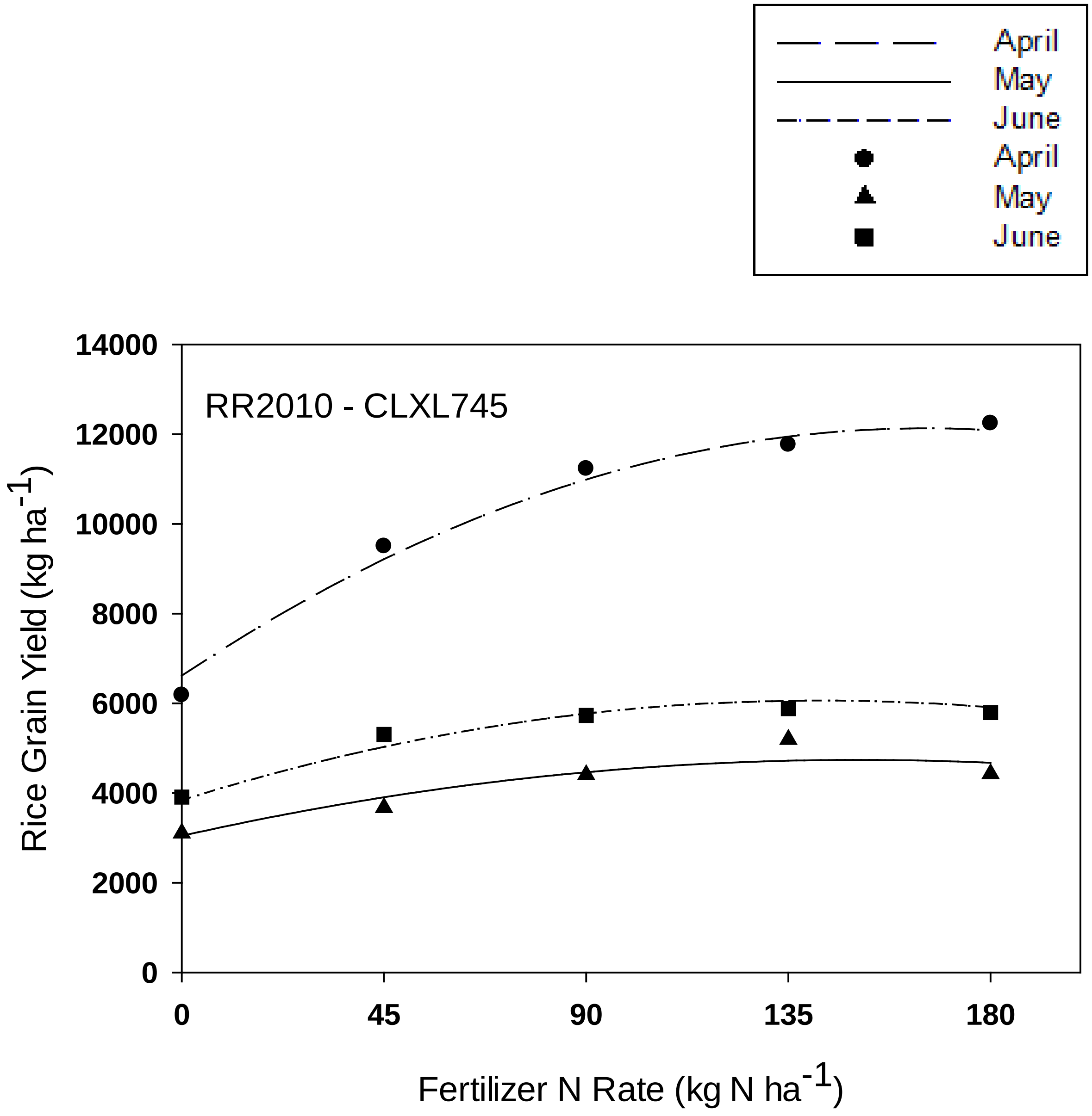

Figure 4.6E. Grain yield of CLXL745 in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

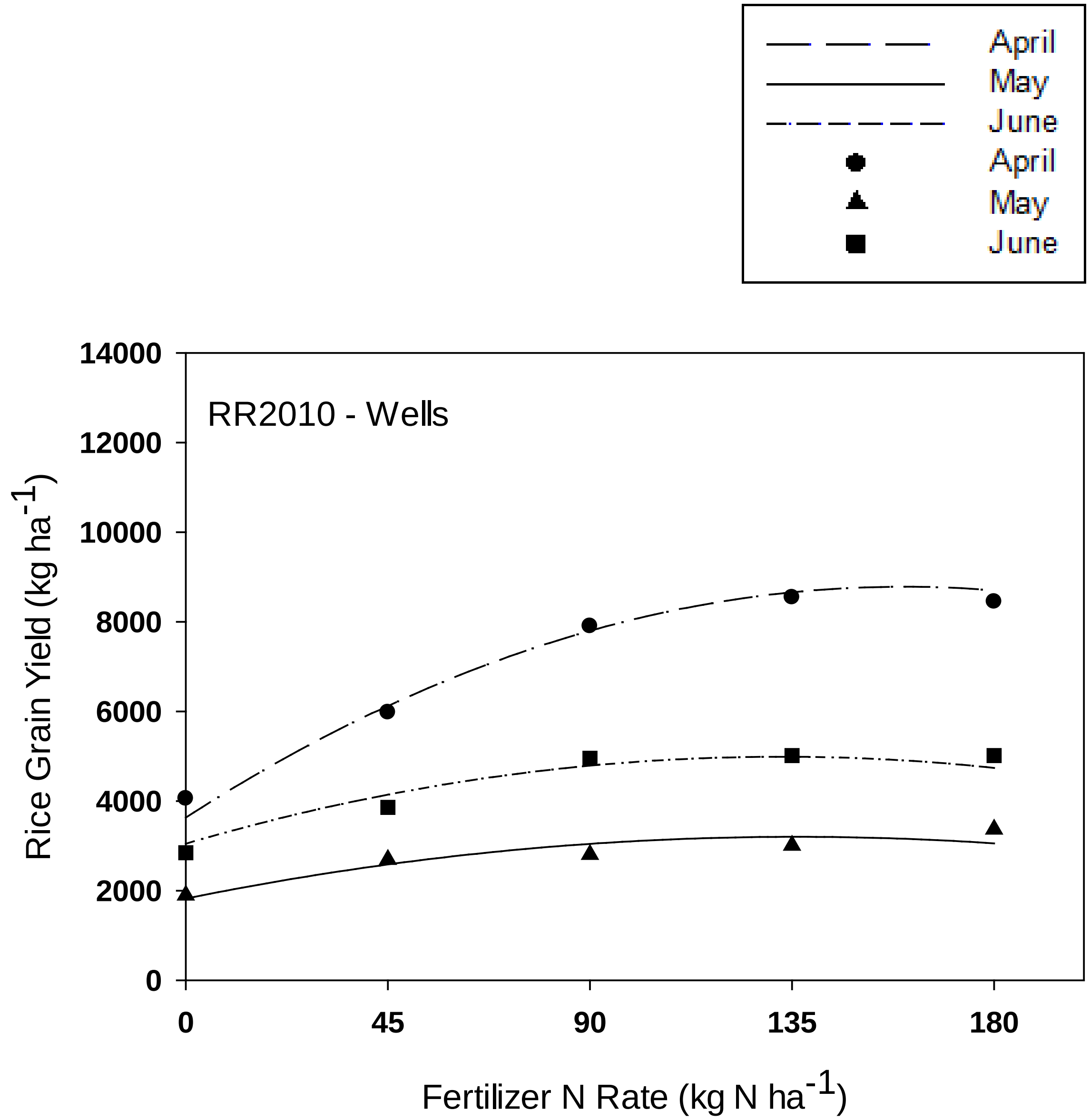

Figure 4.6F. Grain yield of Wells in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2010 (RR2010).

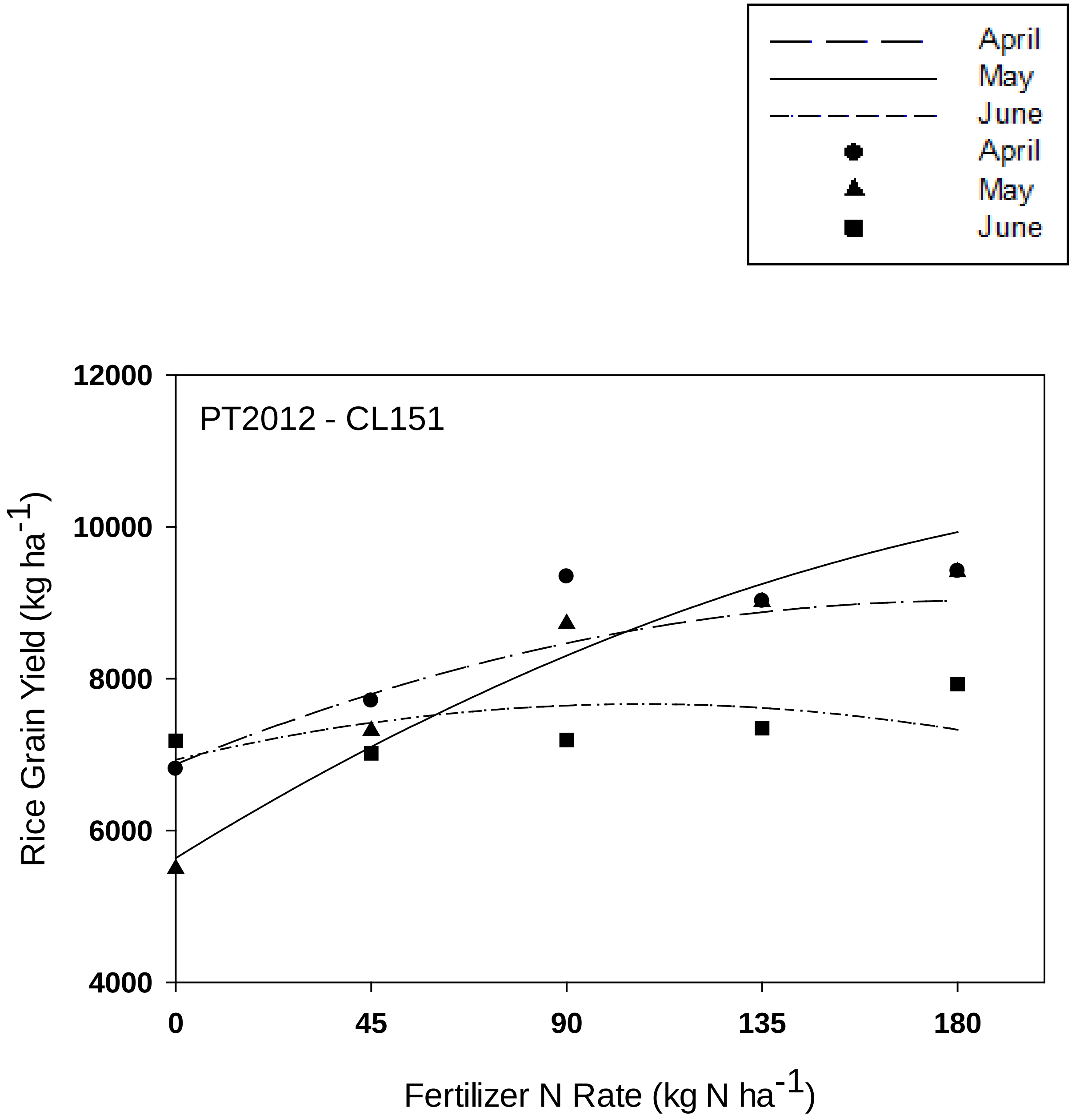

Figure 4.7A. Grain yield of CL151 in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

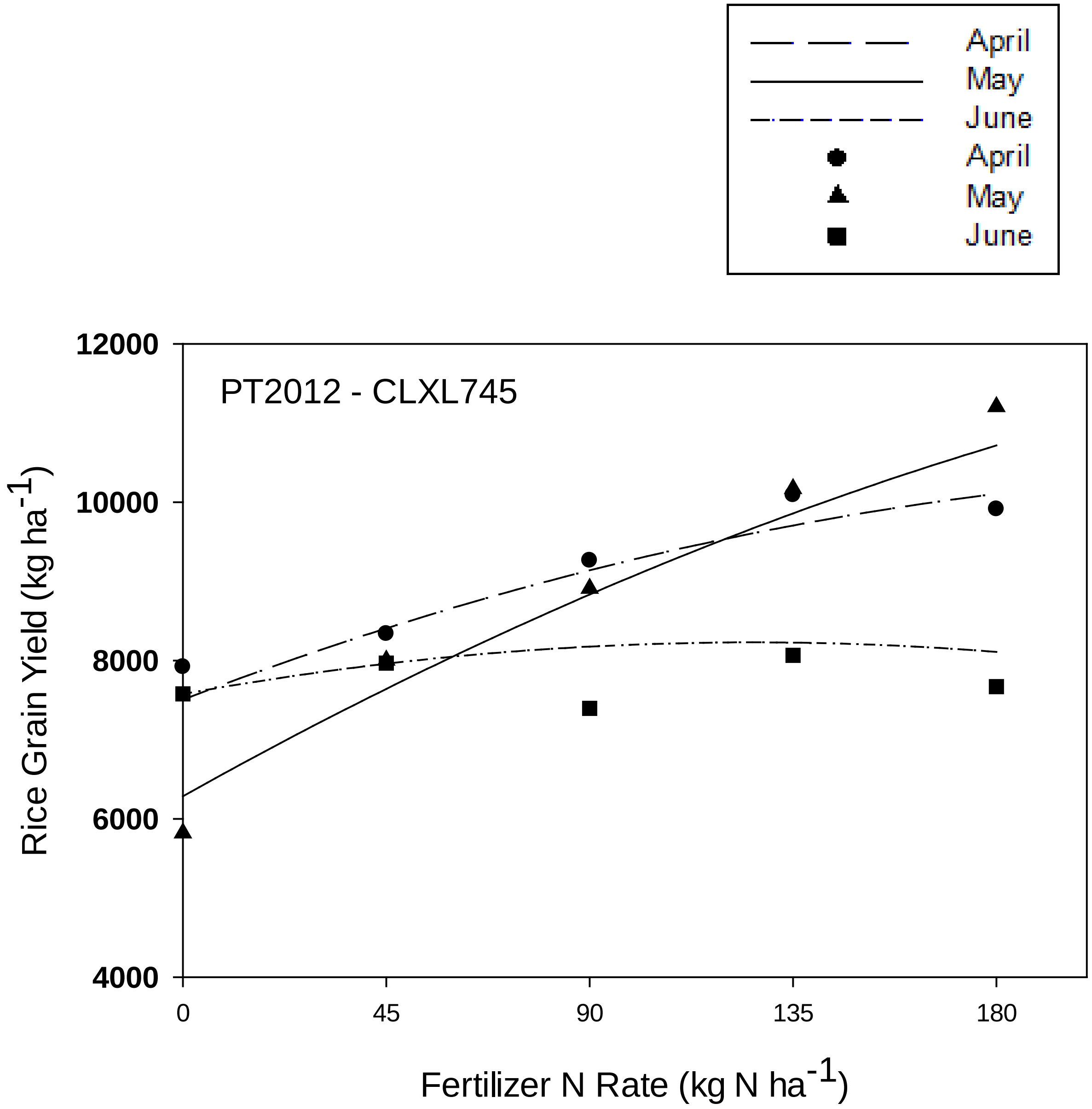

Figure 4.7B. Grain yield of CLXL745 in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

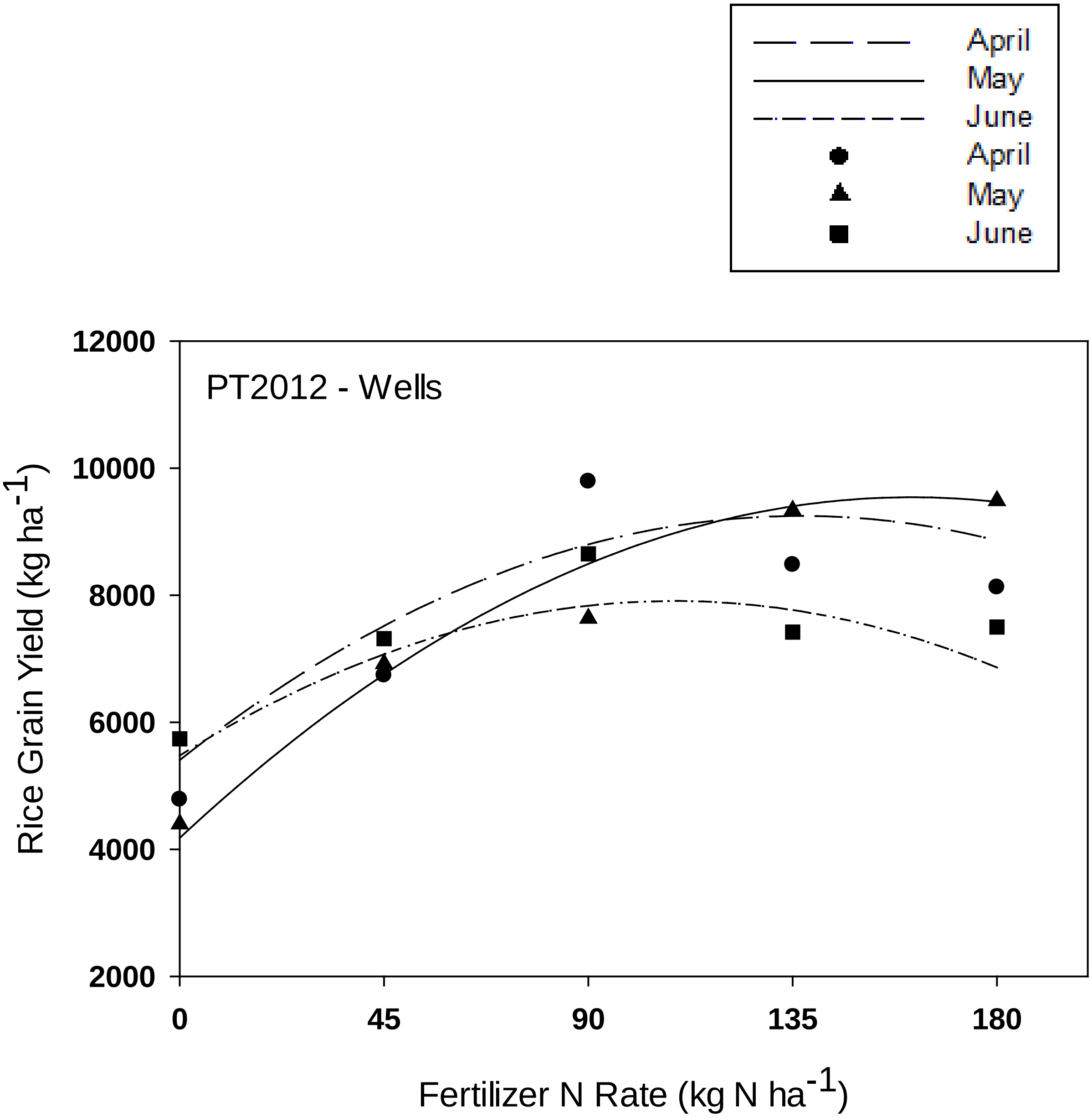

Figure 4.7C. Grain yield of Wells in response to fertilizer N rate when seeded in April, May, or June at Pine Tree Research Station (PTRS) in 2012 (PT2012).

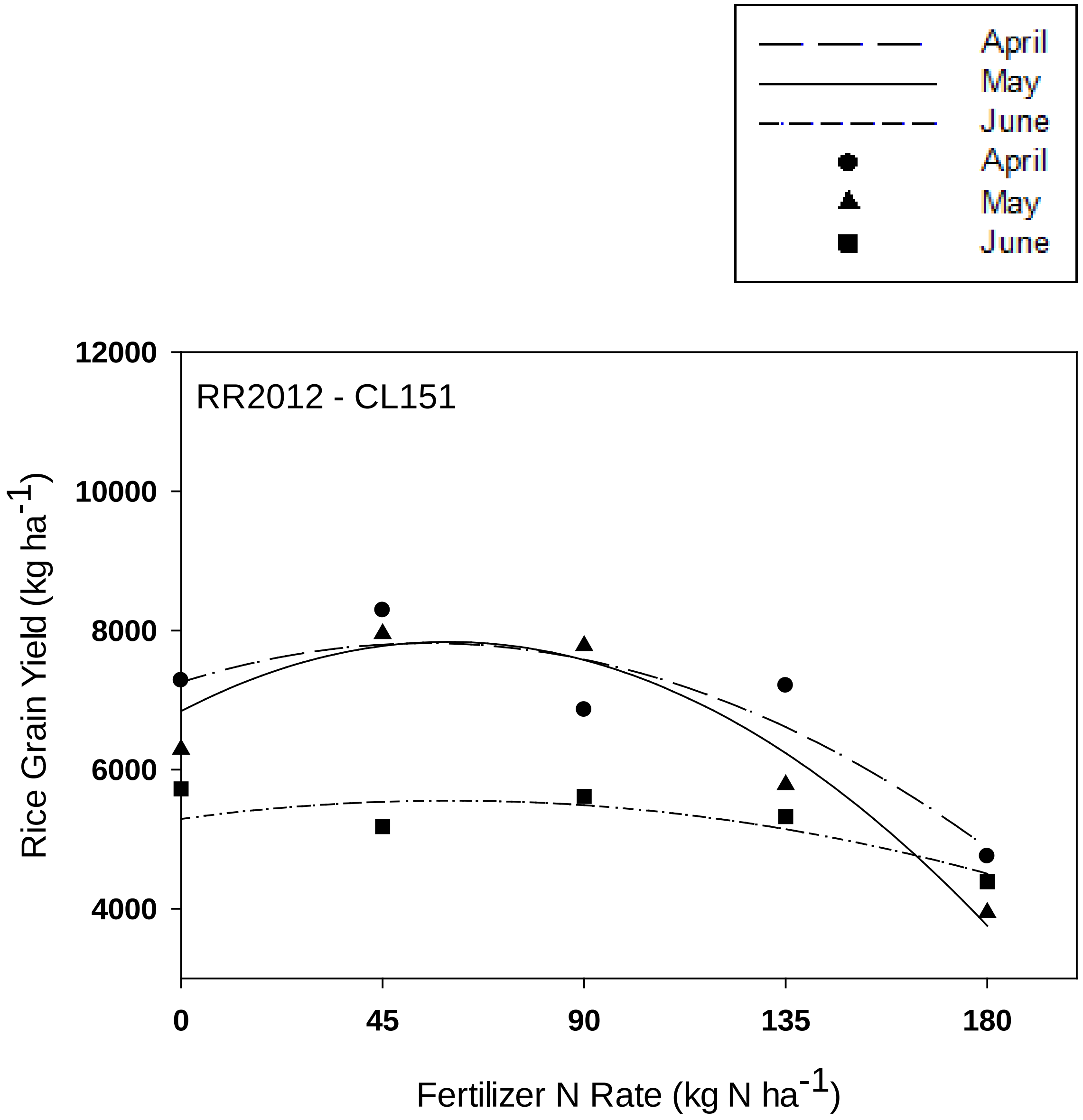

Figure 4.7D. Grain yield of CL151 in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

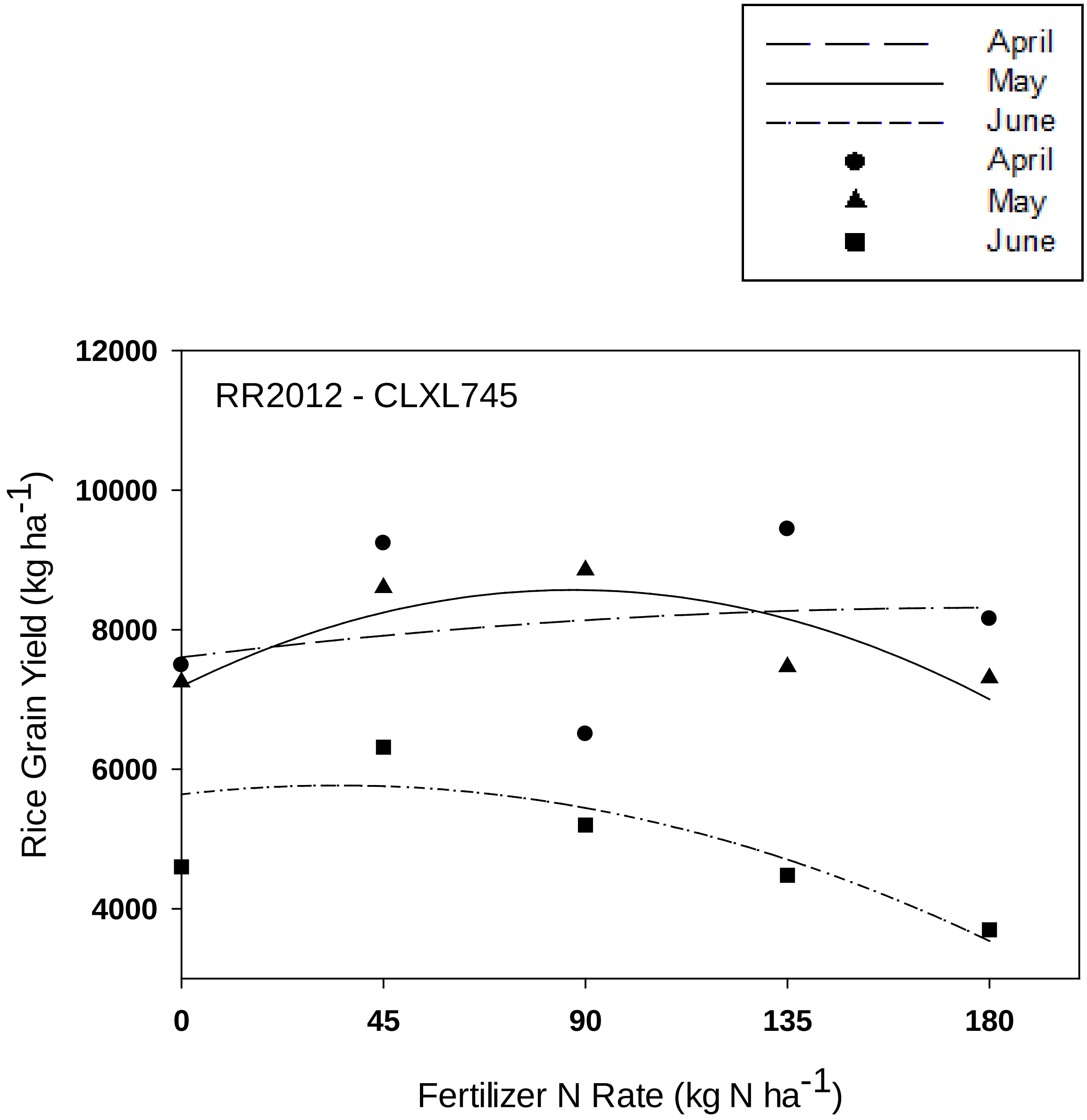

Figure 4.7E. Grain yield of CLXL745 in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

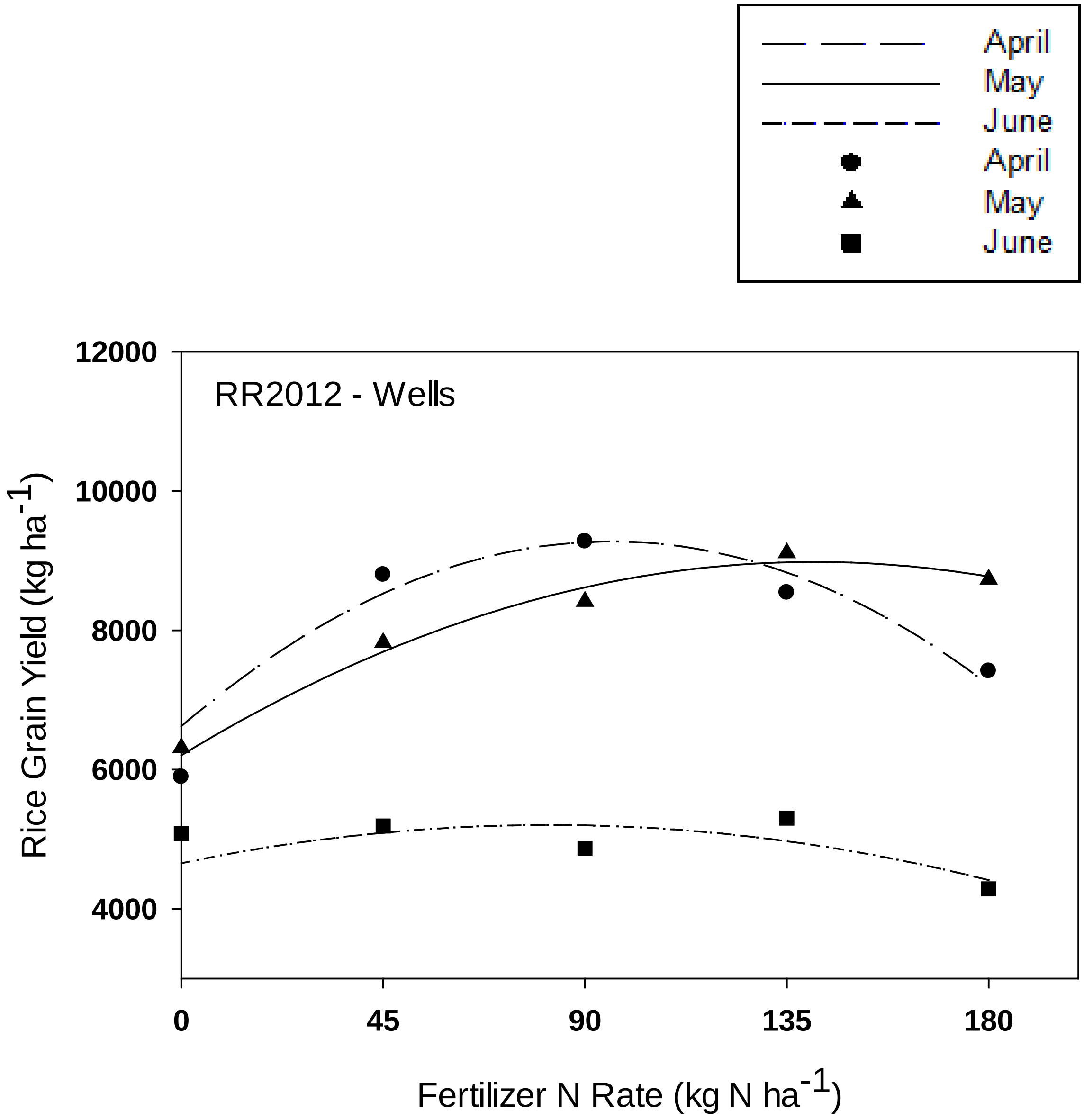

Figure 4.7F. Grain yield of Wells in response to fertilizer N rate when seeded in April, May, or June at Rice Research and Extension Center (RREC) in 2012 (RR2012).

CHAPTER FIVE

QUANTIFICATION OF CLAY-FIXED AMMONIUM USING ALKALINE DISTILLATION OR DRY COMBUSTION

ABSTRACT

Clay-fixed ammonium (NH_4–N) can represent a substantial portion of the total nitrogen (TN) content of agricultural soils. Recently, the Nitrogen Soil Test for Rice (N-STaR) was developed to guide site-specific fertilizer nitrogen (N) rate recommendations for rice (*Oryza sativa* L.) grown on silt-loam soil in Arkansas. The N-STaR method quantifies alkaline hydrolyzable-nitrogen (AH-N) using steam distillation. However, the amount or percentage of clay-fixed NH_4–N which N-STaR may predict to be available for plant uptake has yet to be investigated. Therefore, a laboratory study was conducted to determine the amount and percentage of plant-available, clay-fixed NH_4–N quantified using N-STaR. Nine sites, representing three soil textural classes (i.e., sandy loam, silt loam, and clayey), were sampled to a 45-cm depth in 15-cm increments in Arkansas. Clay-fixed NH_4–N, determined using a dry combustion method, averaged 15, 27, and 33% of soil TN at the 0- to 15-, 15- to 30-, and 30- to 45-cm depth, respectively. The N-STaR method recovered 5, 6, and 4% of the total clay fixed NH_4–N for the sandy loam, silt loam, and clayey soil textural classes evaluated in this study, respectively. The recovery of clay-fixed NH_4–N by N-STaR was lower than the plant availability of clay-fixed NH_4–N based on plant uptake reported in the literature, but similar to the percentage of clay fixed NH_4–N released from soil in the absence of plant growth during soil incubation. The results of this study suggest very little of the total clay-fixed NH_4–N in the top 45-cm soil depth is plant available.

INTRODUCTION

Soil NH_4-N fixation capacity has often been examined to determine the portion of the soil TN that is nonexchangeable NH_4–N. Nonexchangeable NH_4–N is not immediately available for plant uptake and can represent up to 10% of soil TN (Young and Aldag, 1982). Nonexchangeable NH_4–N has been operationally defined as NH_4–N which is not replaced by the prolonged extraction or leaching of soil with a potassium (K^+) salt solution (Barshad, 1951); clay-fixed NH_4–N has been classified as nonexchangeable NH_4–N (Scherer, 1993). Previous research has indicated that a portion of nonexchangeable NH_4–N can become available to plants (Kowalenko and Cameron, 1978; Mengel and Scherer, 1981) and microorganisms (Welch and Scott, 1960; Breitenbeck and Paramasivam, 1995), supporting the belief that nonexchangeable NH_4–N is a dynamic component of the soil N cycle (Allison et al., 1953a).

The clay minerals principally responsible for NH_4–N fixation are illite, montmorillonite, and vermiculite (Allison et al., 1953a; Bremner, 1959; Stevenson and Cole, 1999). These 2:1 phyllosilicate clay minerals are the products of pedogenic processes and can dominate the mineralogical composition of many agricultural soils. The contraction-expansion of the mineral lattice structure is the physical property of 2:1 phyllosilicates that can result in cations becoming 'fixed' or released from interlayer positions. Interlayer cations in a contracted lattice (e.g., NH_4^+ and potassium (K^+)) can be slowly replaced by soil solution calcium, magnesium, sodium, lithium, or hydrogen cations (e.g., Ca^{2+}, Mg^{2+}, Na^+, Li^+, or H^+, respectively) which enable the expansion but not contraction (e.g., Rb^+ and Cs^+) of the clay mineral structure (Nommik and Vahtras, 1982). Ammonium and K^+ are known to contract the structure of 2:1 phyllosilicates, so these cations will not be able to effectively replace one another in the interlayer position when added sequentially to soil.

Native fixed NH_4–N and recently fixed NH_4–N are the two general categories of nonexchangeable NH_4–N common to agricultural soils. The different mechanisms leading to the formation of native and recently fixed NH_4–N can lead to differences in the bioavailability of nonexchangeable NH_4–N. Native fixed NH_4–N and recently fixed NH_4–N can exist simultaneously in soil and soil containing native fixed NH_4–N can fix additional fertilizer-derived NH_4–N (Nommik, 1957; Doram and Evans, 1983). Previous research has used duplicate distillation (Barshad, 1951), difference methods (Bremner, 1959; Bremner and Harada, 1959), pretreatments (Dhariwal and Stevenson, 1958; Silva and Bremner, 1966), or concentrated acids (Rodrigues, 1954) to examine native nonexchangeable NH_4–N. The method of Silva and Bremner (1966) represented an improvement over previous methods aimed at quantifying nonexchangeable NH_4–N and gained a wide acceptance (Mulvaney, 1996); however, recent modifications of the Silva and Bremner (1966) method have included the elimination of alkaline pretreatments (Marzadori et al., 1993; Paramasivam and Breitenbeck, 2000) or concentrated acids (Liang et al., 1999).

The method of Silva and Bremner (1966) utilizes an alkaline potassium hypobromite (KOH–KOBr) pretreatment to oxidize organic-N and the remaining clay mineral residue is digested with a mixture of 5 mol L^{-1} hydrofluoric acid and 1 mol L^{-1} hydrochloric acid (5 mol L^{-1} HF–1 mol L^{-1} HCl), releasing nonexchangeable NH_4–N which is subsequently quantified by acidimetric titration. Nieder et al. (1996) eliminated the 5 mol L^{-1} HF–1 mol L^{-1} HCl digestion step of the original Silva and Bremner (1966) method and nonexchangeable NH_4–N was determined by dry combustion. The modification of Nieder et al. (1996) allowed for the quantification of nonexchangeable NH_4–N while expediting the analytical procedure and reducing labor. However, the elimination of 5 mol L^{-1} HF–1 mol L^{-1} HCl digestion prior to the quantification of clay-fixed NH_4–N only achieved wider acceptance following a comprehensive examination of the dry combustion method (Liang et al., 1999; Soon and Liang, 2008).

The dry combustion method has been proposed as a suitable alternative to the Silva and Bremner (1966) method for quantitative analysis of nonexchangeable NH_4–N (Liang et al., 1999). Liang et al. (1999) evaluated soil from across Canada with nonexchangeable NH_4–N concentration ranging from 0 to 350 mg N kg^{-1} and reported the dry combustion and 5 mol L^{-1} HF–1 mol L^{-1} HCl methods were strongly correlated ($r^2 = 0.97$) with a slope near one and an intercept not significantly different from zero. Further examination of the soil residue that was previously subjected to dry combustion indicated the procedure of Liang et al. (1999) achieved 100% recovery of nonexchangeable NH_4–N. Additionally, the study of Beuters and Scherer (2012) compared the procedure of Silva and Bremner (1966) to the dry combustion method for soils with clay content ranging from 3 to 50% and found the two methods were in agreement and differed by $\leq$ 10% across a range of nonexchangeable NH_4–N concentrations from 82 to 242 mg NH_4–N kg $soil^{-1}$.

Soil N fractionation using acid and alkaline hydrolysis has been evaluated to determine the influence of nonexchangeable NH_4–N on the measurement of soil organic-N (Greenfield, 2001) and mineralizable-N (Kowalenko and Babuin, 2009). Previous research has shown an average of 61% (range 34–103%) of nonexchangeable NH_4–N was released during acid hydrolysis (Kowalenko and Babuin, 2009), whereas an average of 11% (range 0–30%) of nonexchangeable NH_4–N was released during alkaline hydrolysis (Greenfield, 2001). The lower recovery of nonexchangeable NH_4–N using alkaline hydrolysis may be the result of methodological differences between acid and alkaline hydrolysis. Hanway and Scott (1956), using the alkaline distillation method of Barshad (1951), found nonexchangeable NH_4–N concentration was much lower compared to the nonexchangeable NH_4–N concentration determined using the 4:1 (v:v) mixture of hydrofluoric acid–sulfuric acid ($HF–H_2SO_4$) method of Rodrigues (1954). The conclusion of Hanway and Scott (1956) was the low concentration of nonexchangeable NH_4–N quantified using alkaline distillation could have resulted from an inability of sodium hydroxide (NaOH) to effectively replace NH_4–N fixed in the interlayer of clay minerals. Ammonium present in the interlayer of clay minerals has been identified as nonexchangeable NH_4–N or fixed NH_4–N (Scherer and Mengel, 1986) as well as intercalary NH_4–N (Osborne, 1976). Due to differences in terminology used throughout the literature for this fraction of soil N, the N which remains in soil following removal of organic-N and exchangeable inorganic-N will henceforth be referred to as clay-fixed NH_4–N.

The utilization of clay-fixed NH_4–N by plants and microorganisms suggests clay-fixed NH_4–N is a biologically important form of N, and conditions which influence clay-fixed NH_4–N may also influence the quantity of plant-available NH_4–N. The interaction between clay-fixed NH_4–N and immediately available N also suggests clay-fixed NH_4–N should be considered as a potential source of plant-available N (Baethgen and Alley, 1987). Several factors influence the availability of clay-fixed NH_4–N for plant uptake. The degree of K^+ saturation and clay mineral type as well as the concentration of exchangeable and soil solution NH_4–N can influence the release of clay-fixed NH_4–N (Nieder et al., 2011). The seasonal change of clay-fixed NH_4–N has been examined previously and maximum N uptake of winter wheat was shown to coincide with an observed decrease of clay-fixed NH_4–N during the growing season, while clay-fixed NH_4–N was shown to increase as mineral N increased following harvest as a result of increased soil N mineralization (Nieder et al., 1996). The release of clay-fixed NH_4–N, as a result of changes in the solution and exchangeable NH_4–N concentration, suggests plant available NH_4–N may include the portion of clay-fixed NH_4–N recovered by the crop during the growing season. The uptake of clay-fixed NH_4–N by cereal crops has been investigated. Barley (*Hordeum vulgare* L.) grown under field conditions utilized as much as 30% of nonexchangeable NH_4–N (Soon, 1998), while millet (*Pennisetum glaucum* L.) grown under greenhouse conditions utilized as much as 12% of clay-fixed NH_4–N (Allison et al., 1952).

The Nitrogen Soil Test for Rice, referred to as N-STaR, is a soil test method that quantifies AH-N using steam distillation to provide an index of potentially mineralizable-N (Bushong et al., 2008; Roberts et al., 2009). Hydrolyzable NH_4–N may include exchangeable NH_4–N, amino sugar-N and amino acid-N (Roberts et al., 2009). The recent validation of N-STaR fertilizer N rate recommendations (Roberts et al., 2013) indicates the AH-N concentration of silt-loam soils is indicative of fertilizer N responsiveness by rice grown in Arkansas. Previous research has evaluated the recovery of organic-N compounds by the N-STaR method to gain a better understanding of the organic-N forms and amounts of each form of N quantified using the N-STaR soil test method (Roberts et al., 2009; Roberts et al., 2012). However, the amount and percentage of clay-fixed NH_4–N recovered by N-STaR has not been investigated. The objective of this study was to determine the amount and percentage of clay-fixed NH_4–N recovered by N-STaR and predicted to be available in selected sandy loam, silt loam, and clayey soils sampled to a 45 cm depth in Arkansas.

MATERIALS AND METHODS

Soil Sampling and Analysis

Nine field sites were sampled using a Dutch Auger Probe (AMS Inc., American Falls, Idaho) in Arkansas in 2013. Soil samples were obtained from a 45-cm depth and divided into three individual 15-cm sampling depth increments (i.e., 0–15, 15–30, and 30–45 cm) for each site. Selected soil physical and chemical properties as well as the location and taxonomic classification of the sites evaluated in this study are presented in Table 5.1. Soil was dried at 60°C within 24 to 48 h after removal from the field, ground to pass a 2-mm sieve and stored in cardboard containers prior to chemical analysis. Exchangeable NH_4–N and NO_3–N were determined using a 1:10 (m:v) 2 mol L^{-1} KCl extraction and the N concentration of the filtered (Whatman No. 42) solution was determined colorimetrically (Mulvaney, 1996). Soil total carbon (TC; Nelson and Sommers, 1996) and soil TN (Bremner, 1996) were determined using an Elementar CN

Variomax (Elementar Americas, Inc., Mt. Laurel, NJ). Soil pH was determined in the supernatant of a 1:2 (m:v) soil-to-water suspension (Thomas, 1996) by Ag/AgCl pH electrode and soil particle size was determined using a modified 12 h hydrometer method (Gee and Or, 2002). Alkaline hydrolyzable-N was quantified by N-STaR procedures (Bushong et al., 2008; Roberts et al., 2009).

Quantification of Clay-Fixed Ammonium

Clay-fixed NH_4–N of surface (i.e., 0–15 cm) and subsurface (i.e., 15–45 cm) soil was measured using dry combustion or N-STaR following removal of organic-N and exchangeable inorganic-N. Briefly, 20 mL of 2 mol L^{-1} KOH–KOBr were added to 1 g of finely ground and sieved (< 0.15 mm) soil in a 200 mL glass beaker. The soil–solution mixture was covered and kept at room temperature (~ 21°C) for 2 h before 60 mL of deionized water was added, heated to approximately 99°C for 5 min and then allowed to slowly cool and settle overnight (~ 16 h) at room temperature. The 2 mol L^{-1} KOH–KOBr oxidation pretreatment has been shown to effectively remove 96 to 99% of organic-N in soil containing up to 150 g kg^{-1} of organic matter (Silva and Bremner, 1966). There is a range of 5 to 50 g kg^{-1} of organic matter for soils of Arkansas (Espinosa et al., 2013) therefore, 2 mol L^{-1} KOH–KOBr pretreatment would be expected to achieve complete removal of organic-N for the soils evaluated in this study.

Following soil pretreatment with 2 mol L^{-1} KOH–KOBr, the clear supernatant was decanted and three sequential extractions with 0.5 mol L^{-1} KCl were used to remove any exchangeable inorganic-N. The soil residue was quantitatively transferred to a 50 mL glass centrifuge tube with 30 mL of 0.5 mol L^{-1} KCl, shaken for 5 min, centrifuged, and the entire process repeated. Upon completion of the third extraction, the clear supernatant was discarded and the contents of the centrifuge tube were allowed to oven-dry overnight at 105°C.

Pretreated, oven-dried soil was removed from the centrifuge tube, crushed with a mortar and pestle, weighed, and stored at room temperature prior to analysis of clay-fixed NH_4–N. Soil residue recovery ranged from 91 to 100% following removal of organic-N and exchangeable inorganic-N. Plant available clay-fixed NH_4–N was quantified by N-STaR using approximately 1 g of soil residue and 10 mL of 10 mol L^{-1} NaOH during a ~7 min distillation. A total of 35 mL of distillate was collected in 40 g L^{-1} boric acid indicator solution and the N concentration of the distillate determined by acidimetric titration (719 S Titrino, Metrohm Ltd., Herisau, Switzerland). Total clay-fixed NH_4–N was also quantified by dry combustion of approximately 75 mg of pretreated soil residue at the University of California Davis – Stable Isotope Facility using an elemental analyzer equipped with a mass spectrometer (Europa, Sercon Ltd., Cheshire, UK). Quantification of clay-fixed NH_4–N by N-STaR or dry combustion was performed in triplicate.

Statistical Analysis

The quantification of clay-fixed NH_4–N by N-STaR or dry combustion was evaluated using a spilt-split plot arrangement of treatments. Soil textural class was the whole plot factor arranged in a randomized complete block, sampling depth was the split plot factor and method of analysis was the split-split plot factor. Soil series nested within soil textural class and block were considered random factors. Analysis of variance (ANOVA) was conducted using PROC MIXED to determine the influence of the main effects of soil textural class, sampling depth, and analytical method along with all main effect interactions on the quantification of clay-fixed NH_4–N. Means were separated using Fisher's protected least significant difference (LSD) and statistical significance interpreted at the 0.05 probability level. The quantity of clay-fixed NH_4–N was regressed on soil TN and the statistical significance of the linear regression model was evaluated at the 0.05 probability level for each sampling depth (i.e., 0- to 15-, 15- to 30-, and 30- to 45-cm) using PROC REG. All statistical analyses were conducted in SAS v9.2.

RESULTS AND DISCUSSION

Alkaline hydrolyzable-N, determined using N-STaR, ranged from 6.4 to 151.2 mg N kg soil^{-1} across all sites and soil depths (Table 5.2); the range for silt-loam soil (i.e., Sites 2, 5 and 6) of 35.2 to 151.2 mg N kg soil^{-1} was similar to the AH-N concentration reported by Roberts et al. (2011) for other silt loam soils commonly cropped to rice in Arkansas. The AH-N concentration of clayey soils (i.e., Sites 3, 8, and 9) was numerically greater than the AH-N concentration of the silt-loam (i.e., Sites 2, 5, and 6) and sandy loam soils (i.e., Sites 1, 4, and 7) at the 15- to 30-cm and 30- to 45-cm depths. Sites were selected based on differences in soil texture. The wide range in AH-N concentration observed in this study was similar to the results of Roberts et al. (2012) who reported that AH-N, quantified to a 60-cm depth, exhibited a wide range even within a particular soil textural class.

Quantification of Clay-Fixed Ammonium using N-STaR or Dry Combustion

There was a significant soil textural class x method interaction ($P < 0.0001$) for the quantification of clay-fixed NH_4–N (Table 5.3). Averaged over sampling depth, clay-fixed NH_4–N ranged from 86.3 to 265.5 mg NH_4–N kg soil^{-1} using dry combustion (Table 5.4). The quantity of clay-fixed NH_4–N determined by N-STaR to be plant available ranged from only 4.5 to 11.0 mg NH_4–N kg soil^{-1}. Clay-fixed NH_4–N concentration determined to be plant available by N-STaR was not significantly different among soil textural classes. However, the total clay-fixed NH_4–N concentration, determined by dry combustion, of clayey soils was significantly greater compared to sandy loam and silt loam soils, with sandy loam and silt loam soils having a similar total clay-fixed NH_4–N concentration. While the total clay-fixed NH_4–N concentration was influenced by soil textural class, N-STaR did not indicate a difference among the soil textural classes.

Soil Total Nitrogen and Clay-Fixed Ammonium Quantified using Dry Combustion

There was a significant and positive linear relationship between clay-fixed NH_4–N and soil TN concentration at each sampling depth (Figure 5.1A–C). The coefficient of determination (r^2) increased with sampling depth and reached a maximum ($r^2 = 0.70$) at the 30- to 45-cm depth (Figure 5.1C). The slope of the linear regressions indicated that clay-fixed NH_4–N was 15, 27, and 33% of soil TN for the 0- to 15-, 15- to 30-, and 30- to 45-cm sampling depth, respectively. This result suggests clay-fixed NH_4–N represented a greater percentage of soil TN in subsurface (i.e., 15–45 cm) compared to surface (i.e., 0–15 cm) soil. The results of this study are similar to the results of Sparks et al. (1979) who reported clay-fixed NH_4–N averaged 7% of soil TN in the surface (i.e., 0–10 cm) and 33% of soil TN in the subsurface (i.e., 23–46 cm) of a silty clay loam soil. The results of Black and Waring (1972) and Hinman et al. (1964) also indicated that clay-fixed NH_4–N was a larger percentage of soil TN as sampling depth increased due to a decrease of soil TN (i.e., less soil organic N) with depth in the soil profile. In the present study, clay-fixed NH_4–N as a percentage of soil TN increased with depth and this trend was evident when soil textural classes were combined.

Quantification of Plant-Available Clay-Fixed Ammonium using N-STaR

The quantity of clay-fixed NH_4–N determined to be plant available by N-STaR was 5, 6, and 4% of the total clay-fixed NH_4–N concentration of sandy loam, silt loam, and clayey soils, respectively (Table 5.4). Smith et al. (1994) concluded that an average of only 8 and 4% of total clay-fixed NH_4–N was released from surface (0–15 cm) and subsurface (15–30 cm) soil, respectively, during a long-term (168 d) aerobic incubation. Numerous factors such as root growth and root exudates may influence the release and uptake of clay-fixed NH_4–N. Ryegrass (*Lolium perenne* L.) grown in the greenhouse was shown to recover 7 to 72% of clay-fixed NH_4–N was recovered following fertilizer application (Osborne, 1976; Norman and Gilmour, 1987). Additional research has also reported recently fixed NH_4–N derived from fertilizer was released and available for plant uptake (Black and Waring, 1972; Mengel and Scherer, 1981). However, chemical methods used to quantify recently fixed NH_4–N have underestimated the amount released under the influence of plant growth (Scherer and Ahrens, 1996).

Clay-fixed NH_4–N recovered by N-STaR remained low regardless of soil textural class and suggests very little of the total clay-fixed NH_4–N is available for plant uptake. The percent recovery of clay-fixed NH_4–N by N-STaR appeared to be similar to previous research conducted to determine the amount of clay-fixed ammonium released during soil incubation (Smith et al., 1994), suggesting N-STaR may provide an index of plant available clay-fixed NH_4–N. However, the disagreement between clay-fixed NH_4–N measured by N-STaR and plant uptake of clay-fixed NH_4–N previously reported in the literature indicates the amount of clay-fixed NH_4–N released under the influence of plant growth may be greater than the portion of clay fixed NH_4–N released using N-STaR.

Nieder et al. (2011) suggested the inability to adequately distinguish between recently fixed NH_4–N (i.e., plant available NH_4–N) and native fixed NH_4–N (i.e., unavailable NH_4–N) obscured the assessment of the biological importance of clay-fixed NH_4–N. Recently-fixed NH_4–N entrapped near the edges of the clay lattice are not completely retained against alkaline extraction and could therefore be a likely contributor to plant available N (Paramasivam and Breitenbeck, 2000). The results of the present study revealed that plant-availability of clay-fixed NH_4–N was similar among soil textural classes suggesting soil textural class did not influence the amount of clay-fixed NH_4–N predicted to be available for plant uptake by N-STaR (Table 5.4). Similar to the results of the present study, Nieder et al. (2011) reviewed the published literature and reported a similar amount of clay-fixed NH_4–N was apparently released during the growing season for clayey and silty soils despite the typically greater total clay-fixed NH_4–N for clayey soils compared to silty soils. The quantification of clay-fixed NH_4–N by N-STaR represented only a small percentage of total clay-fixed NH_4–N determined by dry combustion for sandy loam, silt loam, and clayey soils. The N-STaR method would only be expected to quantify the portion of clay-fixed NH_4–N which is available for plant uptake and therefore the incomplete recovery of clay-fixed NH_4–N by N-STaR was not an unexpected result of this study.

CONCLUSIONS

There is considerable variation in the clay-fixed NH_4–N content of arable soils within the plow layer (~ 30 cm depth) and there are factors (e.g., parent material, soil texture, clay content, etc.) which are known to influence clay-fixed NH_4–N (Nieder et al., 2011). Recent adoption of the N-STaR soil test method for rice grown on silt-loam soil in Arkansas, correlation and calibration on clayey soils, and questions about the importance of clay-fixed NH_4-N on plant-available N warranted an evaluation of clay-fixed NH_4–N quantified using N-STaR for soils under row crop production. Clay-fixed NH_4–N concentration, averaged over a 45-cm sampling depth and determined by dry combustion and N-STaR methods was significantly greater for clayey soils compared to sandy loam and silt loam soils. However, despite a significantly different total clay-fixed NH_4–N concentration, the amount of clay-fixed NH_4–N predicted to be available for plant uptake by N-STaR was similar regardless of soil textural class. Clay-fixed NH_4–N percentage of soil TN ranged from 15 to 33%, increased with sampling depth, and was similar to the range of percentages reported previously for surface and subsurface soil.

Results indicated dry combustion may be a suitable alternative to the laborious and time consuming methods previously developed to quantify clay-fixed NH_4–N and may be applicable across a wide range of soil textural classes when sampled to a 45-cm depth in Arkansas. The N-STaR method estimated that only 4 to 6% of the total clay-fixed NH_4–N was available for plant uptake. The release of clay-fixed NH_4–N using alkaline steam distillation suggests soil test methods such as N-STaR measure a biologically important portion of total clay-fixed NH_4–N as AH-N for sandy loam, silt loam, and clayey soils sampled to a 45-cm depth. The clay-fixed NH_4–N quantified by N-STaR was reported only after complete removal of organic-N and exchangeable inorganic-N. Therefore, additional research is required to determine the recovery of clay-fixed NH_4–N prior to the removal of organic-N and exchangeable inorganic-N to verify the amount of plant-available, clay-fixed NH_4–N quantified by N-STaR.

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Table 5.1. Selected physical/chemical and location characterization of the nine sites sampled in Arkansas and utilized for the laboratory study.

Site	GPS	Soil Series	Taxonomy	Texture	Depth (cm)	pH†	NO_3–N‡	NH_4–N‡	TN§	Clay-Fixed NH_4–N§	TC§	Sand¶	Clay ¶
							mg kg^{-1}		g kg^{-1}			%	
1	35° 52’ N, 90° 52’ W	Bosket	Hapuludalfs	sandy loam	0–15	6.8	4	11	0.63	0.13	5.87	67	16
					15–30	6.5	6	12	0.40	0.13	3.08	68	19
					30–45	6.6	9	13	0.38	0.14	2.13	67	19
2	35° 52’ N, 90° 56’ W	Amagon	Endoaqualfs	silt loam	0–15	6.7	2	16	0.69	0.11	6.25	44	7
					15–30	7.2	2	11	0.70	0.08	6.47	45	7
					30–45	7.0	4	12	0.70	0.09	6.38	40	9
3	35° 50’ N, 90° 53’ W	Jackport	Epiaquerts	clay loam	0–15	6.7	9	13	1.20	0.20	12.59	15	41
					15–30	6.4	14	8	1.03	0.19	11.52	16	43
					30–45	5.4	2	6	0.84	0.19	8.31	13	46
4	35° 34’ N, 91° 15’ W	Beulah	Dystrudepts	sandy loam	0–15	5.6	< 1	7	1.02	0.11	9.01	60	10
					15–30	5.8	< 1	5	0.64	0.11	4.77	59	13
					30–45	6.0	1	4	0.42	0.11	2.91	58	11
5	35° 36’ N, 91° 14’ W	Dexter	Hapludalfs	silt loam	0–15	6.5	1	6	1.19	0.12	10.43	63	8
					15–30	6.5	< 1	4	0.54	0.11	4.22	63	8
					30–45	6.6	< 1	4	0.35	0.08	2.42	68	6
6	36° 5’ N, 94° 10’ W	Captina	Fragiudults	silt loam	0–15	5.9	< 1	7	0.71	0.05	6.18	40	6
					15–30	5.8	1	5	0.48	0.06	3.15	32	14
					30–45	6.4	1	4	0.46	0.08	2.76	30	22
7	35° 22’ N, 94° 13’ W	Roxana	Udifluvents	sandy loam	0–15	7.3	< 1	4	0.27	0.07	2.71	54	9
					15–30	7.3	< 1	3	0.09	0.03	0.92	71	3
					30–45	7.4	< 1	2	0.06	0.03	0.47	76	3
8		Sharkey	Epiaquerts	clay	0–15	7.5	1	6	1.12	0.32	12.01	23	45

Table 5.1. Continued.

Site	GPS	Soil Series	Taxonomy	Texture	Depth (cm)	pH†	NO_3–N‡	NH_4–N‡	TN§	Clay-Fixed NH_4–N§	TC§	Sand¶	Clay¶
							---------mg kg^{-1}-------		----------------g kg^{-1}---------------			--------%-------	
8	35° 40’ N, 90° 5’ W				15–30	7.6	2	6	1.00	0.34	9.75	10	57
					30–45	7.6	2	6	0.91	0.38	8.90	6	58
9	35° 40’ N, 90° 5’ W	Tunica	Epiaquepts	clay	0–15	6.8	4	8	0.96	0.28	9.91	14	43
					15–30	7.1	6	6	0.80	0.28	7.96	13	42
					30–45	6.9	6	5	0.71	0.23	6.73	14	39

†Determined on a 1:2 (m:v) soil-to-water suspension (Thomas, 1996);

‡Determined using salicylate colorimetric techniques (Mulvaney, 1996).

§TN, total N, TC, total carbon, and clay-fixed NH_4–N determined using dry combustion (Bremner, 1996; Nelson and Sommers, 1996; Liang et al., 1999);

¶Determined using hydrometer method (Gee and Or, 2002).

Table 5.2. Mean alkaline hydrolyzable-N (AH-N) concentration, as estimated by the Nitrogen Soil Test for Rice (N-STaR), for nine sites sampled to a 45-cm depth in Arkansas. Number shown in parentheses is the standard error.

		N-STaR†		
Site	Textural class	0–15 cm	15–30 cm	30–45 cm
		------------------mg N kg soil^{-1}	-----------------	
1	sandy loam	76.4 (1.5)	45.6 (1.5)	36.0 (1.5)
4	sandy loam	138.8 (0.4)	77.7 (3.4)	46.4 (0.4)
7	sandy loam	32.9 (3.0)	7.9 (2.6)	6.4 (1.8)
2	silt loam	115.6 (0.4)	69.4 (4.2)	64.9 (0.2)
5	silt loam	151.2 (4.2)	63.0 (3.2)	35.2 (1.0)
6	silt loam	79.6 (0.9)	49.9 (0.6)	39.2 (2.0)
3	clay loam	149.1 (3.0)	138.3 (0.8)	107.8 (2.7)
8	clay	119.0 (1.2)	110.0 (3.1)	104.1 (4.2)
9	clay	122.4 (0.6)	100.9 (0.8)	85.9 (0.6)

†Mean of three replicate determinations

Table 5.3. Analysis of variance (ANOVA) *P*-values for the quantification of clay-fixed NH_4–N as influenced by soil textural class (T), sampling depth (D), analytical method (M) and all main effect interactions.

Source of Variation	df	*P*-value
Soil Textural Class (T)	2	0.0100
Sampling Depth (D)	2	0.8203
Analytical Method (M)	1	< 0.0001
T x D	4	0.9584
T x M	2	< 0.0001
D x M	2	0.9439
T x D x M	4	0.9953

Table 5.4. Clay-fixed NH_4–N concentration, averaged over sampling depth, as influenced by soil textural class and analytical method.

	N-STaR	Combustion
Soil Textural Class	------------(mg NH_4–N kg soil^{-1})----------	
Sandy loam	4.5	94.7
Silt loam	5.0	86.3
Clayey	11.0	265.5

$LSD_{0.05}$ ($P < 0.0001$) to compare mean clay-fixed NH_4–N, mg N kg soil^{-1},
for the same soil textural class across analytical methods = 19.1;
for the same analytical method across soil textural class = 56.4

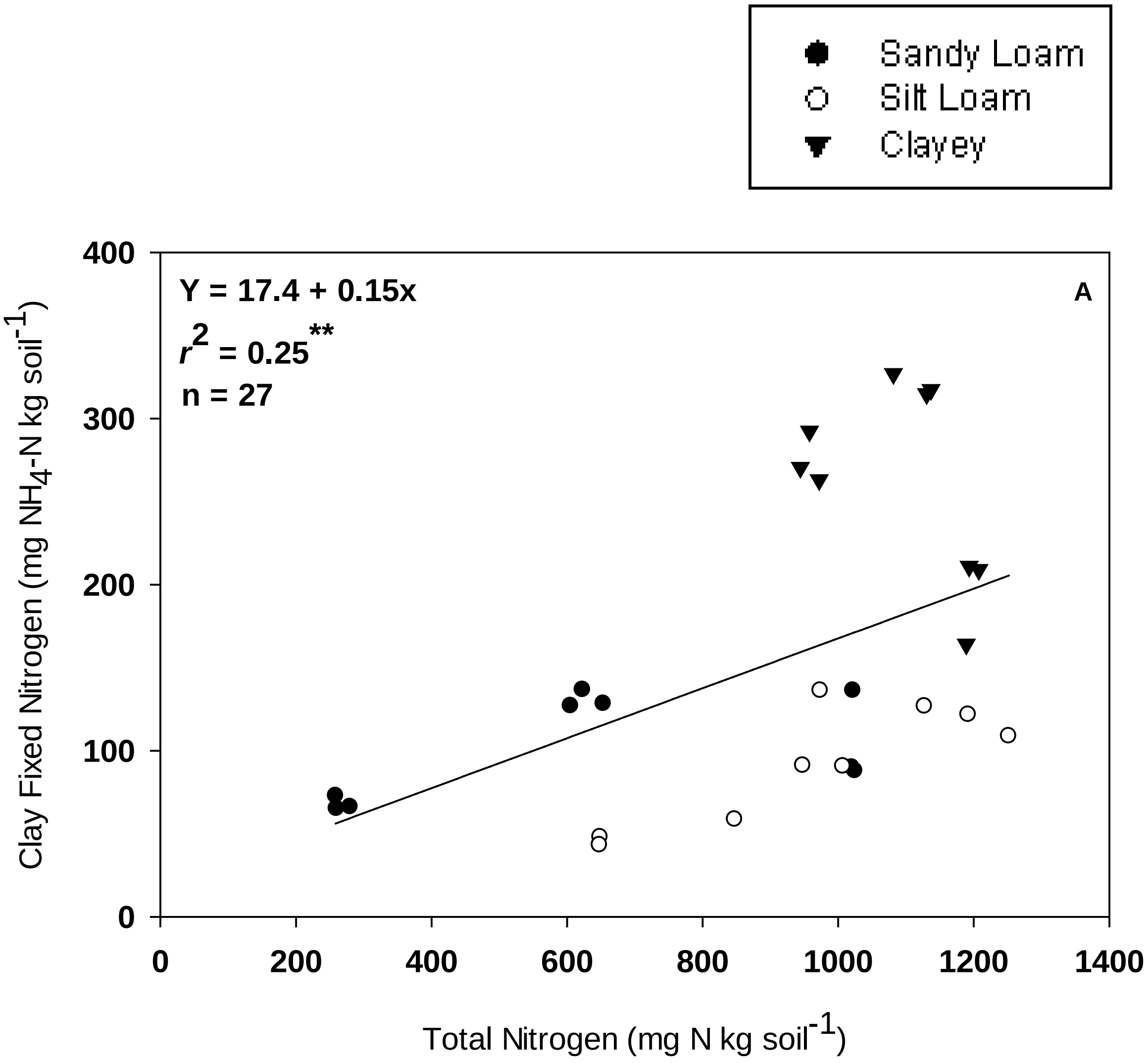

Figure 5.1A. Clay-fixed NH_4–N, determined using dry combustion, regressed on soil total N (TN) with the regression equation developed using a sampling depth of 0- to 15-cm. ** denotes statistical significance of the linear regression at the 0.01 level of probability.

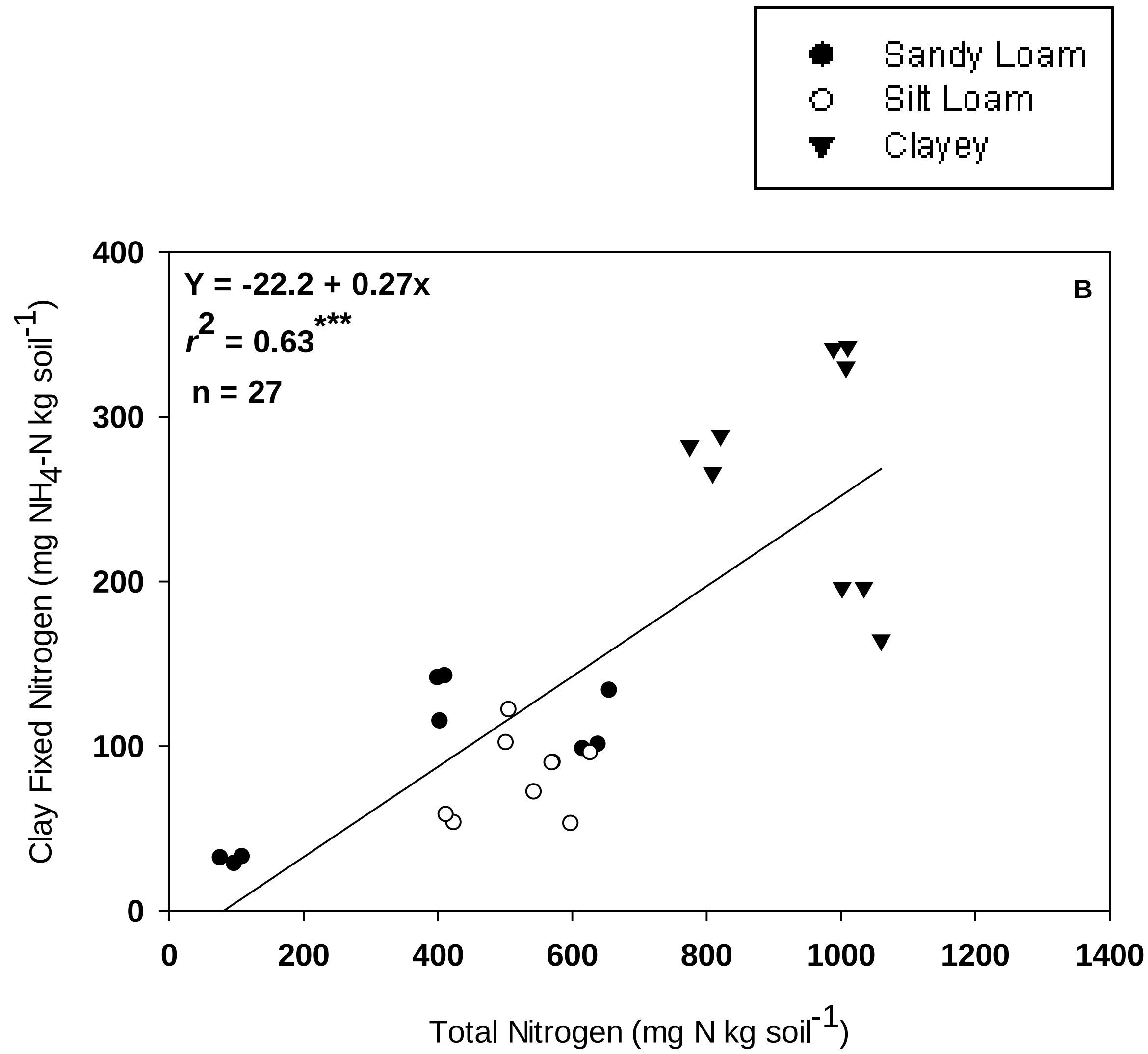

Figure 5.1B. Clay-fixed NH_4–N, determined using dry combustion, regressed on soil total N (TN) with the regression equation developed using a sampling depth of 15- to 30-cm. *** denotes statistical significance of the linear regression at the 0.001 level of probability.

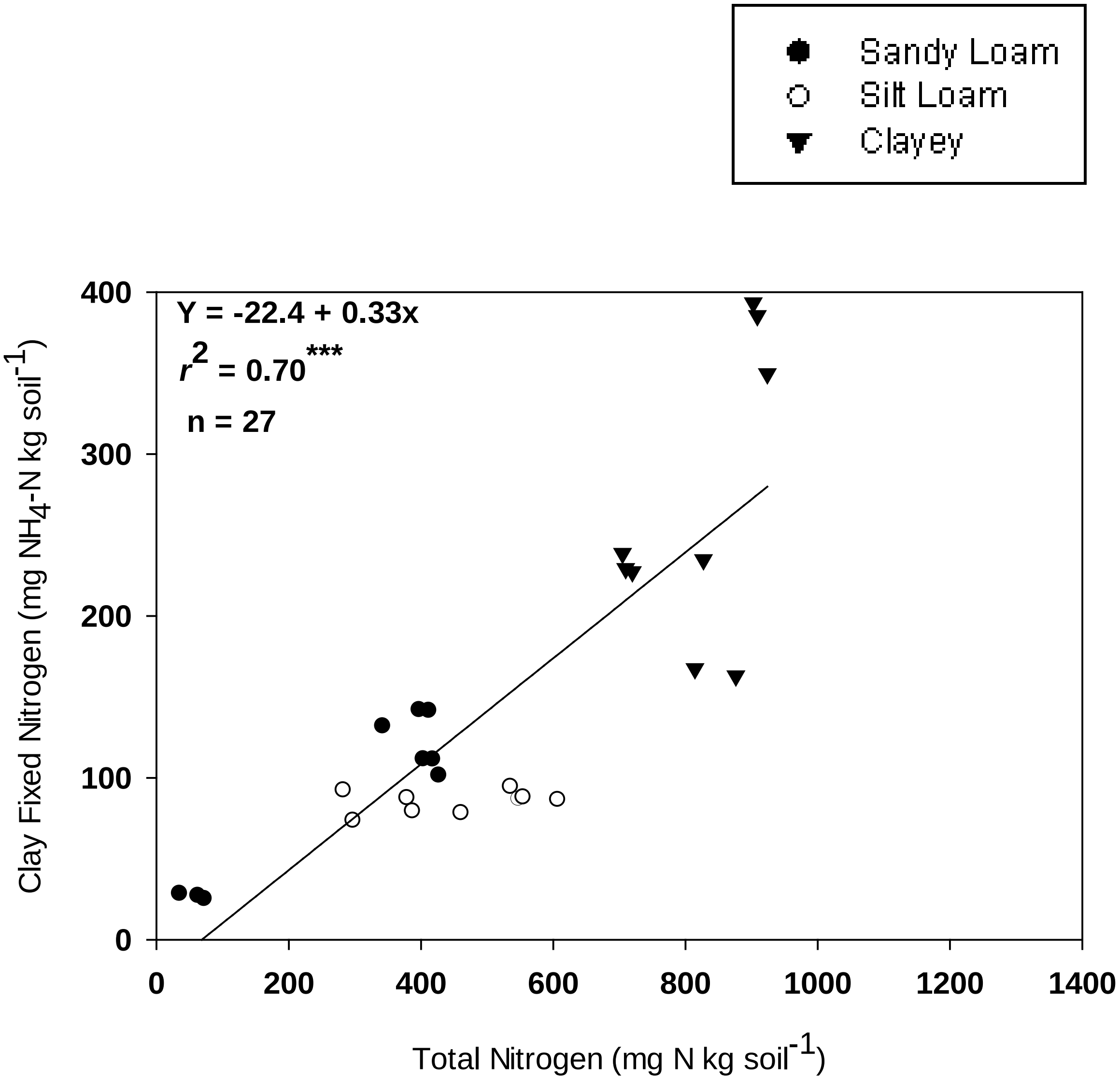

Figure 5.1C. Clay-fixed NH_4–N, determined using dry combustion, regressed on soil total N (TN) with the regression equation developed using a sampling depth of 30- to 45-cm. *** denotes statistical significance of the linear regression at the 0.001 level of probability.

CHAPTER SIX

CONCLUSIONS

The successful validation of the Nitrogen Soil Test for Rice (N-STaR) fertilizer N rate recommendations for rice (*Oryza sativa* L.) grown on silt-loam soil supported the belief that alkaline-hydrolyzable N (AH-N) could be utilized as an indicator of rice response to N fertilization. Several research questions were raised during the development of N-STaR including the validity of N-STaR fertilizer N rate recommendations when rice was grown on clayey soils. Alkaline hydrolyzable-N of clayey soils was subsequently correlated to rice response parameters including: total N (TN) uptake, check plot grain yield, and percentage of relative grain yield when no N fertilizer was applied (% RGY_{0N}). The influence of soil sampling depth was reflected in the predictive ability of each linear regression model. The 0- to 30-cm sampling depth was shown to provide the greatest coefficient of determination (r^2) among the depth-averaged sampling intervals when TN uptake, check plot grain yield, or % RGY_{0N} were regressed on AH-N values determined by ISNT or N-STaR. The 0- to 30-cm sampling depth also explained 83% of the observed variability in the fertilizer N rate required to achieve 95% RGY using ISNT or N-STaR. The correlation data supported the results of fertilizer N rate calibration and suggested the utilization of a sampling depth (i.e., 0–30 cm) appropriate for rice grown on clayey soils could maximize the predictive ability of the ISNT and N-STaR soil test methods.

In addition to AH-N, seeding date was also investigated as a potential indicator of rice response to N fertilization. The seeding date studies conducted in Arkansas revealed TN uptake and aboveground dry matter at 50% heading exhibited a tendency to increase as seeding date was delayed until June, although grain yield tended to decrease as seeding date was delayed until June. Results suggested the yield advantage of April seeded rice was not due to greater TN uptake or dry matter accumulation compared to rice seeded in May or June in Arkansas. Any differences in the fertilizer N requirement to maximize grain yield of late-seeded rice compared to early-seeded rice remained unclear following the completion of the seeding date studies in 2012. Research is still needed to definitively identify the N requirement of rice when seeding date is delayed beyond the optimal time period in Arkansas.

Organic-N compounds and their percent recovery using N-STaR have been documented, however, there have been no studies concerning the amount of clay-fixed NH_4–N recovered by N-STaR. Results of a laboratory study revealed that very little of the clay-fixed NH_4–N of sandy loam, silt loam, and clayey soils was recovered and predicted to be plant available based on the N-STaR method. Alkaline hydrolyzable-N, seeding date, and the soil's clay-fixed NH_4–N content are potential indicators of rice response to N fertilization. The results presented in the preceding studies indicated that AH-N could be used to identify yield-maximizing fertilizer N rates for rice grown on clayey soils, delayed seeding dates altered fertilizer N response trends compared to rice planted within an optimal seeding date window, and plant-available clay-fixed NH_4–N, determined using N-STaR, represented a low percentage of total clay-fixed NH_4–N for soils of agricultural importance in Arkansas.