

## AN ABSTRACT OF THE THESIS OF

Todd E. Shearin for the degree of Master of Science in Soil Science presented on September 22, 1999.

Title: Winter Wheat Response to Nitrogen, Phosphorus, Sulfur, and Zinc Supplied by Municipal Biosolids

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Dan Sullivan

Biosolids are stabilized solids derived from municipal wastewater treatment that meet federal criteria for land application. Biosolids application rates are usually based on supplying the crop with adequate nitrogen (N). Wastewater treatment plant operators need to provide evidence that adequate N was supplied by biosolids applications. The main objectives of this research were to: (i) determine the agronomic rate of biosolids and available N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) for dryland wheat production, (ii) evaluate the use of flag leaf tissue testing as an indicator of plant nutritional status, and (iii) determine the residual soil fertility effects via soil and plant tissue testing. Anaerobically-digested and dewatered biosolids (17% dry matter, 83% water) were supplied by the Unified Sewerage Agency (USA) of Washington County (OR). Three biosolids rates (low, 150 to 190 kg N/ha; medium, 250 to 380 kg N/ha; and high, 470 to 570 kg N/ha) were applied in the fall after crop harvest and in the spring prior to the first fallow tillage, at two locations in Sherman County, OR. The biosolids applications were compared to both an anhydrous ammonia control (56-67 kg N/ha) and an unfertilized control. An application rate of 230-370 kg biosolids N/ha (5.2-7.6 Mg biosolids/ha) provided 90 to 95% of the maximum grain yields, with no decrease in grain quality. Available soil N from biosolids application ranged from 15-25% of the biosolids N applied, when sampled in the spring of the crop year. Postharvest soil sampling showed no significant increase in available soil N at either location. Sampling the flag leaf during the flowering development stage (Feekes 10.5 to 10.51) produced the best correlation between flag leaf N concentrations and 90 to 95% maximum grain yields. Critical nutrient concentrations (CNC) were found

to be 3.5 to 3.7% N at heading, 3.0 to 3.2% N at flowering, and 2.8 to 3.0% N at milk development. Flag leaf N concentrations were also correlated with grain N ( $R^2 = 0.66$  to  $0.69$  at flowering). Biosolids increased Bray-1 P and DTPA-extractable Zn soil test values (0-15 cm). During the fallow sampling, 14% of the total S applied was recovered as  $SO_4$ -S. Average coefficient of variation (CV) for the flag leaf at flowering was 8% for N, 11% for S, and 9% for Zn. Due to the quickness and ease of sampling, wide range of nutrient concentrations, and low CV values, flag leaf samples appear to be the best plant tissue to sample for determination of N, S, and Zn status. Future research focusing on long term effects of biosolids on residual soil fertility and evaluation of flag leaf CNC at multiple locations are needed. Overall, biosolids were a suitable nutrient source for dryland wheat production in the Pacific Northwest at our locations.

WINTER WHEAT RESPONSE TO NITROGEN, PHOSPHORUS, SULFUR, AND  
ZINC SUPPLIED BY MUNICIPAL BIOSOLIDS

by  
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Dean of Graduate School

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Todd E. Shearin, Author

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# WINTER WHEAT RESPONSE TO NITROGEN, PHOSPHORUS, SULFUR, AND ZINC SUPPLIED BY MUNICIPAL BIOSOLIDS

## INTRODUCTION

For over a quarter of a century, land application of biosolids (formerly called sewage sludge) has been a desirable recycling alternative. Due to improvements in the reduction of heavy metal concentrations in wastewater, land application has become an economically viable option for increasing numbers of wastewater treatment plants. Current economics make it feasible for large western Oregon wastewater treatment plants to transport and apply biosolids in north-central Oregon.

Agronomic biosolids applications are usually based on supplying the crop with adequate nitrogen (N) without excessive risk of nitrate losses via leaching. In addition to providing crops with N, biosolids supply organic matter and other plant nutrients. The addition of organic matter can be especially beneficial in traditional wheat-fallow cropping systems and low organic matter soils common to north-central Oregon. The application of additional nutrients can increase soil fertility levels and possibly correct micronutrient deficiencies.

In order for biosolids applications to be economically desirable for wheat farmers, grain yields must be equivalent to or greater than those produced with traditional N fertilizer. Likewise, grain quality must be maintained. Biosolids managers must be able to provide evidence to farmers that adequate N was supplied by biosolids applications. An N monitoring procedure that is easily performed and does not require large capital investments is needed.

Investigation of biosolids land application to dryland wheat systems in the Pacific Northwest can be of benefit to biosolids managers. These benefits include determination of agronomic rates, reliable estimates of N availability, and cost-effective procedures to assess the nutritional status of a dryland soft white winter wheat crop.

CHAPTER 1

LITERATURE REVIEW

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## BIOSOLIDS AND LAND APPLICATION

### History and Regulations

Biosolids are the stabilized solids produced from municipal wastewater treatment. This wastewater treatment by-product must meet federal criteria in order to be suitable for land application. Land application of biosolids is regulated by the United States Environmental Protection Agency (USEPA) under the guidance of The Standards for the Use or Disposal of Sewage Sludge (Title 40 of the Code of Federal Regulations [CFR], Part 503) which became effective on March 22, 1993 (USEPA, 1994).

Biosolids are considered an inexpensive nutrient and organic matter source. Land application is a practical recycling and management alternative which utilizes this valuable resource (Oberle and Keeney, 1994; Forste, 1997; Sullivan, 1998). About 50% of the biosolids produced in the United States is being land applied to "beneficially... improve soils", rather than being disposed of in landfills. (USEPA, 1994). Successful land application of biosolids has occurred in Oregon for the past 20 years. Due to current economics, long distance transport to central and eastern Oregon is a viable option for large, western Oregon wastewater treatment facilities. Biosolids application rates are usually based on supplying adequate nutrients for the crop, without excessive nitrate losses via leaching. The application rate of biosolids that substitutes for normal N fertilization practices is known as the agronomic rate.

Biosolids must meet Federal (USEPA) requirements for trace element content, pathogen reduction, and vector attraction reduction. The trace element concentration limitations are based on 20 years of field research and extensive risk assessment (Ryan, 1994; Muchovej and Pacovsky, 1997; Sullivan, 1998). Depending on the quality of biosolids, records of the cumulative levels of pollutants (trace elements) added to land may or may not be required by the state or federal government (Bastian, 1994; USEPA, 1994). If any of the regulated trace metal concentrations are above the Ceiling Limit concentration, the biosolids cannot be legally land applied.

## Benefits of Land Application

Land application of biosolids can improve soil quality by increasing water-holding capacity, improving tilth, reducing soil erosion, increasing cation exchange capacity (CEC), correcting micronutrient deficiencies, and increasing soil biotic (earthworms, microbes, etc...) activity (Muchovej and Pacovsky, 1997; Sullivan, 1998; Havlin, 1999). Soil organic matter is especially beneficial to the wheat-fallow system, where losses of carbon (C) and N can be extreme. In the wheat-fallow rotation, crop residue (straw) is only produced every other year. The reduced vegetative production leads to greater risks of soil erosion and soil organic matter oxidation by microbes (Rasmussen and Parton, 1994). Rasmussen and Parton (1994) reported that soil organic matter in a Walla Walla silt loam (Pendleton, OR) continued to decline with time in a wheat-fallow rotation, except for a feedlot manure application treatment. Results for manure treatments (average 100 kg N/ha/crop) showed that organic C levels in the soil surface (0-30 cm depth) remained at about 45 Mg/ha while organic N levels increased from 3.4 to 3.8 Mg/ha. Surface organic N levels in unfertilized treatments continued to decrease throughout the study following a linear trend. Organic C and N levels in the subsurface (30-60 cm depth) remained the same for unfertilized and manure amended treatments. Other studies have shown that the addition of manure (22 Mg/ha) to a conventionally tilled wheat-fallow system increased soil organic matter (Havlin et al., p. 447, 1999). In addition to the increased soil organic matter, grain yields were higher than with addition of traditional N fertilizer (45 kg N/ha).

## Field Studies

In order for biosolids to be land applied at agronomic rates, reliable estimates of N availability are required. Field trials with anaerobically-digested, dewatered biosolids in central Washington, showed that 26 to 31 % of the applied biosolids N was recovered in the fall of the fallow year as plant available N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ), 9 to 12 months after application (Cogger et al., 1998). The lowest biosolids application rate in the study ( $\cong$

340 kg N/ha) produced grain yields equivalent to the anhydrous ammonia treatment (56 kg N/ha); however, higher grain protein was observed with biosolids applications. Biomass (grain + straw) N uptake increased with higher biosolids rates. All biosolids treatments had significantly higher grain N uptake values as compared to the anhydrous ammonia control. During the second fallow sampling, there was evidence of significantly higher amounts of nitrate recovered at soil depths greater than 90 cm from the high biosolids rates. This indicated that downward movement (transport) of nitrate had occurred, although precipitation was below the 30-year average. The authors stated that the agronomic rate was at some rate less than the lowest rate ( $\cong$  340 kg biosolids N/ha), since soil and plant data indicated higher amounts of available N from biosolids than from anhydrous ammonia.

In dryland hard red winter wheat management systems in Colorado, Lerch et al. (1990a) recommended that 7 Mg sludge/ha ( $\cong$  140 kg N/ha) be used as the maximum safe loading rate. This rate increased available soil N while minimizing the potential of  $\text{NO}_3\text{-N}$  leaching to groundwater. This application rate resulted in adequate plant N levels, with increased grain protein levels. In contrast to soft white winter wheat management systems, high grain protein is desired for hard red winter wheat (Lerch et al., 1990b). Although yields were not increased with sludge application, the premium prices obtained for grain protein content ( $> 12\%$ ) resulted in an additional \$112/ha of increased revenue per year in comparison to N fertilizer alone.

Other researchers have looked at estimating first-year N net mineralization from biosolids. Barbarick et al. (1996) found that 27-62% of biosolids (combination of liquid and sand-bed dried) N applied was recovered as total soil N following 11-yr of application to hard red winter wheat. They estimated first-year N net mineralization rates of 13-43% and 41-67% for 6.7 Mg biosolids/ha and 26.8 dry Mg biosolids/ha application rates, respectively.

## Nitrate Transport

Another major concern of biosolids applications is preventing excessive transport of NO<sub>3</sub>-N deep in the soil profile and possible leaching into groundwater (USEPA, 1994; Bastian, 1994; Pierzynski, 1994). Although precipitation in central Oregon is low, transport of NO<sub>3</sub>-N below the root zone could potentially cause problems in the future. Movement of NO<sub>3</sub>-N below the root zone also constitutes a wasted resource for wheat farmers in the dryland production areas of the Pacific Northwest. Evans et al. (1994) found that NO<sub>3</sub>-N leaching in dryland agroecosystems (mainly wheat-fallow or sorghum) was not a major concern for any landscape position or climatic zone on cultivated soils in Colorado. In contrast, Bauder et al. (1993) reported that summer fallow practices associated with dryland farming (and subsequent mineralization of organic-N) were most likely contributing to localized NO<sub>3</sub>-N contamination of shallow groundwater in Montana. The authors also reported that 50-60% of the variability in groundwater NO<sub>3</sub>-N concentrations from private drinking wells was associated with soil texture and summer fallow practices.

## Laboratory Studies

In addition to field studies, numerous lab incubation studies have been completed to estimate availability of the organic N fraction of organic wastes. Douglas and Magdoff (1991) found that 23-41% of the organic N supplied by anaerobically digested sewage sludge was mineralized during 67 days of laboratory incubation at 25° C. The amount of N mineralized was significantly correlated with numerous chemical indices. The Walkley-Black N index appeared to be the most useful. Lindemann et al. (1988) reported that 35% of the organic N applied (as anaerobically digested, air-dried sewage sludge) was recovered as inorganic-N during 16 weeks of incubation. In soils previously amended with sludge, they found that 21-22% and 34-35% of the organic-N was recovered as inorganic forms for non amended and sludge amended soils, respectively. The authors also reported that the mineralization curves failed to flatten out after 16

weeks. In other studies, N availability from sludge (including anaerobically digested, dewatered sludge and aerobically digested sludge) ranged from 15-40% of the organic N applied (King, 1984; Parker and Sommers, 1983; Garau et al., 1991). Incubation times ranged from 5 (Garau et al., 1991) to 16 weeks (King, 1984; Parker and Sommers, 1983). Also, lab incubations are typically "best-case" scenarios, with optimum temperature and moisture content. This is rarely the case in field applications, especially in the dryland wheat-fallow systems of north-central Oregon and eastern Washington.

## **ASSESSMENT OF PLANT NUTRITIONAL STATUS**

### Nitrogen Availability

Biosolids manager goals are to operate an environmentally friendly facility, while providing farmers with a valuable plant nutrient resource (Forste, 1997). Biosolids managers need to provide evidence that adequate N was supplied by biosolids, and that the N supply was not excessive (Bastian, 1994). Postharvest soil samples can be taken to assess that N was not provided in excess of crop requirements (Sullivan et al., 1993; USEPA, 1994). Monitoring of N availability is necessary for internal management purposes and also for cooperating growers who receive the biosolids.

### Soil Sampling

Soil samples are an important tool in the assessment of inorganic-N. The number of subsamples composited for a soil sample depend on the accuracy and precision desired (Dahnke and Johnson, 1990).

Soil samples are relatively small in relation to the area being investigated, and require special sampling equipment (James and Wells, 1990; Rhoades and Miyamoto, 1990). Soil analyses can estimate the amount of available soil N, since not all available soil N is utilized by the crop (Fiez et al., 1995; Kjelgren, 1984).

The pre-sidedress soil nitrate test (PSNT) is a valuable tool in the assessment of early season N mineralization in fields amended with manure (Marx, 1995; Sims, 1995). Samples for PSNT are collected prior to the rapid N crop uptake period. PSNT results are used to assess the need for additional fertilizer N. The PSNT is a preventive tool, since additional N could be applied via sidedressing, if necessary. Similar to the PSNT, soil samples can be collected prior to the rapid growth of the crop in wheat-fallow cropping systems and analyzed to estimate the amount of plant available N supplied by biosolids. Soil samples can also be collected following grain harvest. This type of sampling would not provide evidence of available N during the growing season, but rather evidence of available N supply in excess of crop demand (Stanford, 1982).

### Plant Tissue Sampling

Plant tissue testing is another approach to assess available N supply relative to crop needs. Although soil testing is an accepted method for estimating the amount of available soil N supplied by biosolids, it is best used in conjunction with plant tissue analyses (Havlin, 1999).

In contrast to soil sampling, plant tissue sampling does not require any special equipment or long training periods (Havlin et al., 1999; Jones and Case, 1990). Another benefit of plant tissue sampling is that the possibility of contamination is minimal in comparison to soil sampling (James and Wells, 1990; Jones and Case, 1990). In contrast to the small area that a soil sample represents in terms of the total plot, a single plant sample can represent approximately 0.03% of the total plot, using estimates for dryland winter wheat root area (Havlin et al., 1999). Since N is a mobile nutrient, sampling the youngest, fully developed leaf will give the best representation of plant N status (Munson and Nelson, 1990). Sampling very mature leaves provides lower N concentrations and sampling immature leaves will not give an accurate prediction of the plant's nutritional status. Sampling a specific leaf is desirable because it is quick, easily obtained, and easily repeatable throughout the field in a short period of time; whereas, sampling the entire above ground portion of the plant is time consuming and labor intensive, and generally

not recommended (Jones and Case, 1990). It must be considered however, that tissue sampling is only an indication of nutritional status at the time of sampling (Havlin et al., 1999).

In contrast to the PSNT, end-of-season tissue tests have been evaluated to determine the supply of available N (Hooker and Morris, 1999). The end-of-season corn stalk test determines if optimal or above-optimal available N was supplied by manure applications. Early season leaf tissue sampling has been used to estimate the spring N requirements based on N concentrations (Vaughn et al., 1990a; Papastyliano et al., 1984). This practice is a valuable management tool but is based on an estimation of yield potential. The early season leaf tissue samples are collected prior to the rapid growth of the crop and any inclement weather during the period of rapid growth would decrease the yield potential.

## **DEVELOPMENT OF CRITICAL NUTRIENT CONCENTRATIONS (CNC) FOR PLANT TISSUE**

### Plant Tissue Nitrogen Concentration Calibration

Previous studies have correlated plant tissue N concentrations with maximum yield response. The N concentration that correlates with maximum yield, or 90 to 95% of maximum yield, is considered the critical nutrient concentrations (CNC) level. A wide range of critical nutrient concentrations have been reported for wheat, because the CNC depends on physiological leaf age, wheat variety, and climate.

As a plant matures, N concentrations in the tissue decline (Hanway, 1962; Havlin et al., 1999). The decline in N concentrations is due to increased biomass accumulation and decreased N uptake. There are three distinct phases of N uptake for a winter wheat crop: (i) slow uptake, (ii) rapid uptake, and (iii) redistribution within the plant (Sullivan et al., 1999). For winter wheat, the rapid N uptake period begins at tillering (Feekes 5) and ceases with head emergence (Feekes 10). During the last stage, N is redistributed

within the plant (from leaves to grain) and coincides with continued biomass accumulation.

Wheat variety plays a role in N fertilization goals (Munson and Nelson, 1990; Karow, 1994). In hard red wheat management systems, excess N is desired for increased protein levels. N is normally applied at rates above that which is required for maximum yields. In this scenario, luxury consumption of N occurs. Luxury consumption occurs when plant uptake of a nutrient is in excess of plant physiological needs. The increase in nutrient concentrations above the level required for maximum yield, is common in most plants (Engels and Marschner, 1995; Havlin, 1999) exposed to overfertilization. Luxury consumption increases the N concentration in the grain protein without increasing yield production. In contrast to the hard red wheat systems, soft white winter wheat production systems are managed for low protein. This means that excess N for maximum grain yields is not desired. These differences will consequently have implications on the critical nutrient concentration (CNC) reported for different wheat varieties. McNeal et al. (1966) found significant differences in the leaf dry matter contents of various wheat varieties but only slight differences in leaf N uptake. The leaf N concentrations ranged from 3.2 to 4.0% N at heading (Feekes 10) and 3.0 to 3.5% N at flowering (Feekes 10.5-10.51). Guy et al. (1995) investigated cultivar responses to crop management systems in northern Idaho. Flag leaf N concentrations were higher for an early season maturity soft white wheat ('Malcolm') in comparison to late season maturity soft white wheat ('Lewjain' and 'Kmor') and hard red wheat ('Weston'). This difference was only observable at one location in their experiment for the second year, suggesting that the climatic differences observed between the two locations during the 1992 season influenced leaf N concentrations.

Climatic differences between locations influence the critical nutrient concentrations reported for winter wheat. In central Oregon, the most limiting factor for grain yield is moisture. Plant tissue N concentrations corresponding with maximum yield in dryland cropping systems are expected to be lower than those observed in areas which are irrigated (or not as severely moisture limited). Vaughn et al. (1990a) reported that discrepancies between reported critical N levels could be due to different geographical



regions. They reported that for soft red winter wheat at Feekes 4, the critical whole plant N value ranged from 4-5% in Virginia, to above 4% in Pennsylvania.

Researchers also use different methods for estimating critical nutrient concentrations. The most common method employed by researchers is to correlate plant tissue N concentrations with near-maximum grain yields (90-100% of maximum). This is the approach taken to calculate a CNC in a soil test calibration. This method relates the level of a nutrient to the response of crop yield, quality, or performance (Havlin et al., 1999). To determine the CNC, yield response or relative yield (a percentage of the maximum yield) is plotted (y-axis) versus the tissue nutrient concentration (x-axis). The CNC is usually estimated at the concentration that equates with 90% of the maximum yield. Donohue and Brann (1984) and Papastyliano et al. (1984) used a similar regression analysis to determine the CNC for winter wheat which correlated with maximum grain yields. The authors fit quadratic regression lines to the relationship of relative percent maximum yield (y-axis) and N concentration in the plant tissue (x-axis).

For dryland hard red winter wheat grown in Colorado, leaf N concentrations of 3.4 to 4.3% at Feekes 5 corresponded to maximum grain yields (Follett et al., 1992). Their objectives were to correlate leaf N concentrations, soil N tests, and chlorophyll meter readings to crop N status and N fertilizer recommendations.

Other researchers in eastern Colorado have used grain protein content as an indicator of N sufficiency for hard red winter wheat, due to the significant correlation between grain N and grain yields (Goos et al., 1982). The authors objectives were to determine a the levels of grain protein needed for obtaining maximum yields. They found that grain protein levels of 12.0% or greater indicated sufficient N for maximum yields. In addition to determining the point where N was not limiting for maximum yields, they determined a value below which N was deemed insufficient for maximum yields (11.1%). Since grain protein is highly dependent on water stress, rainfall distribution, and temperature, regardless of soil N availability, a linear relative yield-protein relationship did not exist. This means that grain protein can not be used to predict future N fertilizer needs, but rather, could be used as an indicator of sufficient N for maximum yields using previous grain yield data.

Additional research has been completed on flag leaf N concentrations and grain yields for soft red winter wheat in the southeastern states. Work in the Coastal Plain of Virginia found that flag leaf N concentrations of 4.4 to 4.7% at head emergence (Feekes 10) was closely related to 100% of maximum yield for soft red winter wheat (Donohue and Brann, 1984). Research in Georgia on soft red winter wheat (*Triticum aestivium*) found that flag leaf N concentrations of 3.5 to 4.0% at flowering (Feekes 10.5) related to maximum grain yields (Hargrove, et al., 1983).

Research has also been conducted for irrigated, hard red spring wheat in the western states. Tindall et al. (1995) found that flag leaf N concentrations of 3.7 to 4.3% at heading (Feekes 10) for hard red wheat grown in Idaho correlated to maximum grain yields. They also found that flag leaf N concentrations greater than 4.2% showed no beneficial grain protein response from topdressed N applied after complete head emergence. For flood irrigated wheat grown in California, flag leaf N concentrations of 2.9%, sampled 14 days after flowering, corresponded to maximum grain yields (Wuest and Cassman, 1992). The authors also found that increasing N rates did not increase grain yields, but increased flag leaf N concentrations.

Others have attempted to correlate spring N fertilization requirements with plant tissue N concentrations. Dryland hard red winter wheat leaf nitrogen concentrations at tillering (Feekes 5) were used to determine spring N fertilization recommendations for the western Great Plains Region (Vaughn, et al. 1990a; Vaughn, et al. 1990b). Vaughn et al. (1990a) reported that hard red winter wheat leaf N concentrations of 3.8% and 3.5% were critical concentrations for Feekes 5 and Feekes 7 growth stages, respectively

### Nondestructive Methods

Nondestructive methods such as chlorophyll meters, have been correlated with leaf N and response to applied N (Turner and Jund, 1991; Follett et al., 1992; Piekielek and Fox, 1992). Piekielek and Fox (1992) found that the correlation of yields to chlorophyll meter readings were heavily dependent on the leaf development stage and age. The chlorophyll meter readings were most useful in conjunction with dry matter

yield determination and/or soil nitrate tests for assessment of soil N availability (Reeves et al., 1993). Follett et al. (1992) found a positive correlation between chlorophyll meter readings and hard red winter wheat leaf N concentration (at Feekes 5); further studies were required in order to evaluate other factors such as cultivar, site, soil moisture, and available soil N differences.

## **OTHER NUTRIENTS SUPPLIED BY BIOSOLIDS**

### Phosphorus

In addition to supplying N, biosolids applications supply phosphorus (P). Concentrations of P in biosolids (and sewage sludge) range from 0.8 to 3.4% (Taylor et al., 1978; Lerch et al., 1990a; Diez et al., 1992; Barabrick et al., 1997). Typical concentrations are 1.5 to 3.0% (Sullivan, 1998). Studies have shown that when organic wastes are applied at agronomic N rates, excess P is supplied (Pierzynski, 1994; Edwards, 1997). When sewage sludge was applied at agronomic N rates, excess P was supplied at a rate of approximately three times the amount required by the crop (Pierzynski, 1994). The author stated that application rates of 200 kg plant available N/ha resulted in a two-fold increase in Bray-1 P soil test values, when compared to the unfertilized control. Edwards (1997) reported that addition of organic wastes (in the forms of poultry litter and animal manure) at agronomic N rates produced 300 to 500% increases in P soil test values following three years of application. To alleviate the increase in soil test P levels, researchers have investigated the use of aluminum sulfate (alum) to decrease the availability of P compounds in organic wastes. The addition of alum proved effective in decreasing the amount of extractable P from soils amended with organic wastes (Peters and Basta, 1996) and in the waste itself (Moore and Miller, 1994).

Barbarick et al. (1995) found that in a soil with high to very high P levels, the application of additional P (via biosolids) did not increase plant absorption. Sims (1990) reported that increasing application rates of cocomposted sewage sludge (combination of aerobic and anaerobically digested sewage sludges) increased soil test P (Mehlich I) from

low to medium values, based on University of Delaware guidelines. Soil test P values were increased from 10 mg/kg to 25 mg/kg, with application rates of 44 Mg/ha of cocomposted sewage sludge. An application rate of 2 Mg/ha provided 90 kg total P/ha, an excess of 70 kg P/ha for non-irrigated corn, based on Delaware fertilizer guides. Although increases in the P soil test values were reported, no increases in plant P concentrations were observed. Taylor et al. (1978) found that extractable P levels measured immediately after compost addition to sand did not increase in proportion to the amount of compost added.

## Sulfur

Biosolids also supply sulfur (S). The S contained in biosolids is present in numerous organic and inorganic forms (Sommers et al., 1977; Elseewi et al., 1978; Schaumberg et al., 1982; Bohn et al., 1986; Freney, 1986). Typical S concentrations in biosolids range from 0.6 to 1.3% (Sullivan, 1998).

The amount of S required by most crops is small in relation to the amount of N required (Oregon State University, 1980; Havlin, 1999). S fertilization is second only to N in application volume and frequency to cereals in the Pacific Northwest (Rasmussen and Kresge, 1986). S has been shown to be deficient in Oregon, due to the low levels in the soils, and fertilization of grain crops has been recommended (Western Fertilizer Handbook, 1985; Brengle, 1982). Studies in the Pacific Northwest showed that when S was applied with excessive N application rates, straw production was increased but grain yield was not (Rasmussen and Kresge, 1986). Smith (1962) found that the yield decrease associated with excessive N fertilization was eased with the addition of S in above-average precipitation years, but was magnified when precipitation was below average.

The main form of plant available S is sulfate ( $\text{SO}_4\text{-S}$ ) (Salisbury and Ross, 1985; Thompson et al., 1986; Havlin, 1999). Because  $\text{SO}_4\text{-S}$  is an anion, it behaves similarly to  $\text{NO}_3\text{-N}$  in the soil profile (Brady and Weil, 1999; Havlin, 1999). Due to the complexes formed with iron (Fe) and aluminum ( $\text{Al}^{3+}$ ) oxides, the ability of soils to adsorb  $\text{SO}_4\text{-S}$  is much stronger than  $\text{NO}_3\text{-N}$ .

The use of soil test S values to make fertilizer recommendations is limited due to the effects of soil properties and pH on  $\text{SO}_4\text{-S}$  adsorption and leaching (Duke and Reisenauer, 1986). Gavlak et al. (1994) reported that extracting soil  $\text{SO}_4\text{-S}$  with calcium phosphate ( $\text{Ca}(\text{H}_2\text{PO}_4)_2$ ) and subsequent determination with an ion chromatograph was comparable to the methylene blue method described by Dick and Tabatabai (1979). The advantage of the ion chromatograph method was the increased precision and accuracy at lower concentrations. Plant tissue tests for S include total S, soluble S, and protein S (Blanchar, 1986).

## Zinc

In addition to supplying N, P, and S, biosolids applications also supply zinc (Zn). Concentrations of Zn in biosolids range from 618 to 2540 mg Zn/kg biosolids (Lerch et al., 1990a; Sims, 1990; Barbarick et al., 1998).

Zn is required in small quantities by most crops (Murphy and Walsh, 1972; Lindsay and Norvell, 1978; Kabata-Pendias and Pendias, 1992; Havlin et al., 1999). Deficiencies are generally observed on alkaline, calcareous soils with low amounts of organic matter (Lucas and Knezek, 1972; Kabata-Pendias and Pendias, 1992). In dryland cropping, N fertilization can induce Zn deficiency in cereals on soils with marginal levels of Zn (Brengele, 1982).

Zn is relatively immobile in soils when sludge is applied, due to the high amounts of organic matter and P compounds present in the sludge amendment (Kabata-Pendias and Pendias, 1992). Soil testing of extractable Zn has been investigated and correlated with crop response to Zn fertilization (Lindsay and Norvell, 1978). Lerch et al. (1990a) reported that the addition of biosolids (751-2540 mg Zn/kg sewage sludge) to dryland winter wheat fields in Colorado resulted in an increase in the DTPA-extractable soil test Zn levels. In the same study, increased biosolids rates resulted in small increases in grain Zn concentrations (Barbarick et al., 1995). Sims (1990) found that the application rate of co-composted sewage sludge had no effect on the Zn concentrations of whole wheat plants at harvest.

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## CHAPTER 2

## AGRONOMIC RATES OF BIOSOLIDS FOR DRYLAND WHEAT PRODUCTION

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## ABSTRACT

Biosolids application rates are based on supplying the crop with adequate N. Previous studies have focused on rates of biosolids greater than 336 kg N/ha. The main objectives of this research were to determine: (i) the agronomic rate of biosolids application for dryland wheat production based on grain yield and quality response, (ii) the amount of available soil N ( $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$ ) under dryland conditions, and (iii) residual soil fertility status (specifically P, S, and Zn) under dryland conditions. Anaerobically-digested and dewatered biosolids (17% dry matter, 83% water) were supplied by the Unified Sewerage Agency (USA) of Washington County, OR. Three rates of biosolids (low, medium, and high) were applied in the fall after crop harvest and in the spring prior to the first fallow tillage at two locations in Sherman County, OR. The biosolids applications were compared to an anhydrous ammonia control (56-67 kg N/ha) and an unfertilized control. An application rate of 230-370 kg biosolids N/ha (5.2-7.6 Mg biosolids/ha) maintained or increased grain yields as compared with those achieved with anhydrous ammonia, with no decrease in grain quality. The amount of available soil N from biosolids application (sampled spring of the crop year) ranged from 15-25% of the total biosolids N applied. Biomass N uptake values indicated that 10-11% of the total biosolids N was recovered in the above ground biomass of the first crop after application. Postharvest soil sampling showed no significant increase in available soil N at either location. Biosolids increased Bray-1 P soil test values (0-15 cm) and DTPA-extractable Zn (0-15 cm). Approximately 14% of the total S applied was recovered as  $\text{SO}_4\text{-S}$  in the soil profile during fallow at one location. Biosolids appeared to be a good nutrient source for winter wheat based on plant and soil responses. Future research focusing on long term effects of biosolids on residual soil fertility is needed.

## INTRODUCTION

Land application of biosolids has occurred in Oregon for the past 20 years. Due to current economics, long distance transport to central and eastern Oregon is a viable option for large, western Oregon wastewater treatment facilities. Biosolids application rates are based on supplying adequate nitrogen (N) for the crop, without excessive nitrate losses via leaching. The application rate of biosolids that substitutes for normal N fertilization practices is known as the agronomic rate.

In order for biosolids applications to be advantageous to farmers, grain yields must be as high as production rates obtained using traditional fertilizer, with no decreases in grain quality. Field trials with anaerobically-digested, dewatered biosolids in central Washington showed that application rates of approximately 340 kg biosolids total N/ha (lowest rate) to soft white wheat produced grain yields equivalent to the anhydrous ammonia treatments (56 kg N/ha), but with higher grain protein than the anhydrous ammonia (Cogger et al., 1998). Soil analyses revealed that 26 to 31 % of the applied biosolids N was recovered as plant available inorganic N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) 9 to 12 months after application, in the fall of the fallow year. Although precipitation was below the 30-year average, application of the high biosolids rates increased the amounts of nitrate recovered during the second fallow sampling in the deeper depths (> 90 cm). The agronomic rate was less than the lowest rate used in the study ( $\cong$  340 kg biosolids N/ha). Soil and plant data indicated higher amounts of available N from the lowest rate of biosolids than from anhydrous ammonia. In dryland hard red winter wheat management systems in Colorado, Lerch et al. (1990a) recommended that 7 Mg sludge/ha (140 kg N/ha) be used as the maximum safe loading rate. This rate increased available soil N while minimizing the potential of  $\text{NO}_3\text{-N}$  leaching to groundwater. This application rate resulted in adequate plant N levels, with increased grain protein levels. In contrast to soft white winter wheat management systems, high grain protein is desired for hard red winter wheat (Lerch et al., 1990b). Although yields were not increased with sludge application, the premium prices obtained for grain protein content (> 12%) resulted in an additional \$112/ha of increased revenue/year compared to N fertilizer alone.

In addition to field studies, numerous lab incubation studies have been completed to estimate availability of the organic N fraction of organic wastes. Typical first year mineralization estimates range from 15-40%, depending on incubation time and biosolids production processes (Douglas and Magdoff, 1991; Lindemann et al., 1988; King, 1984; Parker and Sommers, 1983; Garau et al., 1991). Use of the conservative end of the range would overestimate the N availability and could lead to under-application of biosolids. Also, lab incubations are typically "best-case" scenarios, with optimum temperature and moisture content. This is rarely the case in field applications, especially in the dryland wheat-fallow systems in central Oregon and eastern Washington.

The major concern besides supplying the crop with adequate N is preventing excessive  $\text{NO}_3\text{-N}$  leaching into groundwater. Although precipitation in north-central Oregon is low, transport of  $\text{NO}_3\text{-N}$  below the root zone could potentially cause problems in the future. Movement of  $\text{NO}_3\text{-N}$  below the root zone also constitutes a wasted resource. Evans et al. (1994) found that  $\text{NO}_3\text{-N}$  leaching in dryland agroecosystems (mainly wheat-fallow or sorghum) was not a major concern for any landscape position or climatic zone on cultivated soils in Colorado. In contrast, Bauder et al. (1993) reported that summer fallow practices associated with dryland farming (and subsequent mineralization of organic-N) were most likely contributing to localized  $\text{NO}_3\text{-N}$  contamination of shallow groundwater in Montana. The authors also reported that 50-60% of the variability in groundwater  $\text{NO}_3\text{-N}$  concentrations from private drinking wells was associated with specific soil types and summer fallow practices. Accumulation of  $\text{NO}_3\text{-N}$  in the soil profile can also increase grain protein. Cochran et al. (1978) reported that N accumulation deep (> 60 cm) in the soil profile prior to the rapid growth of wheat resulted in increased grain protein levels. Deep placement of N was achieved by broadcasting calcium nitrate [ $\text{Ca}(\text{NO}_3)_2$ ] in the fall and allowing winter precipitation to transport  $\text{NO}_3\text{-N}$  to depths deeper than 60 cm. Even though annual precipitation ranged from 20-37 cm, the amount was sufficient to transport  $\text{NO}_3\text{-N}$ .

In addition to supplying N, biosolids applications also supply sulfur (S). S fertilization is second only to N in application volume and frequency to cereals in the Pacific Northwest (Rasmussen and Kresge, 1986). Studies in the Pacific Northwest showed that when S was applied with excessive N application rates, straw production



increased but grain yield did not (Rasmussen and Kresge, 1986). Smith (1962) found that the yield decrease associated with excessive N fertilization was eased with the addition of S in above-average precipitation years, but was magnified when precipitation was below average.

The present research evaluated agronomic rates of biosolids for soft-white wheat production in the 25-35 cm precipitation zone of north-central Oregon. The objectives of this research were to determine the effects of a one time biosolids application on: (i) grain yield and quality, (ii) the amount of available soil N, and (iii) residual soil fertility status (specifically P, S, and Zn).

## MATERIALS AND METHODS

### Field Study

Field locations were located in Sherman County, Oregon. Pinkerton Farm (P-95) was located in the SE  $\frac{1}{4}$ , SE  $\frac{1}{4}$  Section 33, T1N, R17E, 5 km north of Moro on Hwy 97. McClennan farm (M-96) was located in the SW  $\frac{1}{4}$ , SW  $\frac{1}{4}$  Section 16, T1N, R17E, 3 km south of Wasco on Hwy 97. Location designation is referred to as the first letter of the farm and year of first biosolids application. Soils were mapped as Walla Walla silt loams (coarse-silty, mixed, superactive, mesic Typic Haploxeroll) and averaged 75-100 cm deep at P-95 and > 120 cm at M-96. The cooperating farmers performed routine tillage and management practices associated with a typical wheat-fallow rotation.

P-95 had winter wheat harvest in 1995 (prior to our study) and in 1997 (completion of our study). Common club wheat *Triticum aestivum* (Rohde) was seeded in early October 1996. M-96 had winter wheat harvest in 1996 (prior to our study) and in 1998 (completion of our study). Common soft-white wheat *Triticum aestivum* (Stephens/Madsen mix) was seeded in late September 1997.

The experiment was constructed in a randomized complete block design and consisted of eight treatments replicated 3 times at P-95 and 4 times at M-96. The biosolids applications were compared to an anhydrous ammonia and an unfertilized

control. At M-96, biosolids and unfertilized plots measured 12 by 106 m and anhydrous ammonia plots measured 18 by 106 m (in order to accommodate the anhydrous ammonia applicator). All plots at P-95 measured 12 by 91 m.

Three biosolids rates (low, medium, and high; Table 2.1) were applied using a rear-delivery manure spreader equipped with a hydraulic ram. Biosolids treatments were applied in the fall after crop harvest and in the spring prior to the first fallow tillage. The interval between biosolids application and the first fallow tillage was about 6 months for the fall application and about 1 month for the spring application. These application dates represent the most practical application times for biosolids in a wheat-fallow cropping system.

Rainfall and temperature data (growing degree days) for the time period from the first biosolids application to the postharvest soil sampling are presented in Appendix A. Climate data was obtained from the Moro Experiment Station, located approximately 5 and 10 km away from P-95 and M-96, respectively.

### Biosolids Source

Biosolids (anaerobically-digested and dewatered; 17% dry matter, 83% water) were supplied by the Unified Sewerage Agency (USA) of Washington County, Oregon. Biosolids trace element concentrations met federal requirements for land application (Table 2.2). All metal concentrations were below Exceptional Quality limits. Therefore, trace elements did not limit cumulative biosolids application amounts. ICP analysis of selenium (Se) and mercury (Hg) was inconclusive due to the high detection limits. Analyses performed by the wastewater treatment plant reveal that levels of Se and Hg were below Exceptional Quality limits (Table 2.2).

The composition of the biosolids varied somewhat from fall to spring, because the wastewater treatment process changed seasonally. Biosolids produced in the fall were a combination of solids from primary, secondary, and tertiary wastewater treatment. Tertiary wastewater treatment removed additional phosphorus (P) from the wastewater using alum (aluminum sulfate). The high-P residuals from tertiary treatment became part

**Table 2.1.** Nutrient application rates and timing. Pinkerton Farm (P-95) and McClelland Farm (M-96), Sherman County, OR. 1995-1997.

Location	Treatment <sup>a</sup>	Application Date	Biosolids Rate Mg/ha	Total Nutrients Applied <sup>b</sup>					
				NH <sub>4</sub> -N	N	P	S	Zn <sup>c</sup>	
				-----kg/ha-----					
P-95	No fertilizer	-	0	0	0	0	0	0	
	AA	03-Jul-96	0	0	56	0	0	0	
	BS1-Fall	06-Nov-95	3.1	42	151	97	33	1.5	
	BS2-Fall		5.2	71	248	162	55	2.5	
	BS3-Fall		9.9	136	474	309	104	4.8	
	BS1-Spring	23-Apr-96	3.1	52	165	70	30	1.8	
	BS2-Spring		5.2	87	270	117	50	3.1	
	BS3-Spring		9.9	166	517	223	96	5.9	
	M-96	No fertilizer	-	0.0	0	0	0	0	0
		AA	09-Jun-97	0.0	0	67	0	0	0
BS1-Fall		16-Oct-96	3.8	45	160	131	40	2.0	
BS2-Fall			7.6	90	320	261	79	3.9	
BS3-Fall			11.4	136	480	392	119	5.9	
BS1-Spring		25-Apr-97	3.8	52	191	101	40	2.1	
BS2-Spring			7.6	105	382	202	80	4.3	
BS3-Spring			11.4	157	572	303	120	6.4	

<sup>a</sup> AA=Anhydrous ammonia; BS=Biosolids applied to standing stubble the fall after crop harvest, or in the spring prior to the first Fallow tillage.

<sup>b</sup> Based on biosolids N, P, and S analyses (Materials and Methods) performed by AgriCheck, Inc., Umatilla, OR.

<sup>c</sup> Based on biosolids Zn analysis (Materials and Methods) performed by OSU Central Analytical Lab, Corvallis, OR.

**Table 2.2.** Trace element concentrations for biosolids used in agronomic trials in relation to USEPA standards. <sup>a</sup>

Element	Symbol	USEPA	USEPA	Site				USA <sup>d</sup>
		Exceptional Quality	Ceiling	P-95		M-96		
		Limit	Limit	Fall <sup>b</sup>	Spring <sup>b</sup>	Fall <sup>b</sup>	Spring <sup>b</sup>	
-----mg/kg-----								
Arsenic	As	41	75	< 8.0	< 8.0	8.2	< 8.0	9.2
Cadmium	Cd	39	85	< 2.0	2.4	< 2.0	2.4	1.8
Copper	Cu	1500	4300	569	546	590	499	573
Lead	Pb	300	840	24	37	24	26	74
Mercury	Hg	17	57	< 100	< 100	< 100	< 100	2.1
Molybdenum <sup>c</sup>	Mo	-	75	4.5	4.0	4.9	4.3	3.3
Nickel	Ni	420	420	17	24	21	24	22
Selenium	Se	100	100	< 200	< 200	< 200	< 200	3.7
Zinc	Zn	2800	7500	485	595	514	560	554

<sup>a</sup> Source: USEPA 40 CFR Part 503.

<sup>b</sup> Analysis performed by OSU Central Analytical Laboratory (Corvallis, OR) with ICP determination.

<sup>c</sup> Molybdenum exceptional quality limit and ceiling limit are under review by the USEPA.

<sup>d</sup> USA (Unified Sewerage Agency) data provided by USA laboratory (Rock Creek wastewater treatment plant). Average of monthly analyses, 1995-1997.

of the fall biosolids. In the spring, the biosolids contained only solids from primary and secondary wastewater treatment.

## Biosolids Sampling and Analysis

### *Biosolids Sampling*

At each biosolids application (06-Nov.-95, 23-Apr.-96, 16-Oct.-96, and 25-Apr.-97), three composite biosolids samples (2-L) were collected. Each 2-L sample was a composite of approximately twenty subsamples. Samples were refrigerated in the field and then frozen until analysis. Biosolids were dried and ground to pass through a 1 mm screen (except for the N analyses which were run on an 'as-is' basis).

### *Biosolids Analysis*

Total N, P, and S analyses were performed by AgriCheck, Inc. (Umatilla, OR). Total N was determined on wet samples by Kjeldahl distillation and titration with 0.1 N HCl (Gavlak et al., 1994). Digestion of dry samples using a nitric acid/hydrogen peroxide wet ashing and use of a Perkin-Elmer Optima 3000 DV Mass-spec ICP (Perkin-Elmer Corporation, Norwalk, CT) provided total P and S concentrations.

Arsenic (As), cadmium (Cd), copper (Cu), lead (Pb), mercury (Hg), molybdenum (Mo), nickel (Ni), selenium (Se), and zinc (Zn) analyses were performed by OSU Central Analytical Lab (Corvallis, OR). Trace metals were digested by adding 2.0 mL of 30% hydrogen peroxide and 0.5 mL of concentrated nitric acid to 0.25 grams of biosolids and digesting in a MDS-2000 microwave digester (CEM Corporation, Matthews, NC). Metal concentrations were determined with a Perkin-Elmer Optima 3000 DV Mass-spec ICP (Perkin-Elmer Corporation, Norwalk, CT).

## Soil Sampling and Analysis

### *Soil Sampling*

Soil samples for N analysis were collected in 30 cm increments in the fall of the fallow year, prior to rapid growth in the spring of the crop year, and following grain harvest. Soil profile sample depths were limited to 75 cm at P-95 due to the presence of a rocks in the profile. Samples at M-96 were collected to 150 cm. The samples at the end of the fallow year were collected manually with a 2.5 cm i.d. push probe (Arts Manufacturing, American Falls, ID). The other soil samples were collected with a hydraulic auger probe (Kauffman Mfg., Albany, OR) mounted on a small tractor. Soil samples were dried at 27°C, ground, and sieved to pass through a 2-mm sieve.

Surface soil samples (0-15 cm) were also collected for analysis of additional plant nutrients including P, Zn, potassium (K), calcium (Ca), magnesium (Mg), sodium (Na), boron (B), iron (Fe), manganese (Mn), and copper (Cu).

### *Soil Analysis*

NH<sub>4</sub>-N and NO<sub>3</sub>-N concentrations in the fallow, spring, and postharvest soil samples were extracted with 2 N KCl and analyzed with an ALPKEM rapid flow analyzer RF-300. The fallow (M-96) and 0-15 cm soil samples were analyzed for SO<sub>4</sub>-S by extracting with calcium phosphate, (Ca(H<sub>2</sub>PO<sub>4</sub>))<sub>2</sub>, and analyzing the extract on a Dionex 2000 ion chromatograph equipped with an AS4A anion exchange column (Horneck et al., 1989).

Additional analyses were performed on the surface soil samples using standard procedures (Horneck et al., 1989). Phosphorus was determined with the dilute acid-fluoride method (Bray-1) and analyzed with a ALPKEM rapid flow analyzer No. RFA-300. Boron was determined by extracting with calcium chloride, CaCl<sub>2</sub>, and analyzed on a Bausch and Lomb Spectronic 88 spectrophotometer set at 420 nm. Zinc, Mn, Cu, and

Fe were extracted with DTPA (diethylenetriaminepentaacetic acid) and analyzed on a Perkin-Elmer model 372 atomic absorption spectrophotometer. Potassium, Ca, Mg, and Na were extracted with ammonium acetate and analyzed on a Perkin-Elmer model 372 atomic absorption spectrophotometer.

In addition to these nutrients, the soil was also analyzed for pH, SMP pH, and soluble salts using standard procedures (Horneck et al., 1989). Surface soil pH was determined using a 1:2 soil to water ratio and the mixture pH read with a pH meter. Lime requirement (SMP pH) was determined by adding SMP buffer solution to dry soil and reading the pH value with a pH meter. Soluble salts were determined by measuring the electrical conductivity of a saturated soil-water paste with a Solu-Bridge Conductivity meter.

### Plant Sampling and Analysis

#### *Biomass Sampling*

Biomass (grain + straw) samples were hand-harvested from ten one-meter sections of row from each plot on 9 Jul 1997 at P-95 and from five one-meter sections of row from each plot on 15 Jul 1998 at M-96. The samples were allowed to air dry for approximately 14 days and then weighed to determine biomass yield.

After the weight of the biomass was collected, the samples were threshed using a small plot combine. Straw yield was calculated by subtracting the grain weight from the biomass weight. Grain harvest index was calculated with the following equation:

$$\text{Grain Harvest Index} = \left( \frac{A}{A + B} \right) \times 100 \quad [1]$$

where

$A$  = Grain yield (g)

$B$  = Straw yield (g)

The straw exiting the combine was collected by placing a tarp on the ground behind the combine. The straw collected during this procedure included the grain chaff. A small sub-sample (approximately 500 g) of the straw was collected, dried, and ground to pass through a 1-mm screen. The straw was then analyzed for nutrients and trace elements.

### *Grain Harvest*

Grain was harvested on 1 Aug. 1997 at P-95 and 24 Jul 1998 at M-96. Prior to harvest, an 8-m swath was removed from the end of each plot. This was done to avoid possible border effects. Grain was harvested from an 8-m swath from the center of each plot, resulting in 0.06 ha harvested at P-95 and 0.07 ha harvested at M-96. The grain was then transferred into a weigh wagon equipped with an electronic scale and the weight recorded. After the weight was recorded, the grain from the weigh wagon was augured into a grain truck, during which a 1 kg subsample was collected. The subsample was used to determine grain test weight and thousand kernel weight. For chemical analysis, grain was dried and ground to pass through a 1 mm screen.

### *Plant Tissue Analysis*

Plant tissue (grain and straw) C, N, and S were determined by Leco CNS 2000 (Leco Corporation, St. Joseph, MI) combustion analysis.

Plant samples for P, S, zinc (Zn), potassium (K), calcium (Ca), magnesium (Mg), manganese (Mn), iron (Fe), copper (Cu), boron (B), and sodium (Na) analysis were digested by adding 2.0 mL of 30% hydrogen peroxide and 0.5 mL of concentrated nitric acid to 0.25 grams of plant tissue and digesting in a MDS-2000 microwave digester (CEM Corporation, Matthews, NC) for 4 minutes at 43% power (518 kPa) followed by 8 minutes at 82% power (1380 kPa). After the digestion was complete, the samples were allowed to cool and then transferred into test tubes and brought up to 10 mL volume with



distilled water. Elemental concentrations were determined with a Perkin Elmer Optima 3000 DV Mass-spec ICP (Perkin-Elmer Corporation, Norwalk, CT).

### Statistical Analysis

Statistics were computed with standard analysis of variance (ANOVA) and regression procedures (SAS Version 6.12, SAS Institute Inc., Cary, NC). Least-significant differences were computed following a significant ( $P < 0.05$ ) F-test. In addition, contrasts between selected treatments were investigated when significant differences ( $P < 0.05$ ) were detected with ANOVA.

Statistical analysis of individual soil depths (N data) required log-10 transformation due to the non-homogeneity of the variances. Homogeneity of variances was tested using Bartlett's test. Transformation of the soil N data produced homogenous variances between treatments and allowed the ANOVA to be computed.

To estimate the net increase in available N across all biosolids rates, regression analysis of the soil profile N data was used. The soil profile N data was not log-10 transformed, since linear regression does not partition the total variation into different components but rather treats each observation equally. Linear regression equations were not performed on the postharvest soil N data due to the lack of significance between treatment means at this sampling date (both locations). In order to accurately predict the availability of N from biosolids and to provide consistency between sample dates, the regression line was fit through the average of the unfertilized control.

Regression analysis of grain yield response to increasing biosolids application rate was described by a 3-parameter sigmoid equation. This type of regression line was used because it increased the precision of regression from a quadratic equation and produced an estimate of maximum yield (plateau). The 3-parameter sigmoid equation is represented by Eq. [1]:

$$y = \frac{a}{1 + \exp\left\{-\frac{x - x_0}{b}\right\}} \quad \text{Eq. [1]}$$

where

$Y$  = grain yield (kg/ha)

$x$  = biosolids N applied (kg/ha)

$a$ ,  $x_0$ , and  $b$  = model parameters.

The parameter  $a$  is equal to the grain yield plateau level (maximum grain yield). To estimate the range of biosolids N ( $x$ ) that would produce maximum grain yields, 90% and 95% of the maximum grain yield (parameter  $a$ ) was substituted into Eq. [1] and solved for  $x$ .

Regression analysis of grain N concentration response to increasing biosolids were described by a quadratic equation. Using a cubic equation did not increase the model's precision as indicated by stepwise regression. The quadratic equation is represented by Eq. [2]:

$$Y = Y_0 + ax + bx^2 \quad \text{Eq. [2]}$$

where

$Y$  = grain yield (kg/ha)

$x$  = biosolids N applied (kg/ha)

$Y_0$ ,  $a$ , and  $b$  = model parameters

**Abbreviations:** AA, Anhydrous ammonia; BS, Biosolids; GHI, Grain Harvest Index; M-96, McClennan farm location; P-95, Pinkerton farm location.

## RESULTS AND DISCUSSION

### Grain Yield and Quality Indicators

In order for biosolids applications to be advantageous to wheat farmers, grain yields must be equivalent or greater than yields produced with traditional fertilizer. In addition to producing similar grain yields, grain quality must be maintained. The majority of the wheat grown in the Columbia Basin is managed to produce low protein (i.e., low

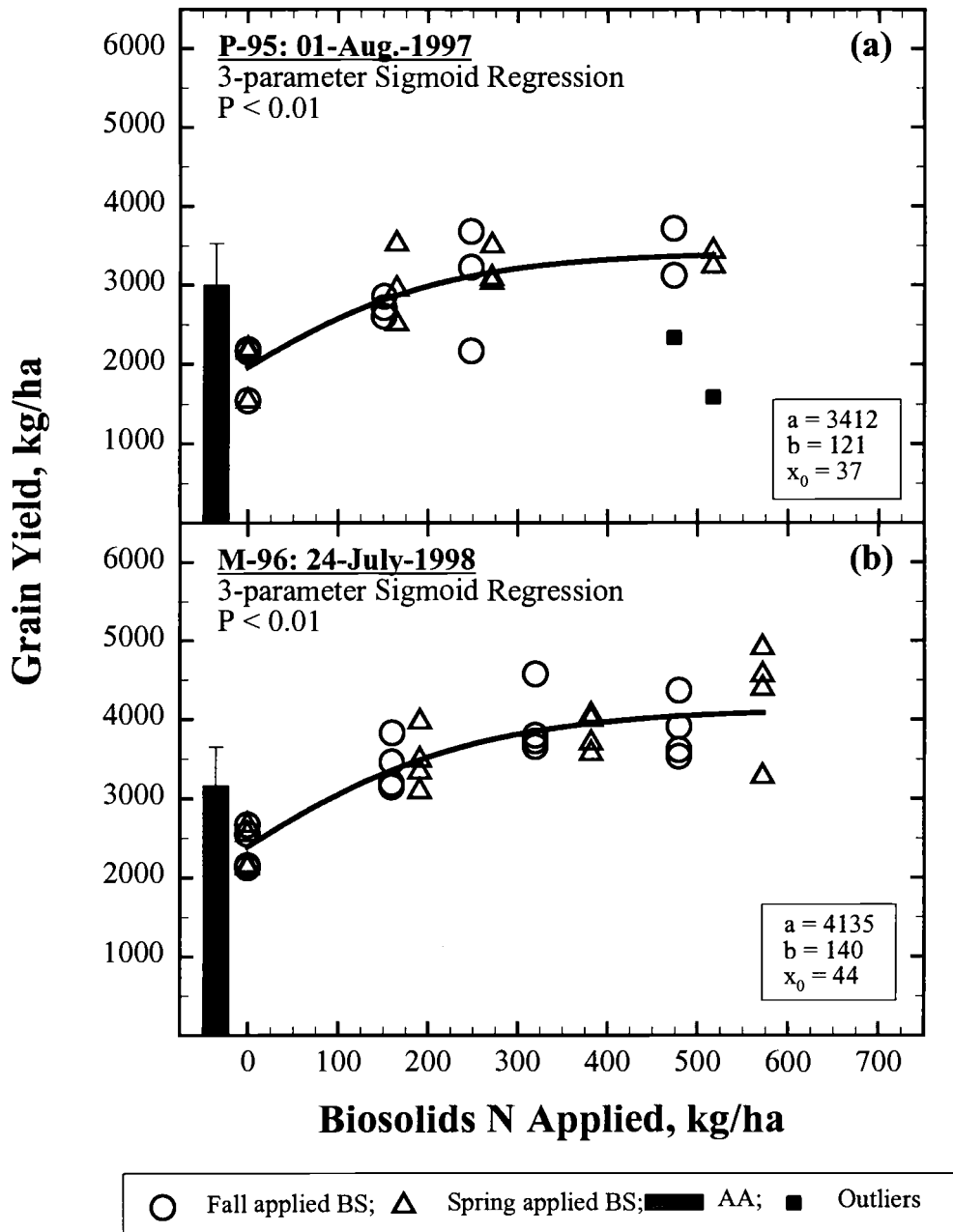
grain N concentrations). High grain N concentrations are achieved by supplying the crop with amounts of N in excess of that required for maximum yields. In order to produce low grain N concentrations, N cannot be applied in excessive amounts.

A number of agronomic measures were used to assess the effects of biosolids application rate on crop response including: grain yield, grain N, grain test weight, and biomass N uptake.

Biosolids maintained or increased grain yields with no decrease in grain quality. Grain yield response to increasing biosolids application rates was described by a 3-parameter sigmoid equation (Figure 2.1). At both locations, the regression line appeared to flatten out (reach the plateau level) at the medium rate of biosolids. Application rates of 230 to 370 kg biosolids N/ha corresponded to 90 to 95% of the maximum yield (plateau level, parameter  $a$ ). There were no observable differences in grain yields from the fall and spring applied biosolids.

At P-95, there were two plots in which the wheat was desiccated early in the grain-fill period because of very shallow (< 30 cm) and rocky soil. These two plots were both in the high rate of biosolids (one spring and one fall applied) and located on adjacent plots at the edge of the experiment area. Due to these reasons, these two plots were determined to be "outliers". Regression analysis was performed with and without these plots. Inclusion of these plots decreased the precision of the regression analyses and actually produced non-significant results; therefore, graphs show regression line for data set minus the outliers.

No detectable differences in grain harvest index (GHI) values, thousand kernel weights (weight of 1000 kernels, indication of grain plumpness), or test weights were observed at P-95 (Table 2.3). At M-96, biosolids increased test weights (781 to 791 kg/m<sup>3</sup>), but had no effect on GHI values (Table 2.4). Biosolids and the AA control increased thousand kernel weights as compared to the unfertilized control at M-96. Increasing biosolids application rate resulted in increased straw yields at both field



**Figure 2.1.** Anhydrous ammonia (AA, 56 kg N/ha at P-95 and 67 kg N/ha at M-96) and biosolids (BS) effects on grain yield at P-95 (a), and M-96 (b). Vertical bar indicates standard deviation of AA replications. At P-95, outlier plots not included in regression analysis. Sherman County, OR. 1997-1998.

**Table 2.3.** Anhydrous ammonia (AA) and biosolids (BS) effects on grain quality and straw production. Pinkerton Farm (P-95), Sherman County, OR. 1997.

Treatment	Biosolids Rate	Total N Applied	Application Date	Grain		Straw Yield	Grain Harvest Index <sup>a</sup>
				Thousand Kernel Weight	Test Weight		
	Mg/ha	kg/ha		g	kg/m <sup>3</sup>	kg/ha	%
No fertilizer	-	-	-	36.7	787	2450	44.7
AA	-	56	03-Jul-96	36.2	792	4640	40.0
BS1-Fall	3.1	151	06-Nov-95	37.2	788	3530	43.3
BS2-Fall	5.2	248		36.6	796	3860	43.3
BS3-Fall	9.9	474		31.8	779	6130	33.3
BS1-Spring	3.1	165	23-Apr-96	37.1	788	4100	42.0
BS2-Spring	5.2	270		37.1	792	3870	46.0
BS3-Spring	9.9	517		31.0	774	4680	36.3
			Significance	NS	NS	**	NS
			CV (%)	7.8	2.1	19	14
			LSD (0.05)	4.86	29.2	1395	10.1
			<b>Contrasts</b>				
			No fertilizer vs. All	-	-	**	-
			No fertilizer vs. AA	-	-	**	-
			Fall vs. Spring	-	-	NS	-
			AA vs. BS2	-	-	NS	-
			Biosolids rate	-	-	**	-

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> Grain Harvest Index computed via Eq. [1]

**Table 2.4.** Anhydrous ammonia (AA) and biosolids (BS) effects on grain quality and straw production. McClennan Farm (M-96), Sherman County, OR. 1998.

Treatment	Biosolids Rate	Total N Applied	Application Date	Grain			Grain Harvest Index <sup>b</sup>
				Thousand Kernel Weight <sup>a</sup>	Test Weight	Straw Yield	
	Mg/ha	kg/ha		g	kg/m <sup>3</sup>	kg/ha	%
No fertilizer	-	-	-	46.0	777	4440	37.3
AA	-	67	09-Jun-97	48.3	787	6190	36.4
BS1-Fall	3.8	160	16-Oct-96	48.6	781	6390	37.3
BS2-Fall	7.6	320		48.4	790	7140	38.7
BS3-Fall	11.4	480		48.5	791	6960	38.1
BS1-Spring	3.8	191	25-Apr-97	47.9	781	7500	34.0
BS2-Spring	7.6	382		47.3	789	7590	36.0
BS3-Spring	11.4	572		46.6	789	9230	34.4
			Significance	**	**	**	NS
			CV (%)	1.7	0.49	17	7.3
			LSD (0.05)	1.17	5.65	1736	3.91
			<b>Contrasts</b>				
			No fertilizer vs. All	**	**	**	-
			No fertilizer vs. AA	**	**	*	-
			Fall vs. Spring	**	NS	*	-
			AA vs. BS2	NS	NS	NS	-
			Biosolids Rate	NS	**	NS	-

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> Grain Harvest Index computed via Eq. [1]

locations. Thousand kernel weights and grain test weight measure the same relative parameter: grain shrivel/fullness; however, thousand kernel weight is considered a more accurate prediction tool. Grain test weight measures grain shrivel/fullness in a density determination procedure and therefore can be affected by how well the grain kernels fit together. Thousand kernel weight is commonly used as a scientific tool whereas grain test weight is used as a price determining factor in the marketplace. All treatments produced high grade (U.S. Grade No. 1) test weights ( $> 772 \text{ kg/m}^3$ ) as defined by the USDA.

Grain N concentrations increased with biosolids rate (Figure 2.2). The majority of the increase was seen in the highest biosolids application rate. Grain N concentrations in Sherman County are typically low, and sometimes a premium is paid for grain that has concentrations of about 1.6% or less (Sandy Macnab, personal communication, 1999). This premium is not paid every year however, and depends on how much grain in the area can be classified as "premium".

Although yield and quality indicators were not significantly different at P-95, the amount of straw harvested from each plot was different. This suggests that the yield potential was not achieved due to the water limitations of dryland cropping. At P-95, annual precipitation was above average while growing season precipitation was equivalent to the 38-year average. At M-96, both growing season precipitation and annual precipitation were above average.

Based on the grain yield response, the agronomic rate of biosolids at both locations was at a similar N rate (230-370 kg N/ha) despite deeper soil and greater growing season precipitation at M-96 than at P-95. Application of biosolids at these rates did not result in a decrease in grain quality when compared to the AA control.

### Available Soil Nitrogen

In addition to providing equivalent (or superior) grain yields as compared to traditional fertilizer, biosolids applications must minimize the risk of  $\text{NO}_3\text{-N}$  leaching. In order to minimize  $\text{NO}_3\text{-N}$  leaching, reliable estimates of N availability from biosolids must be provided. If the availability is overestimated, the crop will be N deficient. If the





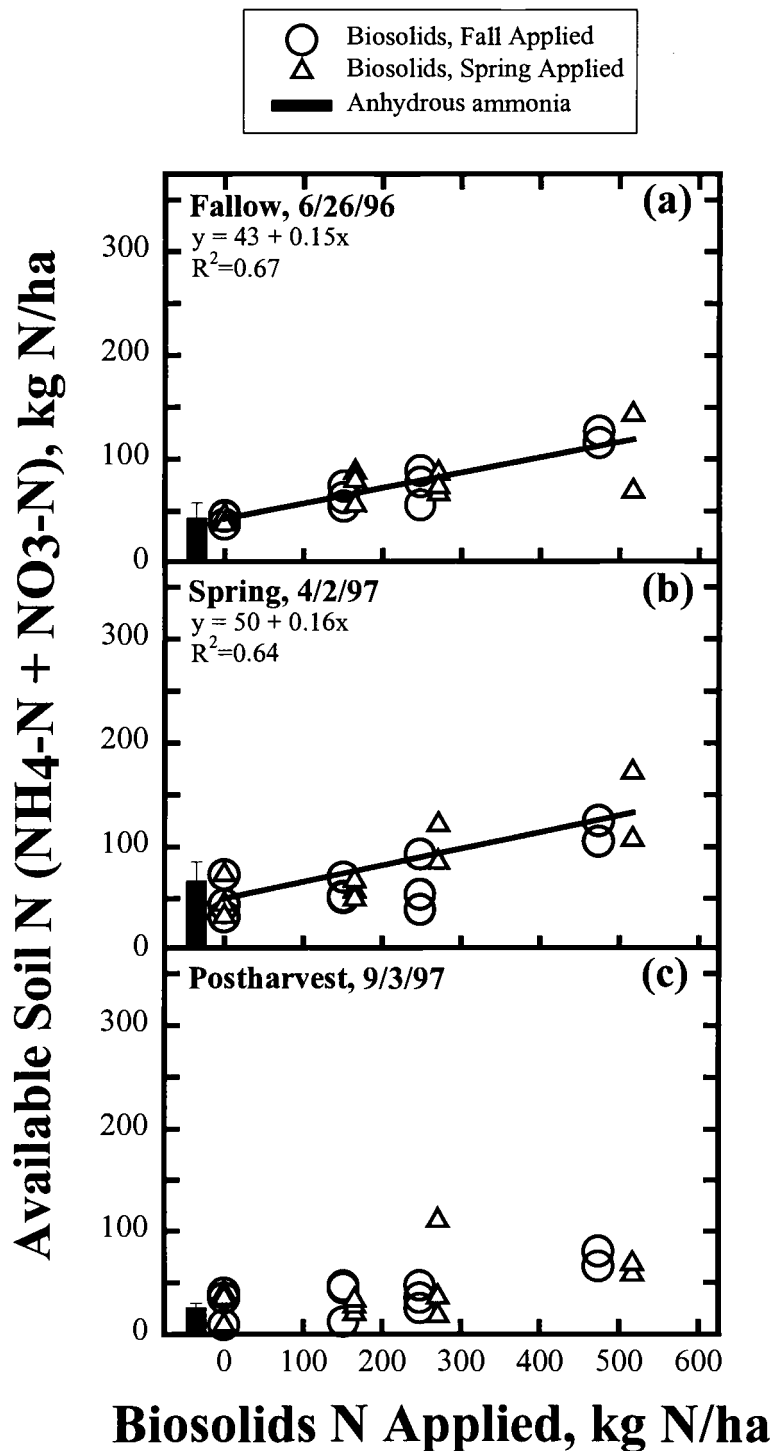
N availability is underestimated, the risk of high grain N concentrations and loss of  $\text{NO}_3\text{-N}$  is increased.

The same general trend was observed at both locations. Biosolids application increased available soil N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) during the summer fallow (Figure 2.3a and Figure 2.4a) and in the spring of the crop year (Figure 2.3b and Figure 2.4b) at both locations. Biosolids application did not increase available soil N following crop harvest at either location (Figure 2.3c and Figure 2.4c); however, there were some points within the highest biosolids application rates that were increased. These points were not significantly different from the other treatments due to the high variability within the sampling, but some field plots with increased  $\text{NO}_3\text{-N}$  concentrations were observed. The slope of the regression line for each sampling date was used to estimate the percentage of total biosolids N recovered as available forms. Biosolids application time (fall vs. spring) did not affect the availability of total available N at either location. The slope of the regression line at P-95 indicates that 15% of the biosolids N applied was recovered in available forms ( $\text{NO}_3\text{-N} + \text{NH}_4\text{-N}$ ) in the fallow sampling (2 to 7 months after application) and the spring sampling (12 to 17 months after application) (Figure 2.3a and 2.3b). For the postharvest sampling, there were no significant differences between treatments in the amount of available soil N recovered (Figure 2.3c).

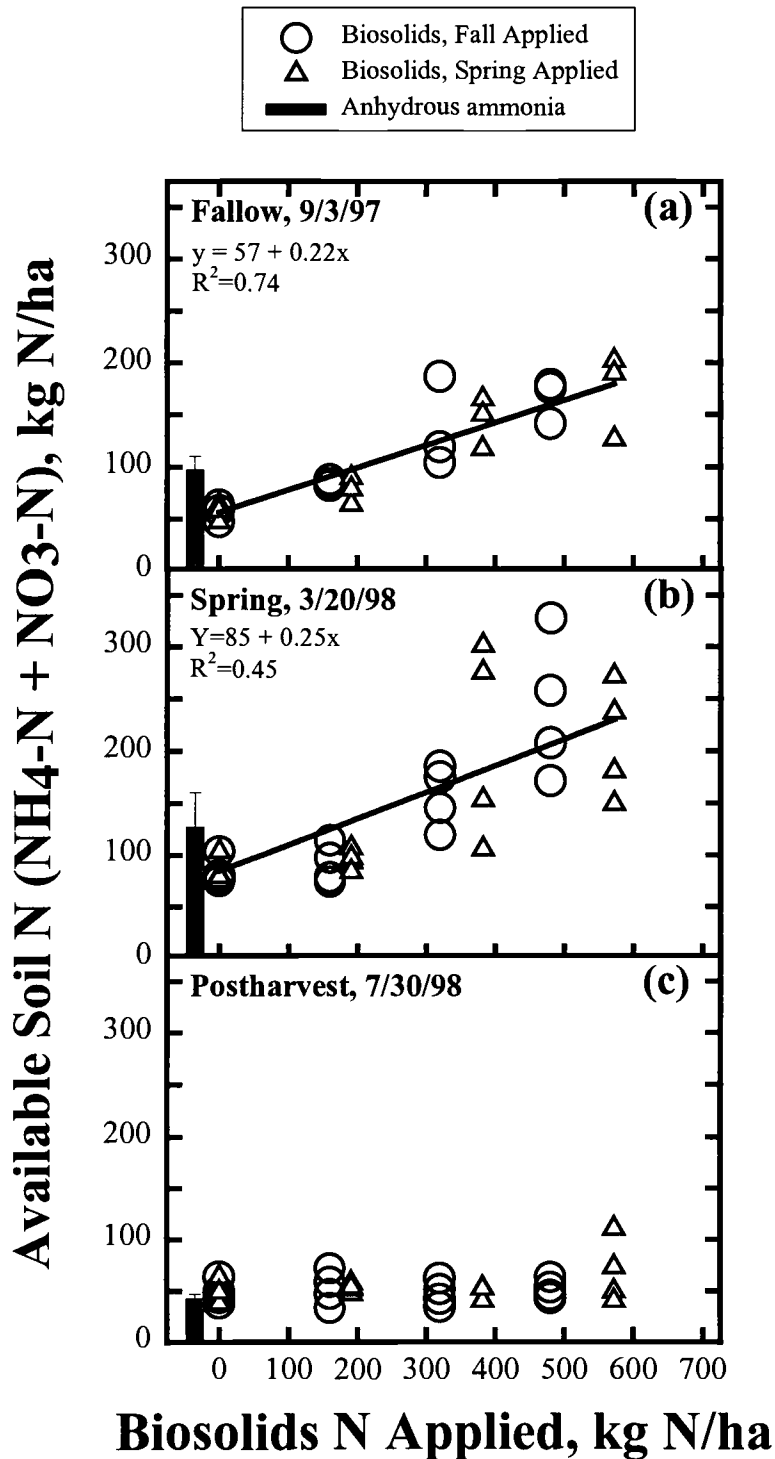
For M-96, the slope of the regression line indicates that 22% and 25% of the biosolids N applied was recovered in available forms ( $\text{NO}_3\text{-N} + \text{NH}_4\text{-N}$ ) in the fallow sampling (4 to 10 months after application) and the spring sampling (11 to 17 months after application), respectively (Figure 2.4a and 2.4b). For the postharvest sampling, there were no significant differences between treatments (log transformed data) in the amount of available soil N recovered (Figure 2.4c).

Monitoring of available soil N provided evidence of mineralization (ammonification and nitrification) as soil moisture and temperature allowed (Figure 2.5 and Figure 2.6). Ammonification is a process mediated by microbial activity that converts organic-N to  $\text{NH}_4\text{-N}$  (Sylvia et al., 1998). The  $\text{NH}_4\text{-N}$  then undergoes nitrification, essentially a two step, two organism process in which the  $\text{NH}_4\text{-N}$  is oxidized to  $\text{NO}_3\text{-N}$ .

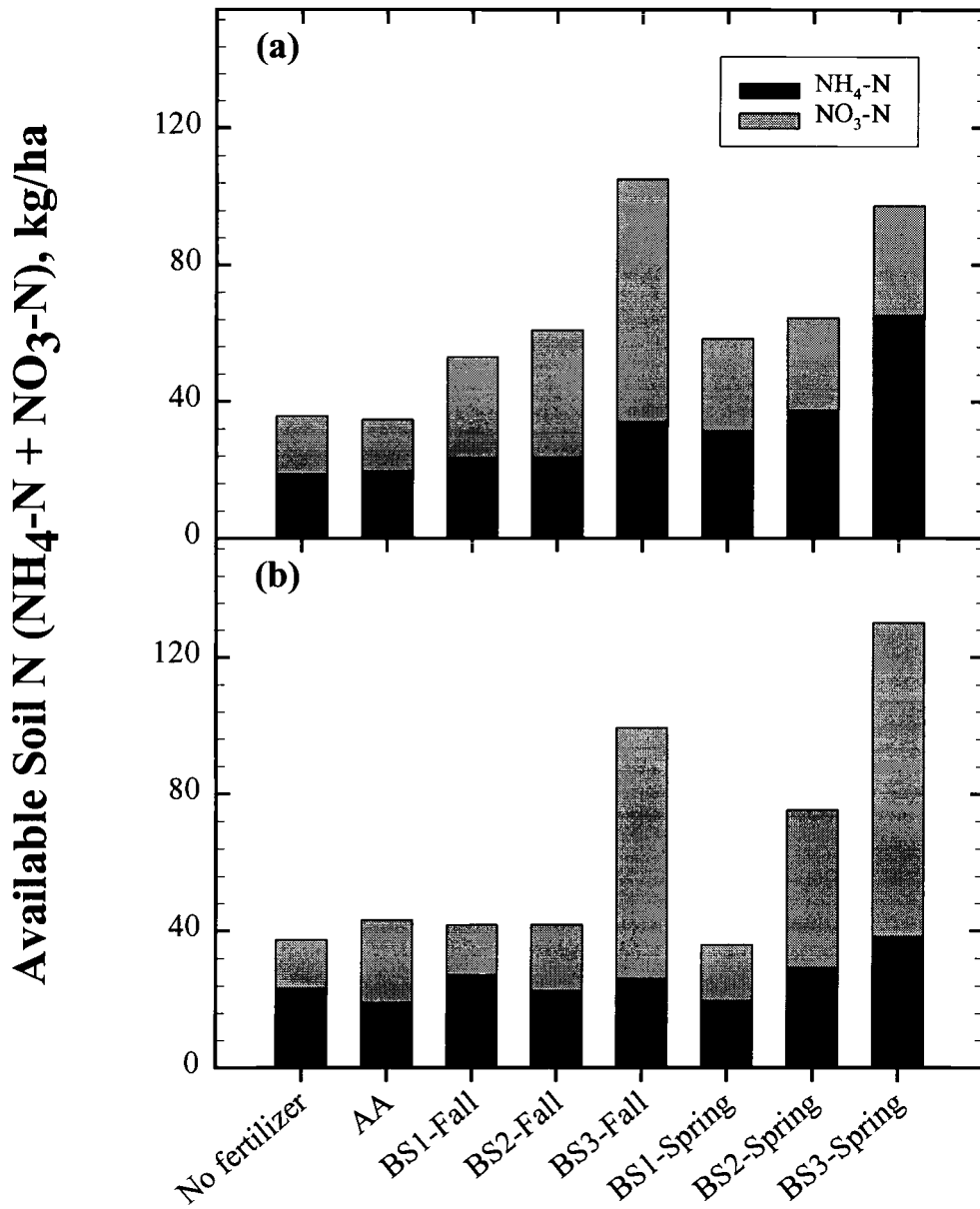
The timing of biosolids application (fall vs. spring) affected the form of N present in the fallow sampling (Figure 2.5a and Figure 2.6a). The fallow surface sampling



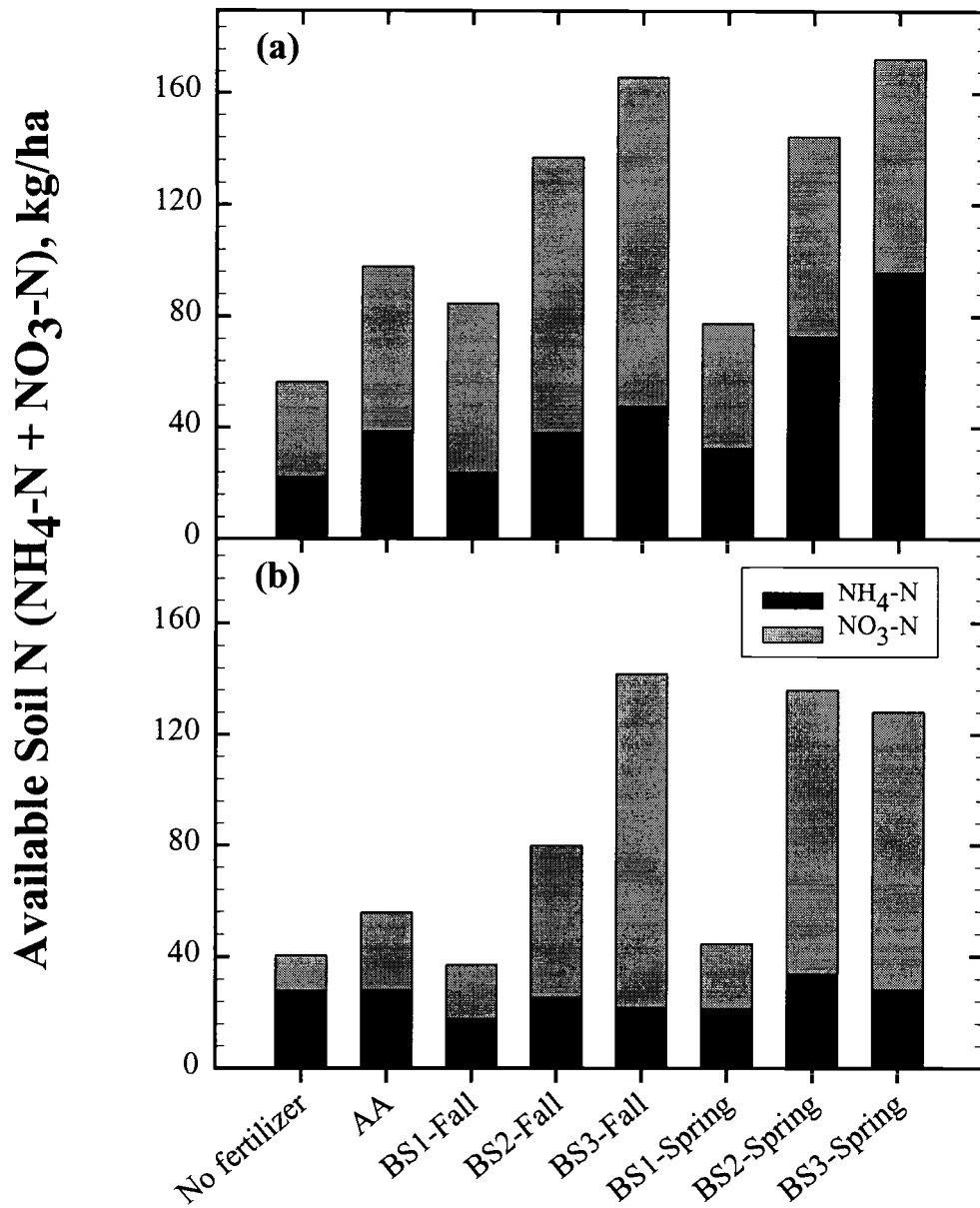
**Figure 2.3.** Anhydrous ammonia (56 kg N/ha) and biosolids (BS) effects on available soil N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) during: fallow (a); spring (b); and post harvest (c) of soft white winter wheat crop. 0-75 cm soil profile depth. Pinkerton Farm (P-95), Sherman County, OR. 1996-1997. Statistical analysis on postharvest soil N not significant using log 10 transformed data. Vertical error bar indicates standard deviation of AA replications.



**Figure 2.4.** Anhydrous ammonia (67 kg N/ha) and biosolids (BS) effects on available soil N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) during: fallow (a); spring (b); and post harvest (c) of soft white winter wheat crop. 0-90 cm soil profile depth. McClennan Farm (M-96), Sherman County, OR. 1997-1998. Postharvest soil N not significant using log 10 transformed data. Vertical error bar indicates standard deviation of AA replications.



**Figure 2.5.** Distribution of available soil N forms during: fallow (a); and spring (b) of the crop year (0-60 cm soil depth). Values are average of three replications. Pinkerton Farm (P-95), Sherman County, OR. 1996-1997.

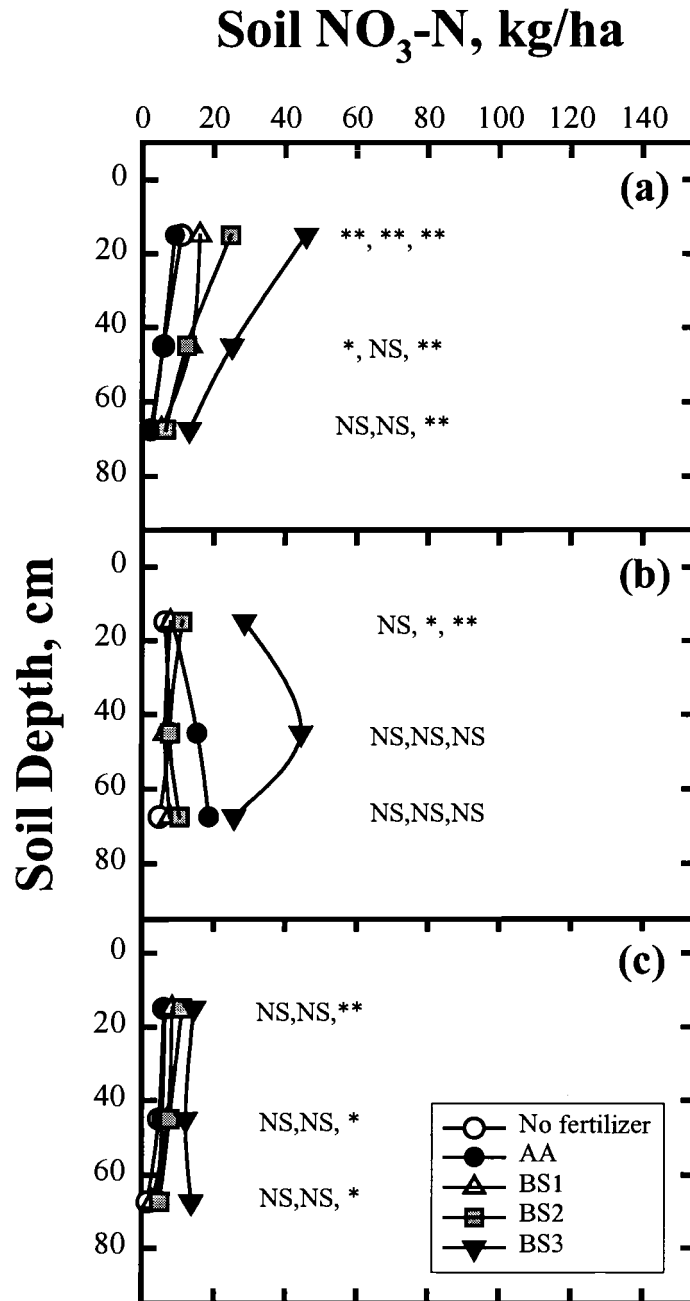


**Figure 2.6.** Distribution of available soil N forms during: fallow (a); and spring (b) of the crop year (0-60 cm soil depth). Values are average of four replications. McClennan Farm (M-96), Sherman County, OR. 1997-1998.

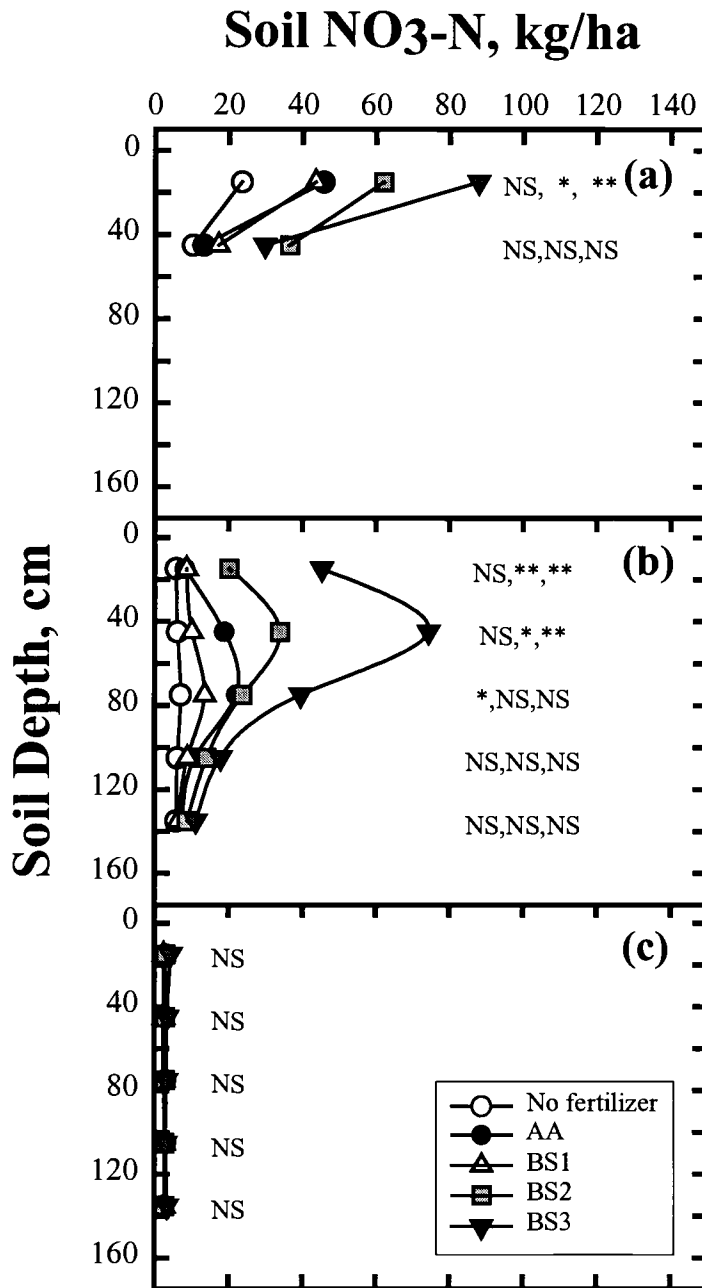
revealed that the total amount of N recovered was not significantly different between fall and spring applied biosolids. As would be expected, the fall applied biosolids had the majority of the N in the nitrate-N form and the spring applied biosolids had the majority in the ammonium-N form. Because of the differences in the form of N recovered from the two application dates, it is apparent that mineralization of the organic-N pool in the fall applied biosolids occurred over the first fallow winter following application. Although there were significant differences between the sampling dates in the forms of N recovered, the majority of the N was in the NO<sub>3</sub>-N form by the spring of the crop year (Figure 2.5b and Figure 2.6b).

Nitrate-N (NO<sub>3</sub>-N) was transported into the soil profile during the winter of the crop year. The transport of NO<sub>3</sub>-N at both locations followed the same pattern. At the fallow sampling, the majority of the NO<sub>3</sub>-N recovered was in the surface (0-30 cm depth). During the spring sampling, the majority of the NO<sub>3</sub>-N recovered was in the 30-60 cm depth. Since the fall applied biosolids had the greatest amount of NO<sub>3</sub>-N recovered in the fallow sampling and the majority (70%) of annual precipitation in the Columbia Basin occurs from September 1 to April 1 (Rasmussen et al., 1989), the fall biosolids application had the greatest potential of NO<sub>3</sub>-N leaching. Because of these reasons, we present more detailed data for the distribution of NO<sub>3</sub>-N throughout the soil profile for the fall applied biosolids only (Figure 2.7 and Figure 2.8). The movement of NO<sub>3</sub>-N to deeper depths is to be expected, and in fact desired, for normal crop growth. Plant available water is typically greater in the subsurface, especially in this region. N in the subsurface depths is utilized as the crop draws on the stored water.

There were no detectable differences observed with NO<sub>3</sub>-N recovered following grain harvest at either location. The lack of soil NO<sub>3</sub>-N recovered in the postharvest sampling does not necessarily reflect complete utilization by the crop. At P-95, it is possible that some N had been transported to depths that could not be sampled with the soil probe, due to the abundance of rocks. At M-96, it is possible that cheatgrass (*Bromus tectorum*) utilized some of the available soil N. Cheatgrass was taller and more dense as biosolids rate increased. The increase in nutrients provided by biosolids near the soil surface provided conditions favorable to cheat grass. Veseth (1987a) reported that the



**Figure 2.7.** Anhydrous ammonia (AA; 56 kg N/ha) and biosolids (BS) effects on NO<sub>3</sub>-N distribution in the soil profile during: fallow (a); spring (b); and postharvest (c) of soft white winter wheat crop. Symbols are the average of the fall applied biosolids only. Pinkerton Farm (P-95), Sherman County, OR. 1996-1997. Notations (left to right) indicate significant contrasts (\* at 0.05 and \*\* at 0.01 probability levels) for AA vs. BS1, AA vs. BS2, and AA vs. BS3, respectively.



**Figure 2.8.** Anhydrous ammonia (AA; 67 kg N/ha) and biosolids (BS) effects on NO<sub>3</sub>-N distribution in the soil profile during: fallow (a); spring (b); and postharvest (c) of soft white winter wheat crop. Symbols are the average of the fall applied biosolids only. McClennan Farm (M-96), Sherman County, OR. 1997-1998. Notations (left to right) indicate significant contrasts (\* at 0.05 and \*\* at 0.01 probability levels) for AA vs. BS1, AA vs. BS2, and AA vs. BS3, respectively.



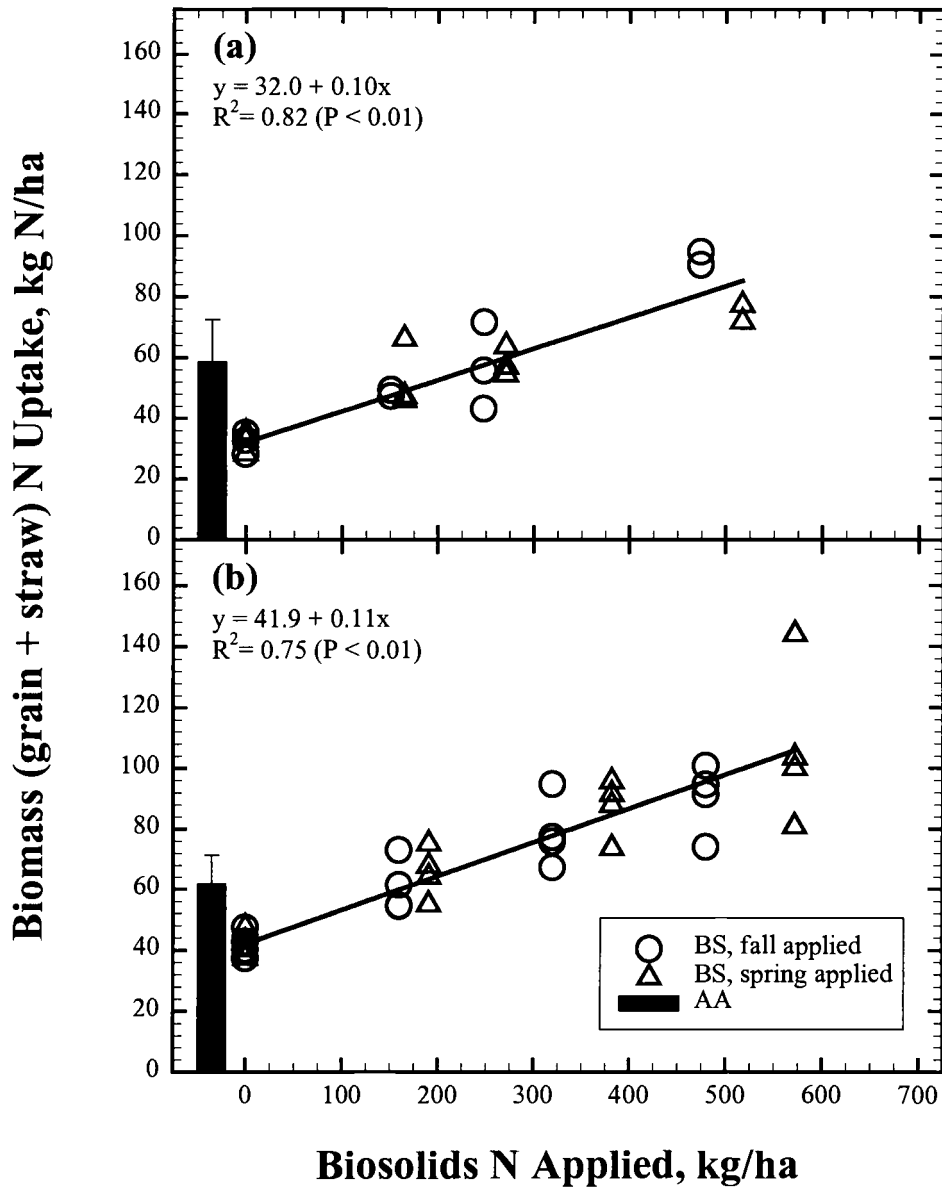
deep banding of N fertilizer (5 cm below the seed) consistently decreased weed dry weight versus broadcasting, although differences were not always statistically significant. Rasmussen (1995) found that broadcasting N fertilizer increased cheatgrass density and growth as compared to banding, which increased growth and N uptake of winter wheat.

It is unlikely that  $\text{NO}_3\text{-N}$  was transported below the sampling depth of 150 cm, even with the above average rainfall experienced in 1997-1998. During the period between the spring soil sampling and final harvest (roughly April through May), estimated crop water use in the Columbia Basin ranges from 8 to 16 cm per month (Cuenca et al., 1992). Records from the Moro experiment station reveal that 6 (P-95) to 13 cm (M-96) of rain fell during the same time period. Due to the difference in crop use and precipitation, it is unlikely that any N was leached through the soil profile.

### Efficiency of Crop Nitrogen Uptake

Biomass (grain + straw) N uptake increased with biosolids rate (Figure 2.9). Biomass N uptake was described by a linear regression model. The slope of the regression line indicates that approximately 10 to 11% of the total biosolids N was taken up by the above-ground biomass. Grain N uptake is important because it constitutes permanent removal of N from the site. Straw N uptake is important because it indicates a temporary removal of N that will eventually be returned to the soil.

Biomass N uptake values ranged from 30-60 kg N/ha, which is lower than values reported (85-98 kg N/ha) for a long term manure amended wheat-fallow rotation study in Pendleton, OR (Rasmussen and Parton, 1994). The main difference here is the higher yields observed, since grain N and straw N concentrations (Rasmussen and Parton, 1994) were similar to those observed in the present study. With applications of 45 and 90 kg N/ha as inorganic N, uptake ranges were 55-74 and 54-100 kg N/ha, respectively. The uptake value for the anhydrous ammonia control in the present study coincide with these values (average of 60 kg N/ha). Thus, the higher uptake values (for the manure additions) in the Rasmussen and Parton study (1994) are attributed to the indirect effect of higher yields.



**Figure 2.9.** Anhydrous ammonia (AA; 56 kg N/ha at P-95, 67 kg N/ha at M-96) and biosolids (BS) effects on biomass (grain + straw) N uptake at: Pinkerton Farm, P-95 (a); and McClennan Farm, M-96 (b). Sherman County, OR. 1997-1998. Vertical error bar indicates standard deviation of AA replications.

Crop N uptake efficiency ranged from 45-90% (for all biosolids treatments) and averaged 90% (for the anhydrous ammonia control) of the available soil N recovered in the soil profile in the spring (Figure 2.3b and Figure 2.4b). This is a similar, and in some cases superior, N uptake efficiency to that (45-70%) reported by others for soft white winter wheat (Fiez et al., 1995; Kjelgren, 1984). A possible reason for the higher uptake efficiency found in the present study compared to values other investigators have reported could be due to mineralization. In the present study, crop uptake was compared to available soil N recovered in the spring of the crop year; thus, any mineralization that occurred after the spring sampling was not accounted for. An underestimated available soil N supply would increase the uptake efficiency value. The high N uptake efficiencies observed at both locations provides additional evidence against NO<sub>3</sub>-N leaching in the spring of the crop year.

### Residual Soil Fertility Effects

In addition to supplying N, biosolids supply other plant nutrients such as P, S, and Zn. Surface (0-15 cm) soil samples were collected to assess the effects of biosolids on the availability of these nutrients. Grain and straw uptake values were also calculated in order to determine the effect of biosolids application on nutrient removal.

#### *Phosphorus*

Since P is relatively immobile, surface soil testing is valid in order to determine the effects of biosolids on soil P. Biosolids application increased the Bray-1 P values in the surface soil (0-15 cm depth) sampling following harvest of the winter wheat crop at P-95 (Table 2.5) and in the spring of the crop year at M-96 (Table 2.6). Bray-1 P values observed with the unfertilized controls at both locations provided evidence of adequate P, with medium to high levels (Marx et al., 1998). Bray-1 P levels were above reported deficiency levels for crops. Fall applied biosolids increased soil test P values more than

**Table 2.5.** Anhydrous ammonia (AA) and biosolids (BS) effects on extractable nutrients in soil (0-15 cm depth). Pinkerton Farm (P-95), Sherman County, OR. Sampled Sept. 1997 (postharvest).

Treatment	Biosolids Rate	Bray-1 P	SO <sub>4</sub> -S	Hot Water B	DTPA				Exchangeable			
					Zn	Mn	Cu	Fe	K	Ca	Mg	Na
		-----mg/kg-----							-----cmol/kg-----			
No fertilizer	0	38.7	3.5	0.27	0.45	25	3.7	170	455	6.3	2.6	0.04
AA	0	38.8	3.5	0.27	0.49	31	1.8	51	508	5.9	2.6	0.07
BS1-Fall	3.1	39.4	4.1	0.24	0.54	26	2.0	55	433	6.6	2.6	0.04
BS2-Fall	5.2	45.6	3.5	0.26	0.70	27	2.0	101	449	6.3	2.5	0.04
BS3-Fall	9.9	55.4	4.3	0.34	0.89	27	2.5	69	438	6.6	2.6	0.04
BS1-Spring	3.1	39.8	4.0	0.34	0.57	30	1.9	62	489	6.2	2.6	0.05
BS2-Spring	5.2	40.9	3.9	0.26	0.53	28	1.9	161	455	6.4	2.6	0.04
BS3-Spring	9.9	45.5	4.0	0.35	0.78	35	2.3	63	406	6.5	2.8	0.04
<b>Deficiency Level <sup>a</sup></b>		< 20	< 10	< 0.5	< 0.6	< 1.2	< 0.2	< 2.5	< 150	< 5	< 0.5	-
Significance		**	NS	**	**	**	NS	NS	NS	*	NS	*
CV (%)		6.7	6.7	9.9	17	8.5	54	60	11	3.2	5.6	20
LSD (0.05)		5.08	1.20	0.05	0.18	4.29	2.12	95.9	90.3	0.35	0.25	0.02
<b>Contrasts</b>												
No fertilizer vs. All		*	-	NS	*	*	-	-	-	NS	-	NS
No fertilizer vs. AA		NS	-	NS	NS	*	-	-	-	*	-	**
Fall vs. Spring		**	-	**	NS	**	-	-	-	NS	-	NS
AA vs. BS2		*	-	NS	NS	NS	-	-	-	**	-	**
Biosolids Rate		**	-	*	*	*	-	-	-	NS	-	NS

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> Crop deficiency levels for Zn, Mn, Cu and Fe as reported by Lindsay and Norvell (1978); All others reported by Marx et al. (1998)

**Table 2.6.** Anhydrous ammonia (AA) and biosolids (BS) effects on extractable nutrients in soil (0-15 cm). McClennan Farm (M-96), Sherman County, OR. Sampled March 1998 (spring of crop year).

Treatment	Biosolids Rate	Bray-1 P	SO <sub>4</sub> -S	Hot Water B	DTPA				Exchangeable			
					Zn	Mn	Cu	Fe	K	Ca	Mg	Na
		-----mg/kg-----							-----cmol/kg-----			
No fertilizer	0	48.7	5.0	0.32	0.85	28	2.1	65	500	5.2	1.7	0.01
AA	0	51.6	3.8	0.34	0.88	32	2.0	72	465	5.5	1.7	0.03
BS1-Fall	3.8	60.7	3.5	0.28	1.19	30	2.4	67	511	5.4	1.7	0.02
BS2-Fall	7.6	81.1	4.6	0.30	1.74	32	2.7	77	481	5.5	1.6	0.01
BS3-Fall	11.4	95.3	5.6	0.28	1.79	31	3.4	73	504	5.2	1.6	0.02
BS1-Spring	3.8	53.9	4.6	0.26	1.11	30	2.3	65	554	5.4	1.6	0.01
BS2-Spring	7.6	59.3	5.3	0.29	1.43	38	2.4	72	491	5.3	1.5	0.01
BS3-Spring	11.4	68.5	4.6	0.35	1.74	41	2.6	77	520	5.2	1.6	0.01
<b>Deficiency Level <sup>a</sup></b>		< 20	< 10	< 0.5	< 0.6	< 1.2	< 0.2	< 2.5	< 150	< 5	< 0.5	-
Significance		**	NS	NS	**	**	**	*	NS	NS	NS	NS
CV (%)		8.34	28	21.39	14.38	7.47	11.70	7.47	10.18	5.98	7.96	108.54
LSD (0.05)		7.95	1.87	0.09	0.28	3.60	0.43	7.77	75.31	0.47	0.19	0.02
<b>Contrasts</b>												
No fertilizer vs. All		**	-	-	**	**	**	*	-	-	-	-
No fertilizer vs. AA		NS	-	-	NS	*	NS	NS	-	-	-	-
Fall vs. Spring		**	-	-	NS	**	**	NS	-	-	-	-
AA vs. BS2		**	-	-	**	NS	**	NS	-	-	-	-
Biosolids Rate		**	-	-	*	*	**	*	-	-	-	-

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> Crop deficiency levels for Zn, Mn, Cu and Fe as reported by Lindsay and Norvell (1978); All others reported by Marx et al. (1998)

the spring applied biosolids at both locations. This was attributed to the seasonally different process involved with biosolids production. For the fall applied biosolids, the wastewater treatment plant is required to remove additional P from the effluent. This is done by precipitating P with aluminum sulfate (alum) during the tertiary treatment. These alum residual solids are added to the primary and secondary solids and become part of the fall applied biosolids. The spring applied biosolids contain only the primary and secondary solids.

Application of biosolids at the agronomic rate increased soil test P values over levels observed with the AA control. This increase was more pronounced with the fall applied biosolids (i.e., produced in the summer) than with the spring applied biosolids (produced in the winter). This suggests that the additional P contained in the fall applied biosolids was more available, since the increase in total P application rates was only 45 to 60 kg/ha (Table 2.1). Recently, environmental concerns have forced many states to develop threshold limits for soil test P values (Sharpley et al., 1999). These limits are based on the potential for P loss in agricultural runoff. Of the states that have adopted these limits, the average regulatory threshold limits for Bray-1 or Mehlich-III soil test P values are 100 mg/kg. Using the average threshold limit and regression analysis of the relationship between P addition and soil test P values (data not shown), an estimation of the effect of repeated biosolids application on soil test P was found for the agronomic (medium) rate of biosolids. The increase ranged from 3 to 11 mg/kg soil test P per 100 kg biosolids P applied. Based on an average soil test P threshold limit of 100 mg/kg, 1 to 8 agronomic biosolids applications (5.2 to 7.6 Mg biosolids/ha) could be performed, depending on the site and biosolids characteristics.

Although higher levels of soil test P were present at high biosolids rates, no significant differences between plant tissue (grain and straw) P concentrations were detected (Table 3.5 and Table 3.6; Chapter 3). Other researchers reported that high amounts of P addition did not result in increased plant uptake (Barbarick et al., 1995; Sims, 1990; Taylor et al., 1978).

Grain P uptake values were not significantly different at P-95 (Table 2.7) but increased with increasing biosolids application rates at M-96 (Table 2.8). The increased yields observed at M-96 resulted in the increased uptake values. The P concentrations in

**Table 2.7.** Anhydrous ammonia (AA) and biosolids (BS) effects on grain and straw P, S, and Zn uptake. Pinkerton Farm (P-95), Sherman County, OR. 1997.

Treatment	Biosolids Rate Mg/ha	Grain Uptake			Straw Uptake		
		P	S	Zn	P	S	Zn
No fertilizer	0	6	1.9	0.03	0.6	0.7	0.01
AA	0	8	2.9	0.04	1.3	1.8	0.02
BS1-Fall	3.1	7	2.6	0.04	0.8	1.1	0.01
BS2-Fall	5.2	8	3.1	0.05	1.0	1.5	0.01
BS3-Fall	9.9	8	3.9	0.05	2.0	4.8	0.03
BS1-Spring	3.1	8	2.8	0.04	0.9	1.3	0.01
BS2-Spring	5.2	8	3.1	0.05	0.9	1.3	0.01
BS3-Spring	9.9	7	3.3	0.05	1.8	3.7	0.03
Significance		NS	NS	NS	**	**	NS
CV (%)		16	21	17	37	54	62
LSD (0.05)		2.0	1.09	0.013	0.75	1.93	0.017
<b>Contrasts</b>							
No fertilizer vs. All		-	-	-	*	*	-
No fertilizer vs. AA		-	-	-	*	NS	-
Fall vs. Spring		-	-	-	NS	NS	-
AA vs. BS2		-	-	-	NS	NS	-
Biosolids Rate		-	-	-	**	**	-

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

**Table 2.8.** Anhydrous ammonia (AA) and biosolids (BS) effects on grain and straw P, S, and Zn uptake. McClennan Farm (P-95), Sherman County, OR. 1998.

Treatment	Biosolids Rate Mg/ha	Grain Uptake			Straw Uptake		
		P	S	Zn	P	S	Zn
		-----kg/ha-----					
No fertilizer	0	8	2.3	0.04	2.2	1.3	0.01
AA	0	10	2.9	0.05	2.1	1.4	0.02
BS1-Fall	3.1	11	3.3	0.06	1.9	2.4	0.02
BS2-Fall	5.2	13	4.5	0.08	2.4	2.9	0.02
BS3-Fall	9.9	12	4.8	0.07	1.9	5.1	0.02
BS1-Spring	3.1	11	3.4	0.06	3.2	2.7	0.02
BS2-Spring	5.2	12	4.4	0.06	2.9	3.7	0.02
BS3-Spring	9.9	13	5.4	0.08	3.1	4.7	0.03
Significance		**	**	**	NS	**	**
CV (%)		12	13	17	32	42	24
LSD (0.05)		1.9	0.77	0.015	1.1	1.88	0.007
<b>Contrasts</b>							
No fertilizer vs. All		**	**	**	-	*	**
No fertilizer vs. AA		*	NS	NS	-	NS	*
Fall vs. Spring		NS	NS	NS	-	NS	*
AA vs. BS2		**	**	**	-	*	NS
Biosolids Rate		**	**	*	-	**	*

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

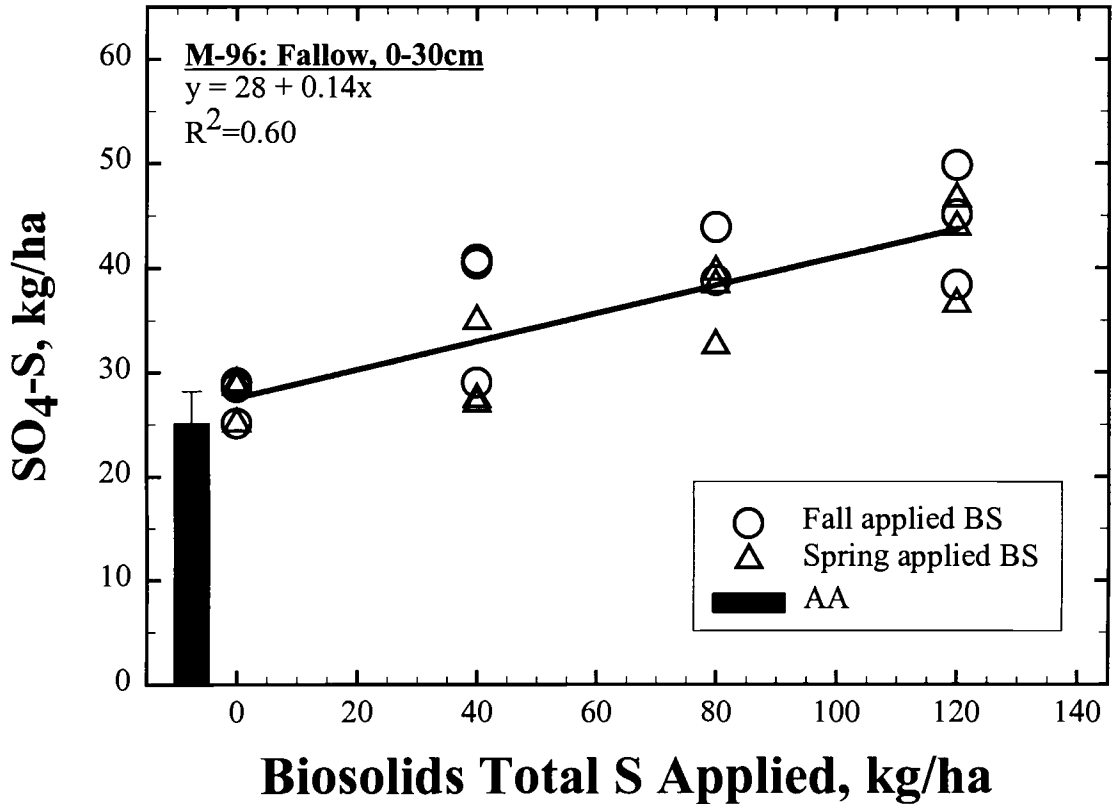


the grain and straw did not show any effects of the increased application rates (Table 3.5 and Table 3.6; Chapter 3). Even though P uptake values were increased at M-96, the amount of increase was small in comparison to the amount applied, representing an increase of 3 kg P/ha (over the anhydrous ammonia control) in comparison to 100-300 kg P/ha applied. Increases in straw uptake values were also small in comparison to application rates. Increases of less than 1 kg P/ha were observed with the straw at both locations.

### *Sulfur*

SO<sub>4</sub>-S is mobile (anion) and therefore would readily be transported to deeper depths in the soil profile, much like NO<sub>3</sub>-N. Soil samples collected during the fallow at M-96 revealed that approximately 14% of the total S applied was recovered as SO<sub>4</sub>-S (Figure 2.10). The ratio of available N to available S supplied by biosolids was found to be roughly 1.5:1. This is a similar availability ratio as reported by others (Sullivan et al., 1995; Cogger et al., 1998). The Sullivan et al. (1995) paper contained the S data and the Cogger et al. (1998) paper contained the N data for the same 3 locations in Washington State. The ratio of N mineralized to S mineralized has been shown to vary from less than 1 to greater than 1 (Frenney, 1986). Tabatabai and Chae (1991) suggested that mineralization of S in soils amended with sewage sludge would vary with soil type and C/N/S ratio of the sewage sludge.

Surface (0-15 cm) soil testing revealed that there was no effect of biosolids on the amount of SO<sub>4</sub>-S recovered in the spring of the crop year (Table 2.6) or following grain harvest (Table 2.5). Soil test values (0-15 cm) represent medium levels based on Oregon State University Extension publications (Marx et al., 1998). Soil test values ranged from 7 kg SO<sub>4</sub>-S/ha to 11 kg SO<sub>4</sub>-S/ha. The Oregon State University fertilizer Guide for non-irrigated winter wheat in the Columbia Plateau suggests that an application rate of 17 to 22 kg S/ha is adequate for two crops (Oregon State University, 1980). Apparently, much of the SO<sub>4</sub>-S provided by biosolids application was either below the sampling depth (0-15 cm), incorporated into soil organic matter, or taken up by the crop.



**Figure 2.10.** Anhydrous ammonia (AA, 67 kg N/ha) and biosolids (BS) effects on available soil S (SO<sub>4</sub>-S) during fallow sampling. 0-60 cm soil profile depth. McClennan Farm, Sherman County, OR. 1997.

Flag leaf N:S values ranged from 8:1 to 14:1 (data not shown) for biosolids amended treatments. Values greater than 17:1 are associated with S deficiency (Rasmussen, 1996). Due to the low N:S ratios observed, we concluded that S was not limiting grain yields at our field locations. S concentrations in plant tissue (grain and straw) increased with increasing S application (Table 3.5 and Table 3.6; Chapter 3).

Grain S uptake values did not increase with biosolids application at P-95 (Table 2.7) but did increase at M-96 (Table 2.8). As with the P uptake values, the increase in S uptake (2.5 kg S/ha) was very small in comparison to the total amount applied (30-120 kg S/ha). Straw S uptake values (for M-96) were higher than the anhydrous ammonia control, but the increase (3.7 kg S/ha) was again very small when compared to the total amount of S applied. This increase in straw S uptake values is important because it constitutes a temporary removal of S that will be returned to the site (not permanently removed).

### *Zinc*

Zn, like P, is relatively immobile and therefore surface soil testing is valid in order to determine the effects of biosolids on Zn availability. Biosolids application increased the amounts of available soil Zn recovered in the surface (0-15 cm) soil sampling in the spring of the crop year at M-96 (Table 2.6) and following harvest of the winter wheat crop at P-95 (Table 2.5). Soil test Zn values without biosolids were near reported deficiency levels (Marx et al., 1998; Lindsay and Norvell, 1978).

Despite the increases in soil availability, there was no increase in the concentrations of Zn these nutrients in the grain or straw (Table 3.5 and Table 3.6; Chapter 3). Most plants have a high tolerance to excessive amounts of Zn (Kabata-Pendias and Pendias, 1992). Sloan et al. (1997) reported that Zn uptake by romaine lettuce was significantly correlated with total soil Zn concentrations in the surface (0-20 cm). Application rates in their study ranged from 60-343 kg Zn/ha. Application rates in our study were significantly lower (Table 2.1). Barbarick et al. (1998) observed increased

concentrations of DTPA-extractable Zn in the subsoil as a result of long term (10 years) biosolids applications.

At P-95, no differences between Zn uptake values were observed between treatments for either the grain or straw tissue (Table 2.7). At M-96, biosolids application increased grain and straw Zn uptake (Table 2.8). Increases were 0.03 kg Zn/ha for the grain and 0.01 kg Zn/ha for the straw. Again, these increases were very small in comparison to the total amount applied (1.5 to 6.4 kg Zn/ha).

### *Soil pH and Electrical Conductivity*

The effect of biosolids on surface (0-15 cm) soil pH, an indicator of active acidity, was varied dependent on sampling time. No effect on pH was observed following grain harvest (Table 2.9). Sampling in the spring of the crop year (M-96) provided significant differences between treatments and biosolids application time (Table 2.10). The net decrease in pH observed with the agronomic rate of biosolids was 0.2 to 0.3 pH units compared to the AA control. The spring applied biosolids produced lower pH values than the fall applied biosolids at M-96. This difference was probably attributed to the recent mineralization of organic-N, during which hydrogen (H) ions are released. Soil pH values at our locations (5.4 to 5.9 at P-95; 6.0 to 6.4 at M-96) were above those found to detrimental to soft-white wheat in the Pacific Northwest (pH < 5.3; Veseth, 1987b).

A similar dependence on sampling time was observed with SMP (Shoemaker-McLean-Pratt) buffer pH response to biosolids application. SMP buffer pH values are an indicator of both active and reserve acidity. SMP buffer pH values are used to make lime application recommendations. No effect on SMP pH was observed following grain harvest at P-95 (Table 2.9). Sampling in the spring of the crop year produced significantly lower SMP pH values with increasing biosolids application rates at M-96 (Table 2.10). The net decrease in SMP buffer pH value for the agronomic rate of biosolids was 0.03 to 0.10 pH units compared to the AA control. Using the SMP buffer pH value for the high biosolids rate suggests that 360 to 730 kg 100-score lime/ha be applied to raise the surface (0-15 cm) soil pH to 6.0 to 6.2 (Marx et al., 1998).

**Table 2.9.** Anhydrous ammonia (AA) and biosolids (BS) effects on soil pH and electrical conductivity (0-15 cm depth). Pinkerton Farm (P-95), Sherman County, OR. Sampled Sept. 1997 (postharvest).

<b>Treatment</b>	<b>Biosolids Rate</b>	<b>Total N Applied</b>	<b>Application Date</b>	<b>pH</b>	<b>SMP Buffer pH<sup>a</sup></b>	<b>Electrical Conductivity</b>
	Mg/ha	kg/ha				dS/m
No fertilizer	0	0	-	6.5	6.90	0.22
AA	0	56	03-Jul-96	6.4	7.00	0.23
BS1-Fall	3.1	151	06-Nov-95	6.5	7.00	0.23
BS2-Fall	5.2	248		6.3	6.90	0.23
BS3-Fall	9.9	474		6.3	6.93	0.32
BS1-Spring	3.1	165	23-Apr-96	6.4	6.93	0.30
BS2-Spring	5.2	270		6.4	6.97	0.25
BS3-Spring	9.9	517		6.0	6.83	0.50
			Significance	NS	NS	*
			CV (%)	3.2	1.1	31
			LSD (0.05)	0.36	0.13	0.171
			<b>Contrasts</b>			
			No fertilizer vs. All	-	-	NS
			No fertilizer vs. AA	-	-	NS
			Fall vs. Spring	-	-	NS
			AA vs. BS2	-	-	NS
			Biosolids Rate	-	-	*

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> SMP (Shoemaker, MacLean, and Pratt) Buffer pH = 7.0 without soil. Measures active and reserve acidity for lime requirement recommendation.

**Table 2.10.** Anhydrous ammonia (AA) and biosolids (BS) effects on soil pH and electrical conductivity (0-15 cm depth) McClennan Farm (M-96), Sherman County, OR. Sampled March 1998 (spring of crop year).

Treatment	Biosolids	Total N	Application Date	SMP		Electrical
	Rate	Applied		pH	Buffer pH	Conductivity
	Mg/ha	kg/ha				dS/m
No fertilizer	0	0	-	5.9	6.75	0.18
AA	0	67	09-Jun-97	5.9	6.68	0.17
BS1-Fall	3.8	160	16-Oct-96	5.8	6.65	0.19
BS2-Fall	7.6	320		5.7	6.63	0.26
BS3-Fall	11.4	480		5.7	6.68	0.30
BS1-Spring	3.8	191	25-Apr-97	5.8	6.70	0.21
BS2-Spring	7.6	382		5.6	6.58	0.31
BS3-Spring	11.4	572		5.4	6.58	0.49
			Significance	**	**	**
			CV (%)	1.5	0.74	36
			LSD (0.05)	0.13	0.07	0.138
			<b>Contrasts</b>			
			No fertilizer vs. All	**	**	NS
			No fertilizer vs. AA	NS	*	NS
			Fall vs. Spring	**	NS	NS
			AA vs. BS2	**	*	NS
			Biosolids Rate	**	*	*

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> SMP (Shoemaker, MacLean, and Pratt) Buffer pH = 7.0 without soil. Measures active and reserve acidity for lime requirement recommendation.

The same trend with electrical conductivity (EC) and increasing biosolids rate was observed at both locations. Electrical conductivity (EC) is an indirect measurement of soluble salts, which can be detrimental to plants. At both locations, the only significant EC increase was observed with the highest rate of biosolids application (Table 2.9 and Table 2.10). The agronomic rate of biosolids had no effect on EC values. The net increase in EC values for the highest biosolids rate was 0.27 to 0.32 dS/m as compared to the AA control. Although EC values were increased with the highest biosolids rate, all values were below levels considered high (1.0 dS/m; Marx et al., 1998).

Overall, the pH changes observed followed biological activity, as evidenced by the different responses between sampling times (and locations). Although the changes in pH values were fairly consistent between locations, the increased variability at P-95 resulted in no significant differences between treatments. The increased salt concentrations observed with the high rate of biosolids probably did affect the measured pH values. In general, the pH of a soil solution decreases with increasing salt concentration (Van Lierop, 1990). In order to accurately determine the effect of biosolids on soil pH, measuring the pH several years after biosolids application when soluble salt concentrations are equivalent would be required.

### *Other Nutrients*

Biosolids application increased the availability of DTPA-extractable Mn and Cu at both locations (Table 2.5 and Table 2.6). Small increases in hot water B (P-95) and DTPA-extractable Fe (M-96) were observed. Although the availability of some nutrients was increased with biosolids application, all nutrients were above reported deficiency levels for crop production. Despite the small increases in availability of some nutrients in the soil, elemental analysis of the grain showed no increase in concentrations (Appendix B and Appendix C). Small increases in nutrient concentrations were observed with the straw (K, Ca, Mg, and Mn). Although these increases were statistically significant, they were associated with the high rate of biosolids only.

## SUMMARY AND CONCLUSIONS

For the two locations where on-farm research has been conducted in Sherman County, the agronomic rate (90 to 95% of the maximum yield) for soft-white wheat production was found to be approximately 230 to 370 kg biosolids N/ha (5 to 8 Mg/ha). The grain yields observed with the agronomic rate of biosolids were equivalent (P-95) or superior (M-96) to that produced with the AA control. The lower rate is recommended for manure spreaders that can accurately deliver 5 Mg/ha. The higher rate is recommended for manure spreaders that are not as accurate. Application rates above 370 kg biosolids N/ha (8 Mg/ha) did not provide any agronomic benefits, and may increase production risks. Greater risks of lodging, grain shrivel, cheatgrass proliferation, and high grain N concentrations are associated with excessive biosolids application rates.

Biosolids maintained or increased grain yields with no decrease in grain quality. Grain yield response to increasing biosolids application rates was described by a 3-parameter sigmoid regression function. At both locations, the regression line appeared to flatten out (reach the plateau level) at the medium rate of biosolids. For grain production, fall and spring-applied biosolids performed similarly. Biosolids applied at 230 to 370 kg N/ha produced 90 to 95% of maximum grain yields. Increased biosolids rates resulted in higher grain N concentrations and straw N concentrations, indicating that more than adequate N was supplied to the crop for maximum grain production. These rates of biosolids also showed no detectable evidence of excessive  $\text{NO}_3\text{-N}$  leaching, as compared to the anhydrous ammonia control. Application of higher rates of biosolids (9.9 Mg/ha, 470-520 kg N/ha) did not increase grain yields, but increased grain N and plant tissue trace element concentrations.

The same general trend was observed at both locations in regards to available soil N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ). The medium rate of biosolids provided 35 to 93 kg available N per hectare for the first crop after application. Biosolids application increased available soil N during the summer fallow and in the spring of the crop year at both locations. Biosolids application did not increase available soil N following crop harvest at either location. The slope of the regression line indicated that approximately 15 to 22% of the total biosolids N was recovered in available forms during the fallow sampling, and 16 to 25% was



recovered in available forms during the spring of the crop year. Application time (fall vs. spring) did not affect the availability of biosolids N at either location. This N availability reported was for biosolids left on the soil surface for an extended period, allowing volatilization of ammonia to occur.

Biomass (grain + straw) N uptake values increased with biosolids rate. Biomass N uptake was described by a linear regression model. The slope of the regression line indicated that approximately 10-11% of the total biosolids N was recovered in the above ground biomass the first year after application. Crop N uptake efficiency ranged from 45-90% (for all biosolids treatments) and averaged 90% (for the anhydrous ammonia control) of the available soil N recovered in the soil profile in the spring. Biomass uptake efficiencies were higher than those reported in other studies. The higher uptake efficiency values in the present study are attributed to organic-N mineralization after spring soil sampling.

Biosolids application increased the Bray-1 P and DTPA extractable Zn soil test values in the surface (0-15 cm) sampling following harvest of the winter wheat crop (P-95) and in the spring of the crop year (M-96). Application of biosolids at the agronomic rate increased soil test P values, maintained or increased DTPA-Zn values, and increased extractable  $\text{SO}_4\text{-S}$  (during fallow) over levels observed with the AA control. Soil samples collected during the fallow at M-96 revealed that approximately 14% of the total S applied was recovered as  $\text{SO}_4\text{-S}$ . Surface (0-15 cm) soil testing revealed that there was no effect of biosolids on the amount of  $\text{SO}_4\text{-S}$  recovered in the spring of the crop year (M-96) or following grain harvest (P-95).

Biomass (grain + straw) P, S, and Zn uptake values were not significantly different at P-95, but increased with biosolids application rate at M-96. The higher yields observed at M-96 resulted in increased uptake values. Although biomass uptake values were increased at M-96, the amount of increase was small in comparison to the amount of P, S, and Zn applied.

The effect of biosolids on surface (0-15 cm) soil pH and SMP buffer pH, varied depending on sampling time. No effect on pH was observed following grain harvest (P-95). Sampling in the spring of the crop year (M-96) provided significant differences between treatments and biosolids application time. Soil pH decreased 0.2 to 0.3 units

with the agronomic rate of biosolids compared to the AA control. Biosolids at the agronomic rate reduced SMP buffer pH by 0.05 to 0.10 units, compared to the AA control. The agronomic rate of biosolids had no effect on electrical conductivity. The pH changes observed followed biological activity, as evidenced by the different responses between sampling times (and locations).

Agronomic rates of biosolids increased the availability of DTPA-extractable Cu (M-96) and exchangeable Ca and Na (P-95). Small increases in hot water B (P-95) and DTPA-extractable Fe (M-96) were observed. Analysis of the grain showed no increase in elemental concentrations. Small increases in the concentrations of some nutrients (K, Ca, Mg, and Mn) were observed with the straw. Although these increases were statistically significant, they were associated with the highest rate of biosolids only.

The detrimental effects of excess N at the high biosolids application rate, such as lodging, grain shrivel, high grain N concentration (> 2.0 %) and increased residual NO<sub>3</sub>-N were not apparent at either location.

Biosolids were a good nutrient source for soft white winter wheat based on plant and soil responses at our locations. Future research focusing on the long term effects of biosolids on residual soil fertility is needed.

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## CHAPTER 3

FLAG LEAF CRITICAL NUTRIENT CONCENTRATIONS FOR DRYLAND WHEAT  
GRAIN YIELDS FOLLOWING BIOSOLIDS APPLICATION

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## ABSTRACT

Biosolids managers need to provide evidence that adequate N was supplied by biosolids applications. A sample that is easily obtained, and does not require large capital investments is desired. This study was conducted to evaluate the use of flag leaf N concentrations as an indicator of maximum grain yield following a one-time biosolids application to soft white winter wheat (*Triticum aestivium*) and develop a critical nutrient concentration (CNC) for flag leaf N concentrations. In addition, grain, straw, and flag leaf tissue analyses were compared as indicators of phosphorus (P), sulfur (S), and zinc (Zn) status. The effect of no fertilizer, anhydrous ammonia, and increasing rates of biosolids on soft white winter wheat tissue nutrient concentrations was investigated at two locations in Sherman County, OR. The relationship between flag leaf N concentrations and grain yields was consistent over the two years, despite differences among the locations, maximum yields, and wheat variety. The linear-plateau method was used to determine the CNC at both locations. Sampling the flag leaf during the flowering growth stage produced the best correlation. Critical nutrient concentrations were found to be 3.5 to 3.7% N at heading (Feekes 10-10.1), 3.0 to 3.2% N at flowering (Feekes 10.5-10.51), and 2.8 to 3.0% N at milk (Feekes 11.1). Flag leaf N concentrations were also correlated with grain N ( $R^2 = 0.66$  to  $0.69$  at flowering). The average coefficient of variation (CV) for the flag leaf sampled at flowering (Feekes 10.5-10.51) was 8% for N, 11% for S, 8% for P, and 9% for Zn. The precision of flag leaf nutrient determinations was similar to that observed with grain (CV = 8% for N, 8% for S, 7% for P, and 16% for Zn) and superior to that for straw (CV = 27% for N, 35% for S, 26% for P, and 30% for Zn).

Due to the speed and simplicity of sampling, a wide range of nutrient concentrations in response to added nutrients, and low CV values, flag leaf samples are a suitable plant part to sample for assessment of plant N, S, and Zn sufficiency. No relationship between soil test P and flag leaf P concentration was observed. Future research at more field locations is needed to confirm flag leaf critical nutrient concentrations (N) over a wider range of growing conditions.

## INTRODUCTION

Biosolids managers need to provide evidence that adequate nitrogen (N) was supplied by biosolids applications. A procedure that is easily performed and does not require large capital investments is desired. The options available for wastewater treatment managers include soil sampling/analysis and plant tissue sampling. Plant sampling has many advantages over soil sampling. These advantages are based on: (i) verification of N status (adequate, deficient, excessive), (ii) effort to obtain, (iii) sample size versus total area being investigated, and (iv) risk of sample contamination.

Biosolids managers' goals are to operate an environmentally friendly facility, while providing farmers with a valuable plant nutrient resource (Forste, 1997). Biosolids managers need to provide evidence that adequate N was supplied by biosolids, and that the N supply was not excessive (Bastian, 1994). Postharvest soil samples can be taken to assess whether N was provided in excess of crop requirements (Sullivan et al., 1993; USEPA, 1994). Monitoring of N availability is necessary for internal management purposes and also for cooperating growers who receive the biosolids.

Plant tissue testing is an approach to assess available N supply relative to crop needs. Soil testing is an accepted method for estimating the amount of available soil N supplied by biosolids, but it is best used in conjunction with plant tissue analyses (Havlin, 1999).

In contrast to soil sampling, plant tissue sampling does not require any special equipment or long training periods (Havlin et al., 1999; Jones and Case, 1990). Another benefit of plant tissue sampling is that the risk of contamination is minimal in comparison to soil sampling (James and Wells, 1990; Jones and Case, 1990). In contrast to the small area that a soil sample represents, a single plant sample can represent approximately 0.03% of the total plot, using estimates for dryland winter wheat root area (Havlin et al., 1999). Since N is a mobile nutrient, sampling the youngest, fully developed leaf will give the best representation of plant N status (Munson and Nelson, 1990). Sampling very mature leaves provides lower N concentrations and sampling immature leaves will not give an accurate prediction of the plant's nutritional status. Sampling a specific leaf is desirable because it is quick, easily recognized and obtained, and easily repeatable



throughout the field in a short period of time; whereas, sampling the entire above ground portion of the plant is time consuming and labor intensive, and generally not recommended (Jones and Case, 1990). It must be considered however, that tissue sampling is only an indication of nutritional status at the time of sampling (Havlin et al., 1999). Plant samples have been used to determine if optimal or above-optimal available N was supplied by manure applications (Hooker and Morris, 1999). Early season leaf tissue sampling has been used to estimate the spring N requirements based on N concentrations (Vaughn et al., 1990a; Papastyliano et al., 1984). This practice is a valuable management tool but is based on an estimation of yield potential. The early season leaf tissue samples are collected prior to the rapid growth of the crop and therefore any inclement weather during the period of rapid growth would decrease the yield potential.

Previous studies have correlated plant tissue N concentrations with maximum yield response. The N concentration that correlates with maximum yield, or 90 to 95% of maximum yield, is considered the critical nutrient concentration (CNC). There is a wide range of critical nutrient concentrations reported, due to the dependence on physiological leaf age, wheat variety, and climate.

As a plant matures, N concentrations in the tissue decline (Hanway, 1962; Havlin et al., 1999). The decline in N concentrations is due to the increased biomass accumulation and decrease in N uptake. There are three distinct phases of N uptake for a winter wheat crop: (i) slow uptake, (ii) rapid uptake, and (iii) redistribution within the plant (Sullivan et al., 1999). For winter wheat, the rapid N uptake period begins at tillering (Feekes 5) and ends at head emergence (Feekes 10). During the last stage, N is redistributed within the plant (from leaves to grain) and coincides with continued biomass accumulation.

Wheat variety plays a role in N fertilization goals (Munson and Nelson, 1990; Karow, 1994). In hard red wheat management systems, excess N is desired for increased protein levels. Nitrogen (N) is normally applied at rates above what is required for maximum yields with the anticipation that luxury consumption will occur. Luxury consumption is an increase in nutrient concentrations above the level required for maximum yield, and is common in most plants (Engels and Marschner, 1995; Havlin,

1999). Luxury consumption increases the N concentration in the grain without increasing yields. In contrast to the hard red wheat systems, soft white winter wheat production systems are managed for low protein. Managing for low protein means that excess N for maximum grain yields is not desired. The differences in protein goals will consequently have implications on the CNC reported for different wheat varieties. McNeal et al. (1966) found significant differences in leaf dry matter contents of different wheat varieties but only slight differences in leaf N uptake. The leaf N concentrations ranged from 3.2 to 4.0% N at heading (Feekes 10) and 3.0 to 3.5% N at flowering (Feekes 10.5-10.51).

Climatic differences between locations influence the CNC reported for winter wheat. In north-central Oregon, the most limiting factor for grain yield is moisture. Plant tissue N concentrations corresponding with maximum yield in dryland cropping systems are expected to be lower than those observed in areas which are irrigated (or not as severely moisture limited). Vaughn et al. (1990a) reported that discrepancies between reported critical N levels could be due to different geographical regions. They reported that for soft red winter wheat at Feekes 4, the critical whole plant N value ranged from 4-5% in Virginia to above 4% in Pennsylvania.

Despite differences in variety and geographic location, reported critical nutrient concentrations for wheat generally range from 3.5 to 4.75% N (Donohue and Brann, 1984; Follett et al., 1992; Wuest and Cassman, 1992; Tindall et al., 1995). Others have attempted to correlate spring N fertilization requirements with plant tissue N concentrations. Dryland hard red winter wheat leaf nitrogen concentrations at tillering (Feekes 5) were used to determine spring N fertilization recommendations for the western Great Plains Region (Vaughn, et al. 1990a; Vaughn, et al. 1990b). Vaughn et al. (1990a) reported that hard red winter wheat leaf N concentrations of 3.8% and 3.5% were critical concentrations for Feekes 5 and Feekes 7 growth stages, respectively

In addition to environmental variables like physiological leaf age, wheat variety, and geographical differences, researchers use different methods for estimating the CNC. The most common method employed by researchers is to correlate plant tissue N concentrations with near-maximum grain yields (90-100% of maximum). Donohue and Brann (1984) and Papastyliano et al. (1984) used regression analysis to determine CNC ranges for winter wheat that correlated with maximum grain yields. They fit quadratic

regression lines to the relationship of relative percent maximum yield (y-axis) and N concentration in the plant tissue (x-axis).

In addition to supplying N, biosolids applications supply other nutrients such as phosphorus (P), sulfur (S), and zinc (Zn). Studies have shown that when organic byproducts are applied at agronomic N rates, excess P is supplied (Pierzynski, 1994; Edwards, 1997). The amount of S required by most crops is small in relation to the amount of N required (Oregon State University, 1980; Havlin, 1999). Sulfur (S) has been shown to be deficient in Oregon, due to the low levels in some soils. Fertilization of grain crops with S is sometimes recommended (Western Fertilizer Handbook, 1985; Brengle, 1982). Zinc (Zn) is required in small quantities by most crops (Murphy and Walsh, 1972; Lindsay and Norvell, 1978; Kabata-Pendias and Pendias, 1992; Havlin et al., 1999). Deficiencies are generally observed on alkaline, calcareous soils with low amounts of organic matter (Lucas and Knezek, 1972; Kabata-Pendias and Pendias, 1992). In dryland cropping, N fertilization can induce Zn deficiency in cereals on soils with marginal levels of Zn (Brengle, 1982). Monitoring biosolids application sites for these nutrients is desirable for future management decisions by biosolids managers. The ability to know which plant part (grain, straw, or flag leaf) gives the best representation of the status of these nutrients will aid in these decisions. Previous studies showed that only 12% of the variability between cumulative P added and grain P concentrations, and only 54% of the variability between cumulative Zn applied and grain Zn concentrations could be explained by linear regression (Barbarick et al., 1995). Although correlations did exist, evaluation of other plant parts could identify tissue with greater sensitivity to changes in available soil nutrient concentrations.

The objectives of this study were to: (i) determine if flag leaf N concentrations could be used as indicators of sufficient available N for maximum grain yields and low grain N concentrations for biosolids applied to dryland soft white winter wheat in central Oregon, (ii) estimate flag leaf N critical nutrient concentrations for maximum yields under our growing conditions, and (iii) evaluate plant tissue (grain, straw and flag leaves) as indicators of phosphorus (P), sulfur (S), and zinc (Zn) status.

## MATERIALS AND METHODS

### Field Study

Agronomic rates of anaerobically-digested biosolids for soft white winter wheat (wheat-fallow rotation) were evaluated for two field locations in Sherman County, OR. Club wheat (Rohde) was seeded 15-Oct.-96 at P-95. Common soft white wheat (Stephens/Madsen) was seeded 20-Sept.-97 at M-96. Field locations were designated as the Pinkerton (P-95) site and the McClennan (M-96) site. Locations are referred to by the first letter of the location and year of first biosolids application. Agronomic rates were determined by comparing crop responses to biosolids with responses to anhydrous ammonia and calculating the range of biosolids rates that corresponded with 90 to 95% of maximum grain yields. At both locations, a yield response to N application was observed (Chapter 2). Biosolids were applied at three rates (Table 3.1): low (150 to 190 kg N/ha), medium (248 to 382 kg N/ha), and high (474 to 572 kg N/ha). Biosolids applied at these rates supplied up to 140 kg available N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) per ha as determined by soil testing at tillering (Feekes 2 and 5) during the spring of the crop year. Ammonium-N ( $\text{NH}_4\text{-N}$ ) comprised 1.2 to 1.7% of biosolids total N at application. The majority of the  $\text{NH}_4\text{-N}$  was probably volatilized as  $\text{NH}_3$  from the soil surface shortly after application. Biosolids remained on the soil surface for at least 1 month (depending on application date) before the first tillage. The pH of the biosolids was high ( $\cong 8.0$ ) favoring rapid ammonia ( $\text{NH}_3$ ) loss. Ammonia ( $\text{NH}_3$ ) volatilization studies with similar anaerobically-digested dewatered biosolids have demonstrated loss of 50 to 100% of the initial  $\text{NH}_4\text{-N}$  present within 7 days of surface application (Henry et al., 1999).

A complete discussion on the experimental design and treatments is given in Chapter 2, Materials and Methods.

**Table 3.1.** Nutrient application rates and timing. Pinkerton Farm (P-95) and McClennan Farm (M-96), Sherman County, OR. 1995-1997.

Location	Treatment <sup>a</sup>	Application Date	Biosolids Rate	Total Nutrients Applied <sup>b</sup>					
				NH <sub>4</sub> -N	N	P	S	Zn <sup>c</sup>	
			Mg/ha	-----kg/ha-----					
P-95	No fertilizer	-	0	0	0	0	0	0	
	AA	03-Jul-96	0	0	56	0	0	0	
	BS1-Fall	06-Nov-95	3.1	42	151	97	33	1.5	
	BS2-Fall		5.2	71	248	162	55	2.5	
	BS3-Fall		9.9	136	474	309	104	4.8	
	BS1-Spring	23-Apr-96	3.1	52	165	70	30	1.8	
	BS2-Spring		5.2	87	270	117	50	3.1	
	BS3-Spring		9.9	166	517	223	96	5.9	
	M-96	No fertilizer	-	0.0	0	0	0	0	0
		AA	09-Jun-97	0.0	0	67	0	0	0
BS1-Fall		16-Oct-96	3.8	45	160	131	40	2.0	
BS2-Fall			7.6	90	320	261	79	3.9	
BS3-Fall			11.4	136	480	392	119	5.9	
BS1-Spring		25-Apr-97	3.8	52	191	101	40	2.1	
BS2-Spring			7.6	105	382	202	80	4.3	
BS3-Spring			11.4	157	572	303	120	6.4	

<sup>a</sup> AA=Anhydrous ammonia; BS=Biosolids applied to standing stubble the fall after crop harvest, or in the spring prior to the first Fflow tillage.

<sup>b</sup> Based on biosolids N, P, and S analyses (Materials and Methods) performed by AgriCheck, Inc., Umatilla, OR.

<sup>c</sup> Based on biosolids Zn analysis (Materials and Methods) performed by OSU Central Analytical Lab, Corvallis, OR.

## Plant Sampling

Grain (1 kg subsample) and straw (450 g subsample) were collected at final harvest and prior to final harvest, respectively. Harvest dates and other data collection protocols are described in Chapter 2.

Wheat development stages were evaluated with the Feekes scale, a numerical system for describing the physical plant changes from the first-leaf stage through grain ripening (Nelson et al., 1996). Approximately 30 flag leaves were collected from each plot at three to four development stages (Table 3.2). The flag leaf is the last leaf to emerge immediately prior to heading and just after the third above-ground node is observed.

Nitrogen deficiency often accelerates plant maturity. This difference in maturity poses a problem for experiments with multiple N rates. In the present experiment, the overall development stage for each sampling was estimated for the plants from the medium biosolids rate plots. Thus, the development stage reported is most reflective of the development stage at the agronomic rate.

Records from the Moro Experiment Station (5 to 10 km from field locations) were used to compare the relationship between calendar date and growing degree days for both locations. Cumulative growing degree days were calculated by averaging the maximum and minimum daily temperature (Celsius). Cumulative degree days were then calculated by summing up the daily values from seeding to final harvest, or from 1-Jan. to final harvest. In some cases, the value was less than 0°C. This value was the base temperature and meant that all values less than 0°C were considered equal to zero.

## Plant Analysis

Plant tissue (grain, straw, flag leaf) samples were dried and ground to pass a 1-mm screen. Plant tissue N and S were determined by Leco CNS 2000 (Leco Corporation, St. Joseph, MI) combustion analysis (Sweeney, 1989). Plant samples for P and Zn analysis were digested by adding 2.0 mL of 30% hydrogen peroxide and 0.5 mL of

**Table 3.2.** Sampling dates, development stages, and growing degree days for flag leaf sampling. Sherman County, OR. 1996-1998.

Location	Sampling Date	Feekes Development Stage <sup>a</sup>	Growing Degree Days <sup>b</sup>	
			Seeding	1-Jan
P-95	22-May-97	Head Emergence (10-10.1)	1192	905
	04-Jun-97	Flowering (10.5-10.51)	1385	1098
	10-Jun-97	Milk (11.1)	1476	1189
M-96	13-May-98	Head Emergence (10)	1508	810
	23-May-98	Early Flowering (10.5)	1604	906
	09-Jun-98	Late Flowering (10.51)	1830	1133
	18-Jun-98	Milk (11.1)	1970	1272

<sup>a</sup> Physical description followed by numerical stage.

<sup>b</sup> Growing degree days calculated from seeding or 1-Jan. with 0°C base temperature

concentrated nitric acid to 0.25 grams of plant tissue and digesting in a MDS-2000 microwave digester (CEM Corporation, Matthews, NC; Gavlak et al., 1994). Elemental concentrations were determined with a Perkin-Elmer Optima 3000 DV Mass-spec ICP (Perkin-Elmer Corporation, Norwalk, CT).

### Determination of Critical Nutrient Concentration

To determine the flag leaf N concentration that provided sufficient N for "near-maximum" grain yields, flag leaf N was plotted against grain yield. The point at which near-maximum grain yields are obtained is defined as the point at which the yield response function approached a slope of zero. The linear-plateau method was used to determine the critical nutrient concentration (CNC) at both locations for the relationship between flag leaf N and grain yield. Regression analysis showed that a significant linear relationship existed between grain yield and flag leaf N for the unfertilized control, anhydrous ammonia control, and the low and medium rates of biosolids. Although quadratic regression was significant, it did not provide an increase in precision, as indicated by stepwise regression. This relationship was consistent at both locations. Linear regression was also performed on the relationship between grain yield and flag leaf N for the medium and high rates of biosolids application. Regression analysis showed that no significant relationship occurred. Due to the non-significance of the regression, it was determined that a plateau relationship existed. The plateau level was calculated by taking the average of the grain yields for the medium and high rates of biosolids. The point at which the linear regression line intersected the plateau portion of the graph was deemed the CNC. Above the CNC, grain yields did not increase with higher flag leaf N concentrations.

The linear-plus-plateau model used here is similar to that used by other in describing yield responses to N fertilization (Cerrato and Blackmer, 1990).

The linear-plus-plateau model is defined by Eq. [1]

$$Y = a + bX; \text{ if } X < C \quad [1]$$

$$Y = P \quad ; \text{ if } X \geq C$$



where

$Y$  = grain yield (kg/ha)

$X$  = flag leaf N concentration (%)

$a$  = intercept (kg grain/ha)

$b$  = linear coefficient (kg grain/kg N applied)

$C$  = critical nutrient concentration: the flag leaf N concentration which occurs at the intersection of the linear response and plateau response lines

$P$  = plateau grain yield (kg/ha)

Flag leaf N was also plotted against grain N concentrations (y-axis). Simple linear regression was performed on the entire range of data. Although linear regressions were significant at both locations, use of a quadratic equation increased the precision as indicated by the  $R^2$  values at P-95, but not at M-96. This precision was not increased with the use of a cubic equation at either location.

At P-95, there were two plots in which the wheat was desiccated early in the grain-fill period because of very shallow (< 30 cm) and rocky soil. These two plots were both in the high rate of biosolids (one spring and one fall applied) and located on adjacent plots at the edge of the experiment area. Due to these reasons, these two plots were determined to be "outliers". Regression analysis was performed with and without these plots. Inclusion of these plots decreased the precision of the regression analyses and actually produced non-significant results; therefore, graphs show regression line for data set minus the outliers.

**Abbreviations:** AA, Anhydrous ammonia; BS, Biosolids; CNC, Critical nutrient concentration; M-96, McClennan Farm location; P-95, Pinkerton Farm location.

## RESULTS AND DISCUSSION

In order for calibration of critical nutrient concentrations to be successful, four main criteria must be satisfied. These criteria include: significant response to N fertilization, wide range of N concentrations (from deficient N to excessive N

availability), adequate precision of regression model, and low variation of tissue N concentrations within a sampling unit (fertilizer treatment). Leaf N concentrations must be significantly lower for unfertilized plants than plants with adequate and excessive amounts of N applied. The range of leaf N concentrations between plants with deficient and adequate N supply should be wide. The regression model should be able to explain a majority of the variation as indicated by the  $R^2$  value. Lastly, the coefficient of variation (CV) values within the same fertilizer rate should be low, reflecting high precision.

### Flag Leaf Nitrogen and Grain Yield

The N concentrations in different plant parts all showed significant responses to increasing biosolids rate (Tables 3.3 and 3.4). Flag leaf tissue samples showed the highest sensitivity to N, followed by grain and straw tissue. Straw tissue samples produced the least amount of valuable information due to high CV values and narrow range of N concentration values. Grain samples performed better than the straw samples as indicated by a greater N concentration range and lower CV values. Flag leaf samples showed the greatest sensitivity to available N supply based on the wide range of N concentrations accompanied by low CV values. As the plant matured however, the flag leaf CV values increased. Flag leaves sampled at flowering (Feekes 10.5 to 10.51) had equivalent CV values as the grain samples, but with wider ranges in N concentrations. This means that flag leaves sampled at this development stage are the most sensitive indicator of plant N status. This relationship is further illustrated by the linear response of flag leaf N to total N applied at the flowering development stage (Figure 3.1). Although the data was collected during different years at different locations, the slope of the regression lines indicate a common response.

Flag leaf N:S ratios indicated adequate S was present, based on deficiency levels reported by others for soft white winter wheat (Rasmussen, 1996). Biosolids application increased flag leaf S concentrations at both locations. This response was more pronounced at M-96, where differences in leaf S concentrations were observed for biosolids rate and for fall vs. spring applied biosolids. At P-95, the only difference

**Table 3.3.** Anhydrous ammonia (AA) and biosolids (BS) effects on plant tissue N concentrations. Pinkerton Farm (P-95), Sherman County, OR. 1997.

Treatment	Total N Applied kg/ha	Nitrogen Concentration				
		Grain	Straw	Flag Leaf <sup>a</sup>		
				Head Emergence	Flowering	Milk
-----g/kg-----						
No fertilizer	0	15	2.2	29	24	21
AA	56	16	3.1	34	31	28
BS1-Fall	151	16	2.4	32	29	24
BS2-Fall	248	17	2.7	34	31	28
BS3-Fall	474	22	4.9	40	42	36
BS1-Spring	165	16	2.6	31	29	25
BS2-Spring	270	17	2.6	34	30	27
BS3-Spring	517	22	5.4	38	39	35
Significance		**	*	**	**	**
CV (%)		10	31	5	8	12
LSD (0.05)		3.2	1.79	3.1	4.7	5.9
<b>Contrasts</b>						
Chk vs. All		*	NS	**	**	**
Chk vs. AA		NS	NS	**	**	*
Fall vs. Spring		NS	NS	NS	NS	NS
AA vs. BS2		NS	NS	NS	NS	NS
Biosolids rate		**	**	**	**	**

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

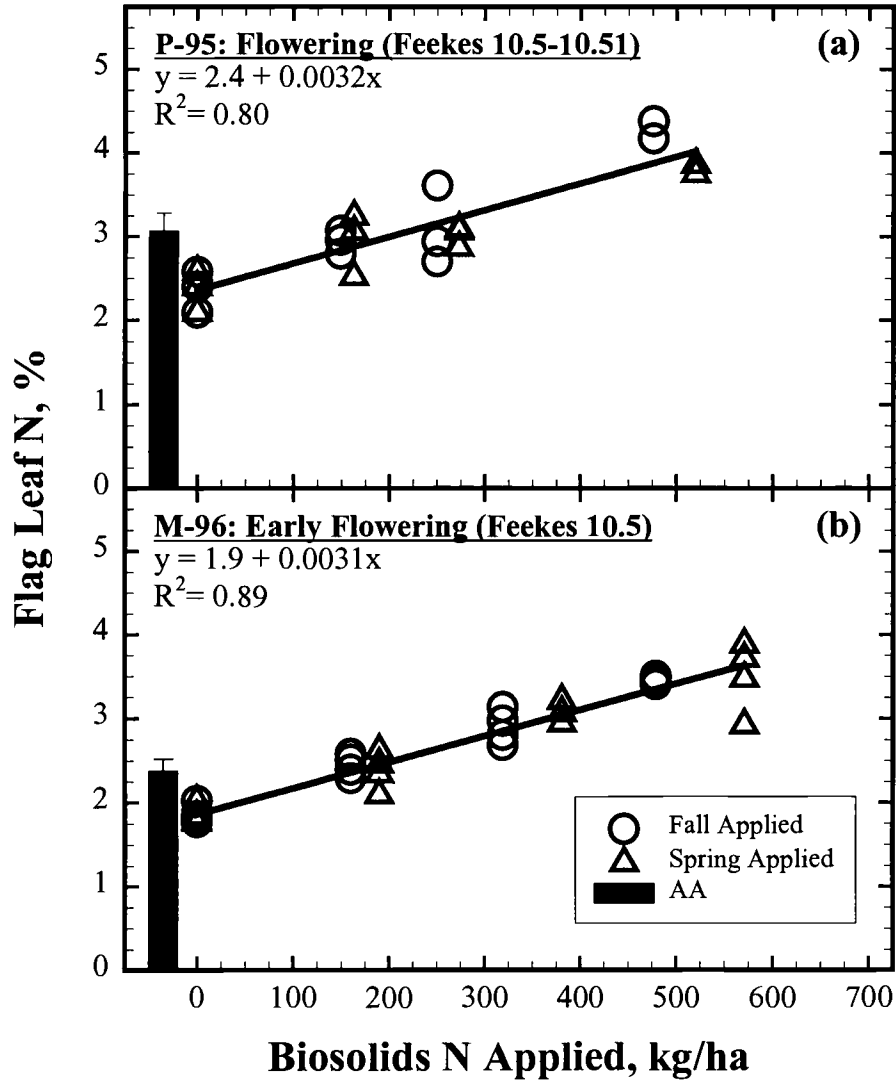
<sup>a</sup> Flag leaf sampled at head emergence (Feekes 10-10.1), flowering (Feekes 10.5-10.51), and milk development (Feekes 11.1)

**Table 3.4.** Anhydrous ammonia (AA) and biosolids (BS) effects on plant tissue N concentrations. McClennan Farm (M-96), Sherman County, OR. 1998.

Treatment	Total N Applied Mg/ha	Nitrogen Concentration					
		Grain	Straw	Flag Leaf <sup>a</sup>			Milk
				Head Emergence	Early Flowering	Late Flowering	
		-----g/kg-----					
No fertilizer	0	14	2.1	24	19	15	12
AA	67	15	2.4	31	24	21	17
BS1-Fall	160	14	2.2	29	24	22	18
BS2-Fall	320	16	2.3	33	29	28	24
BS3-Fall	480	17	3.4	38	34	34	32
BS1-Spring	191	14	2.3	28	24	21	17
BS2-Spring	382	17	3.1	35	31	29	27
BS3-Spring	572	18	3.1	37	35	34	31
Significance		**	*	**	**	**	**
CV (%)		6	23	7	7	16	16
LSD (0.05)		1.3	0.87	4.0	3.0	6.2	5.3
<b>Contrasts</b>							
Chk vs. All		**	NS	**	**	**	**
Chk vs. AA		NS	NS	**	**	NS	*
Fall vs. Spring		NS	NS	NS	NS	NS	NS
AA vs. BS2		*	NS	NS	**	**	**
Biosolids rate		**	**	**	**	**	**

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> Flag leaf sampled at head emergence (Feekes 10-10.1), early flowering (Feekes 10.5), late flowering (Feekes 10.51), and milk development (Feekes 11.1)



**Figure 3.1.** Anhydrous ammonia (AA, 56 kg N/ha at P-95; 67 kg N/ha at M-96) and biosolids (BS) effects on flag leaf N concentrations for: P-95, Flowering/Feekes 10.5-10.51 (a); and M-96, Early Flowering/Feekes 10.5 (b). Sherman County, OR. 1997-1998.

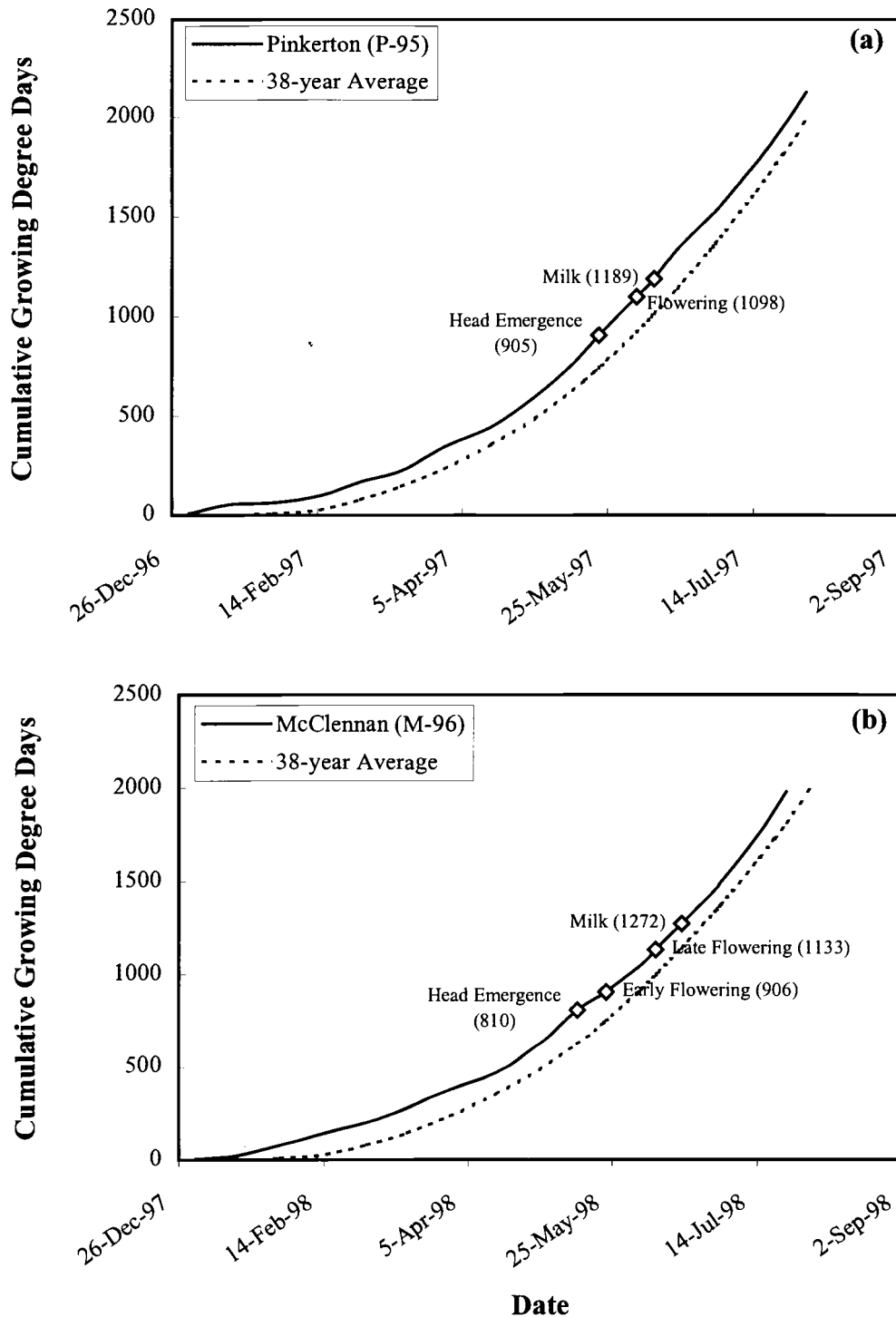
observed was with the high rate of biosolids. Although S concentrations increased with biosolids application rate, the yield response to biosolids was most likely a response to N rather than S. Although the treatments in the present experiment varied the amount of S applied, it was done in conjunction with N application. In order to accurately test if the yield response was due to S, a constant rate of N would have to be applied with varied S rates. The fact that crop response was similar for the anhydrous ammonia control, which supplied only N, and the low and medium rates of biosolids, which supplied N and S, supports the hypothesis that yield response is due largely to N and not S application.

Cumulative growing degree days calculated from seeding showed a large discrepancy (approximately 300-500 d) between locations and growth stages (Table 3.2). In contrast to this, calculating cumulative growing degree days from January 1 provided a closer match between the two locations, with approximately 100 growing degree days difference between respective growth stages (Figure 3.2). Although degree days are typically calculated from seeding, they are normally used to describe and identify disease and pest problems during early growth stages (Klepper et al., 1982). Use of growing degree days from seeding would prove difficult for biosolids managers since records would have to be maintained of seeding dates. If January 1 were used however, sampling programs would be easier for biosolids managers to plan and implement.

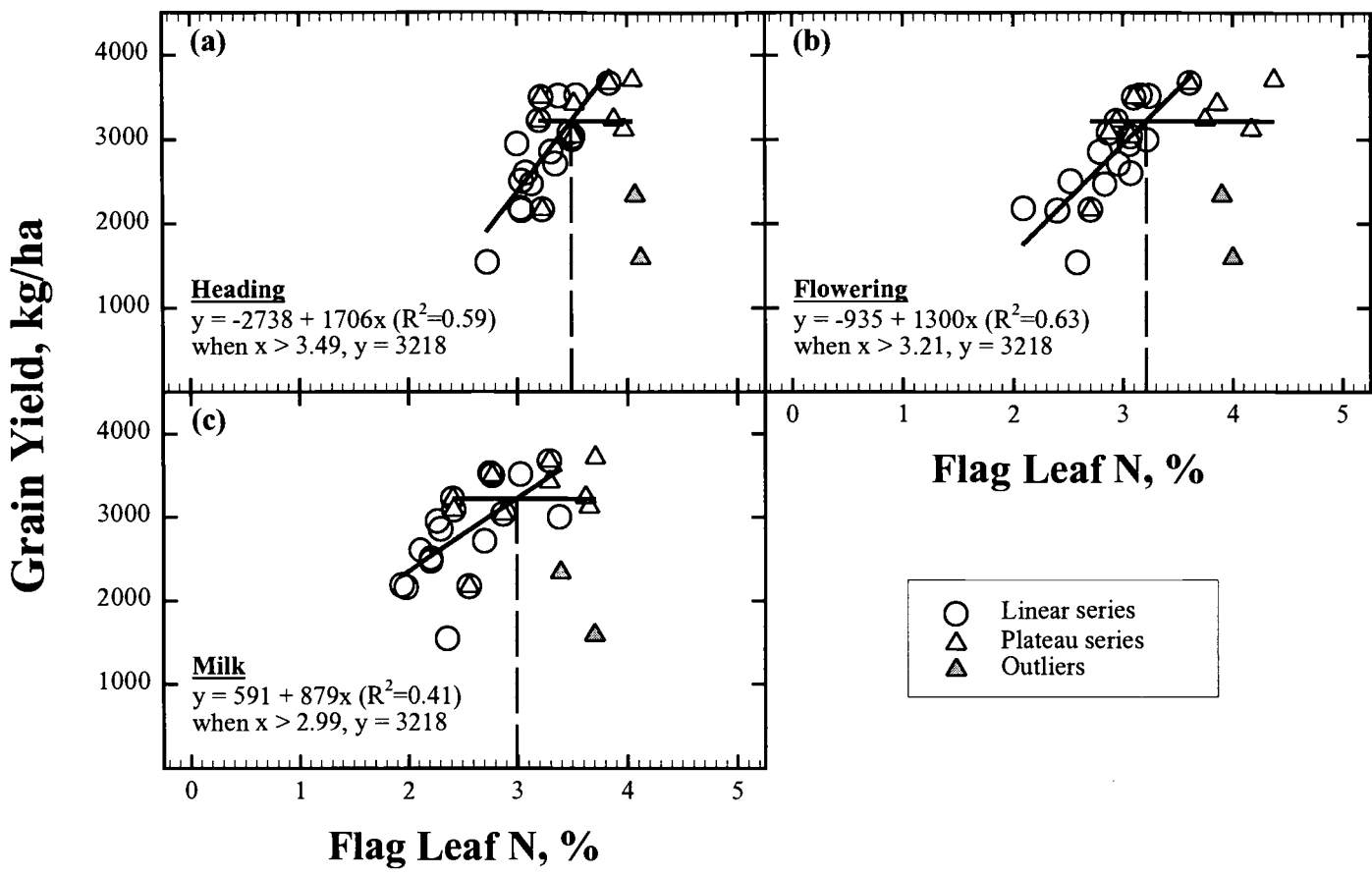
Based on the common responses observed with flag leaf N concentrations and N rate at both locations, further investigation of the relationship between flag leaf N concentrations and grain yields was justified. The further investigation consisted of the development of CNC that corresponded to maximum grain yields (Figure 3.3 and Figure 3.4).

As seen with N concentrations, the N concentration required for maximum grain yield (CNC) decreased as the plant matured. Although this CNC is reported as a single N concentration, there actually exists a range due to the natural variation in plant samples (Table 3.3 and Table 3.4).

In general, the range of flag leaf N concentrations increased as the plant matured. The increase in N concentration ranges was accompanied by an increase in CV values, ranging from 5% at head emergence up to 16% at milk (Table 3.3 and Table 3.4). The

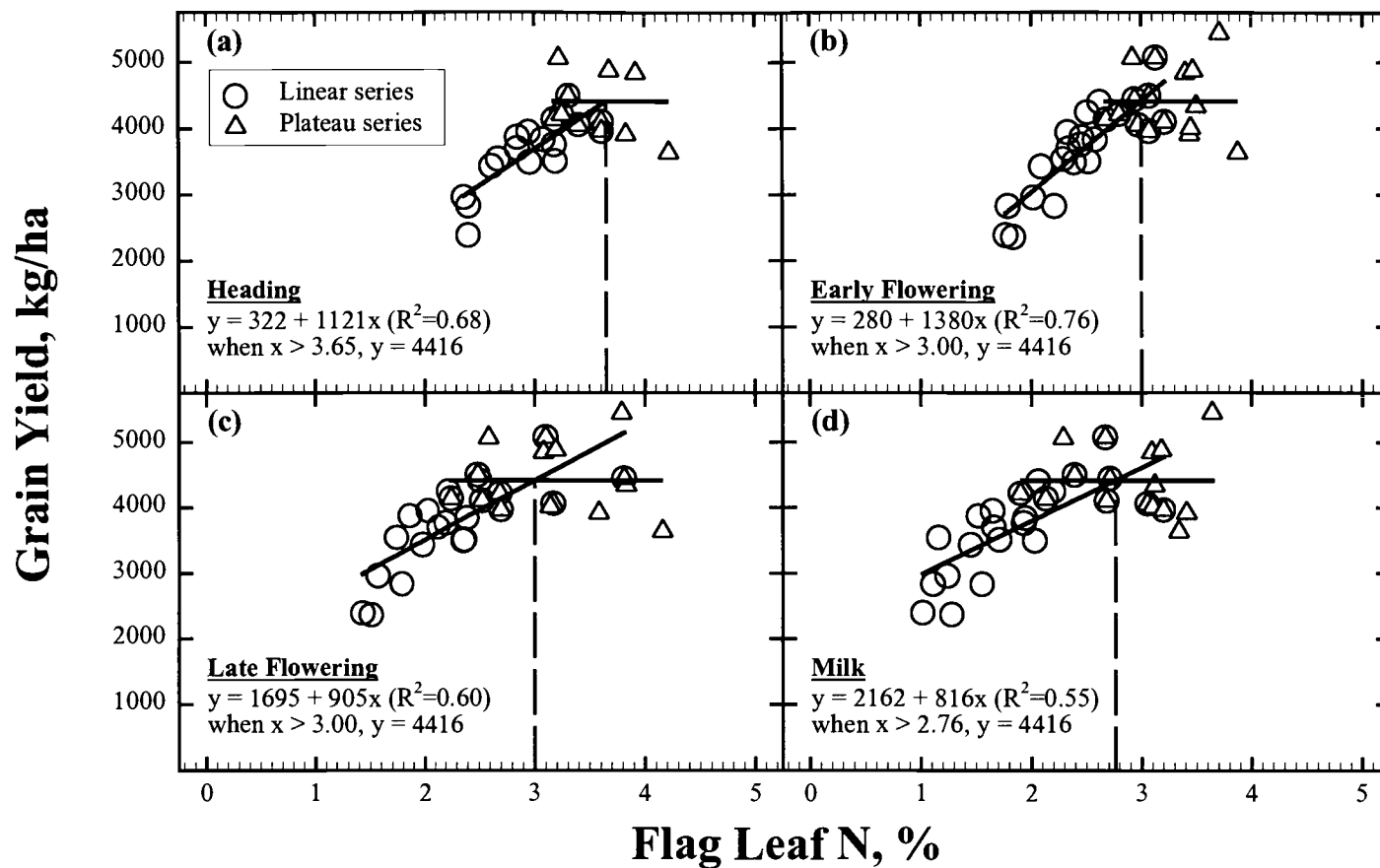


**Figure 3.2.** Cumulative growing degree days (from 1-Jan) as related to calendar date for P-95 (a); and M-96 (b). Symbols represent flag leaf sample times. Values are corresponding cumulative growing degree days. Sherman County, OR.



**Figure 3.3.** Relationship between flag leaf N concentration and grain yield at: heading/Feekes 10 (a); flowering/Feekes 10.5-10.51 (b); and milk/Feekes 11.1 (c). Pinkerton Farm (P-95), Sherman County, OR. 1997. Vertical dashed line indicates critical nutrient concentration (CNC) level as determined by intersection of linear and plateau regression lines (outliers not included).





**Figure 3.4.** Relationship between flag leaf N concentration and grain yield at: heading/Feekes 10 (a); early flowering/Feekes 10.5 (b); late flowering/Feekes 10.51 (c); and milk/Feekes 11.1 (d). McClennan Farm (M-96), Sherman County, OR. 1998. Vertical dashed line indicates critical nutrient concentration (CNC) level as determined by intersection of linear and plateau regression lines.

flowering development stage was determined to be the most suitable development stage. Coefficient of variation (CV) and  $R^2$  values increased at the milk development stage. Sampling during head emergence would have the benefit of a low CV value, but it also has a narrow range between deficient and adequate N concentrations. The CV values have an effect on the CNC range expected within a sample unit. For the flowering development stage at P-95 for example, the CNC was found to be 3.21%. With a CV value of 8%, the critical nutrient range (CNR) is  $3.21\% \pm 0.25\%$ , or 2.96-3.46% for a sample.

The CNC found for two locations in Sherman County, OR ranged from 3.5 to 3.7% at heading, 3.0 to 3.2% at flowering, and 2.8 to 3.0% at milk (Figure 3.3 and Figure 3.4).

Our values are generally lower than those reported in other studies with different wheat varieties and geographical locations. In a similar dryland environment (Colorado), the CNC for maximum grain yields have been reported as 3.4 to 4.3% at Feekes 5 (Follett et al., 1992).

Research has also been conducted for irrigated, hard red spring wheat in the western states. Tindall et al. (1995) found that flag leaf N concentrations of 3.7 to 4.3% at heading (Feekes 10) for hard red wheat grown in Idaho correlated with maximum grain yields. The authors found a consistent relationship between flag leaf N and grain protein. They suggested that future research should focus on the calibration of flag leaf N concentrations with grain yield and grain protein for specific varieties and production areas.

Others have also reported a plateau relationship between flag leaf N concentration (above the CNC) and grain yield for flood-irrigated wheat grown in California (Wuest and Cassman, 1992). Flag leaf N concentrations of 2.9%, sampled 14 days after flowering, corresponded to maximum grain yields. The authors also found that increasing N rates did not increase grain yield, but increased flag leaf N concentration. Cogger et al. (1998) reported that flag leaf (sampled at heading, Feekes 10-10.1) N concentrations increased with biosolids rates, while soft-white winter wheat grain yield did not.

Research in the southeastern states related flag leaf N concentration to grain yield for soft red winter wheat. In the Coastal Plain of Virginia, flag leaf N concentrations of

4.4 to 4.7% at head emergence (Feekes 10) were associated with 100% of maximum yield for soft red winter wheat (Donohue and Brann, 1984). Research in Georgia on soft red winter wheat (*Triticum aestivium*) found that flag leaf N concentrations of 3.5 to 4.0% at flowering (Feekes 10.5) related to maximum grain yields (Hargrove, et al., 1983).

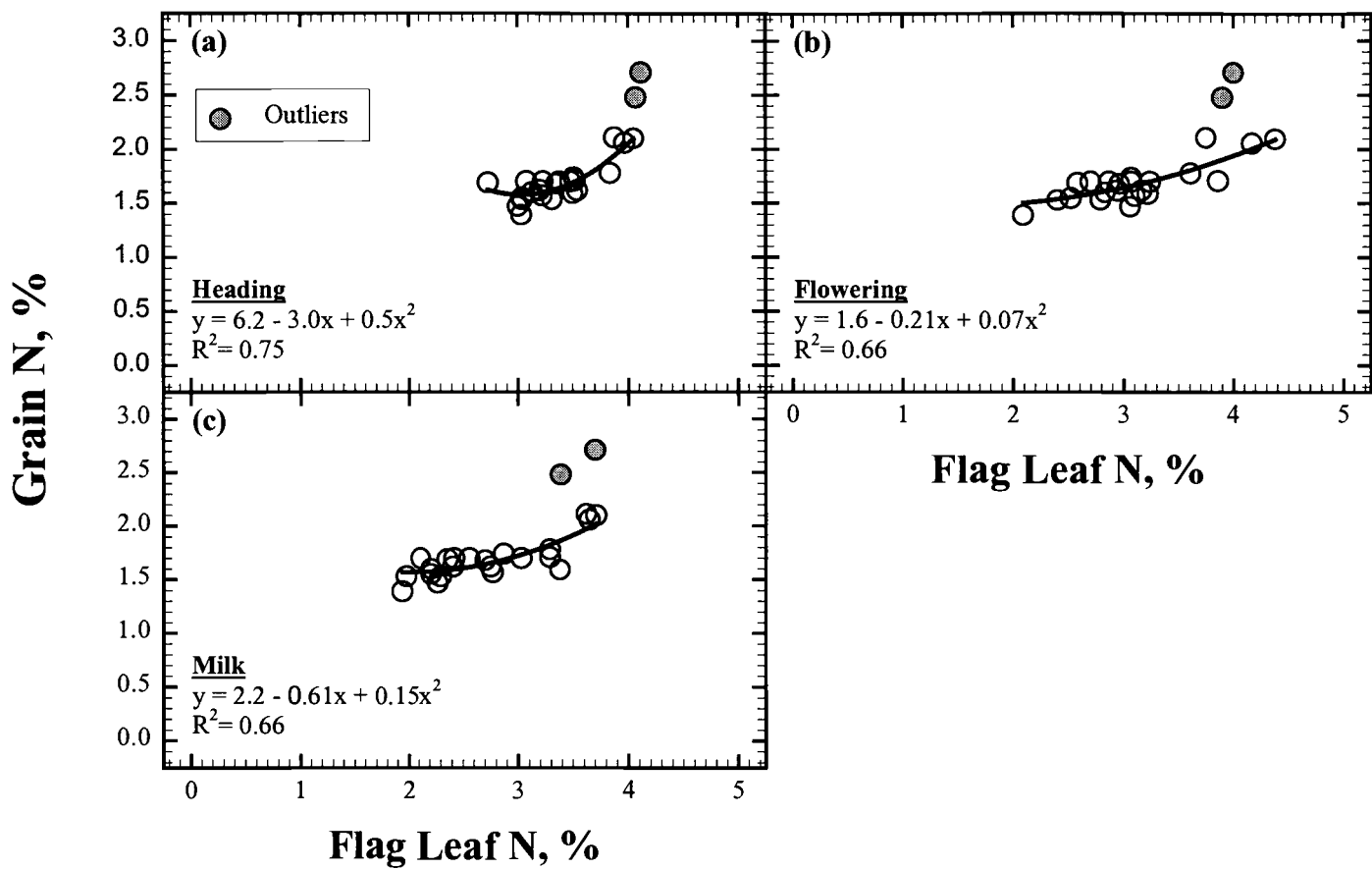
The maximum grain yields observed at P-95 (Figure 3.3) were lower than those observed at M-96 (Figure 3.4). The differences in grain yields did not affect the flag leaf CNC determined for each corresponding development stage. This suggests that the relationship between flag leaf N and maximum grain yield does not depend upon a yield forecast.

### Flag Leaf Nitrogen and Grain Nitrogen

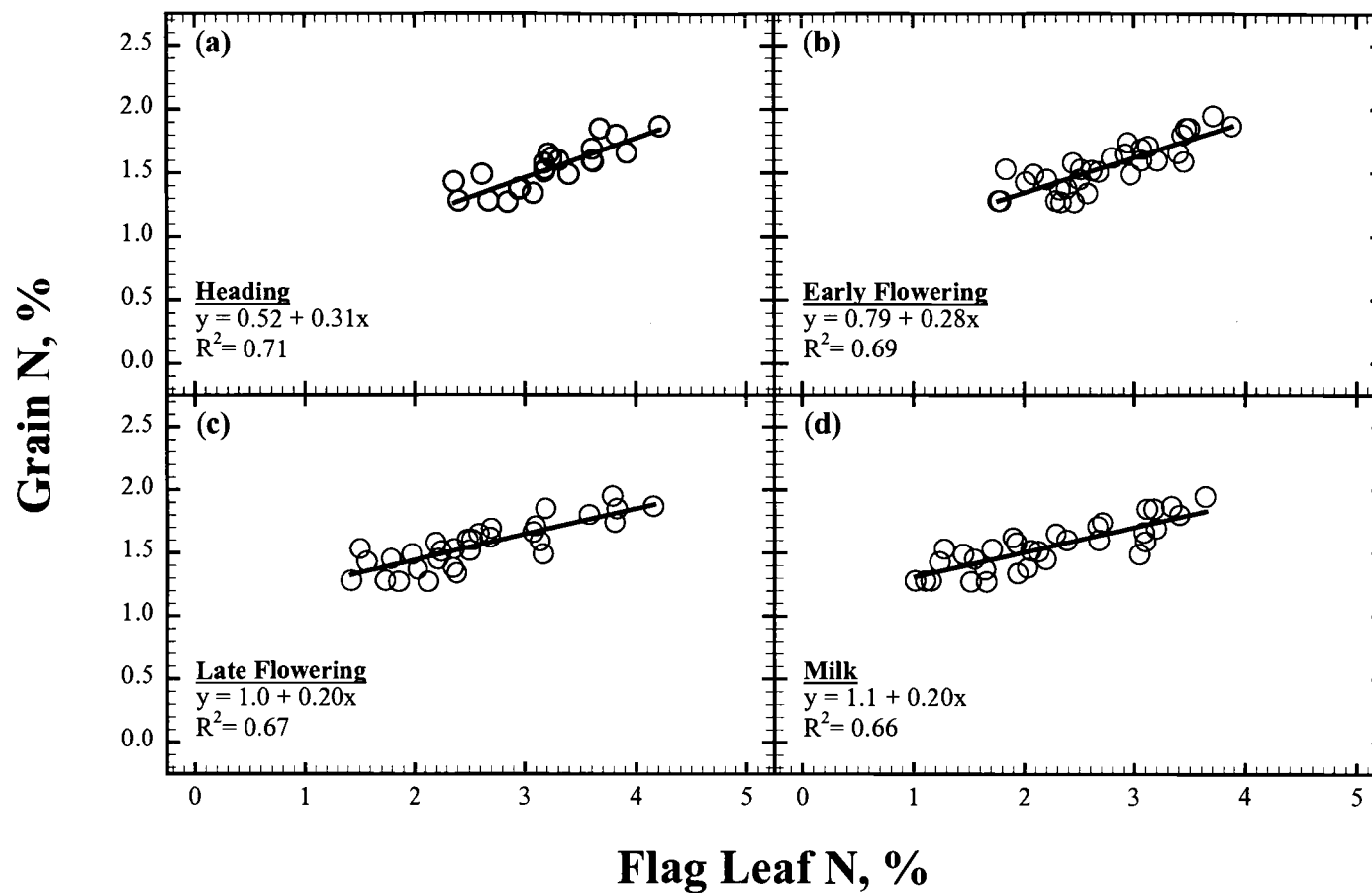
In addition to being highly correlated with grain yields, flag leaf N concentrations were significantly correlated with grain N concentrations (Figure 3.5 and Figure 3.6). Since low grain N concentrations (grain protein) are desired for soft white winter wheat production, a predictive tool that is associated with flag leaf N concentrations and grain N is desired. Sometimes a premium is paid for low protein wheat (< 1.6% grain N) although it is highly dependent on the amount of wheat buyers receive that contain low N concentrations (S. Macnab, Sherman County, OR Extension Agent, personal communication, 1999).

The relationships between flag leaf N concentrations and grain N were best described by quadratic regression at P-95 (Figure 3.5) and linear regression at M-96 (Figure 3.6). The grain N concentrations at P-95 were 1.6 to 1.8% N at the CNC (Figure 3.3). At M-96, the grain N concentrations were 1.6 to 1.7% N at the CNC (Figure 3.4).

In eastern Colorado, grain protein was used as a postharvest indicator of sufficient N for maximum hard red winter wheat yield (Goos et al., 1982). Since grain protein is highly dependent on water stress, rainfall distribution, and temperature, regardless of soil N availability, a linear yield-protein relationship did not exist in the Colorado study.



**Figure 3.5.** Relationship between flag leaf N concentration and grain N concentration at: heading/Feekes 10 (a); flowering/Feekes 10.5-10.51 (b); and milk development (c). Pinkerton Farm (P-95), Sherman County, OR. 1997



**Figure 3.6.** Relationship between flag leaf N concentration and grain N concentration at: heading/Feekes 10 (a); early flowering/Feekes 10.5 (b); late flowering/Feekes 10.51 (c); and milk/Feekes 11.1 (d). McClennan Farm (M-96), Sherman County, OR. 1998.

### Flag Leaf Tissue as an Indicator of Plant Phosphorus, Sulfur, and Zinc Status

In addition to supplying N, biosolids supplies other nutrients such as sulfur (S), zinc (Zn), and phosphorus (P). Monitoring biosolids application sites for these nutrients is desirable for future management decisions by biosolids managers. The ability to know which plant part (grain, straw, or flag leaf) gives the best representation of the status of these nutrients will aid in these decisions.

All plant parts provided detectable differences between treatments in S concentrations (Table 3.5 and Table 3.6). Flag leaf and grain samples provided higher values and greater ranges than straw samples, with lower CV values. The flag leaf was more sensitive to available S supply compared to grain. The flag leaf had a wider range of S concentrations and similar CV values, compared to the grain samples. The flag leaf samples also detected significant differences between the unfertilized and anhydrous ammonia control (Table 3.5) and fall vs. spring applied biosolids (Table 3.6).

Flag leaf samples were the only plant part to show significant differences in Zn concentrations between treatments (Table 3.5 and Table 3.6). The flag leaf CV values were low (8-10%) with a fairly large range of Zn concentrations (10 to 19 mg/kg). Grain Zn concentrations were consistently higher than flag leaf Zn concentrations, but all were well below reported values of maximum tolerable levels for domestic animals (500 mg/kg; National Academy of Sciences, 1980).

Plant P concentrations (flag leaf, grain, straw) did not correlate with P application rates (Table 3.5 and Table 3.6). All soil test values at both locations (> 39 mg/kg Bray-1 P; Table 2.5 and Table 2.6; Chapter 2) were in excess of levels required for crop production (20 mg/kg; Marx et al., 1996). The average CV values for P concentrations were 8% for flag leaf sampled at flowering (Feekes 10.5-10.51), 7% for grain, and 26% for straw.

**Table 3.5.** Anhydrous ammonia (AA) and biosolids (BS) effects on plant tissue P, S, and Zn concentrations. Pinkerton Farm (P-95), Sherman County, OR. 1997.

Treatment	Total N Applied	Phosphorus			Sulfur			Zinc		
		Grain	Straw	Flag Leaf <sup>a</sup>	Grain	Straw	Flag Leaf <sup>a</sup>	Grain	Straw	Flag Leaf <sup>a</sup>
		-----g/kg-----								
		-----mg/kg-----								
No fertilizer	0	3.2	0.23	1.9	1.1	0.3	1.7	18	2.5	11
AA	56	2.8	0.28	2.1	1.0	0.4	2.2	15	5.4	14
BS1-Fall	151	2.9	0.22	2.0	1.1	0.3	2.1	17	2.2	13
BS2-Fall	248	2.8	0.25	2.0	1.1	0.4	2.3	17	2.7	14
BS3-Fall	474	2.9	0.33	2.2	1.5	0.8	3.3	20	4.7	18
BS1-Spring	165	2.9	0.22	2.0	1.0	0.3	2.1	16	2.4	14
BS2-Spring	270	2.6	0.24	1.9	1.1	0.3	2.2	16	3.4	14
BS3-Spring	517	2.8	0.39	2.2	1.4	0.8	3.0	20	5.4	19
Significance		NS	NS	NS	**	**	**	NS	NS	**
CV (%)		7.5	31	8.4	8.7	39	10	19	56	8
LSD (0.05)		0.38	0.151	0.30	0.18	0.30	0.43	5.8	3.49	2.2
<b>Contrasts</b>										
Chk vs. All		-	-	-	NS	NS	**	-	-	**
Chk vs. AA		-	-	-	NS	NS	*	-	-	*
Fall vs. Spring		-	-	-	NS	NS	NS	-	-	NS
AA vs. BS2		-	-	-	NS	NS	NS	-	-	NS
Biosolids rate		-	-	-	**	**	**	-	-	**

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> Flowering (Feekes 10.5-10.51)

**Table 3.6.** Anhydrous ammonia (AA) and biosolids (BS) effects on plant tissue P, S, and Zn concentrations. McClennan Farm (M-96), Sherman County, OR. 1998.

Treatment	Total N Applied	Phosphorus			Sulfur			Zinc		
		Grain	Straw	Flag Leaf <sup>a</sup>	Grain	Straw	Flag Leaf <sup>a</sup>	Grain	Straw	Flag Leaf <sup>a</sup>
		-----g/kg-----						-----mg/kg-----		
No Fertilizer	0	3.3	0.50	2.3	1.0	0.3	1.5	17	2.8	10
AA	67	3.2	0.35	2.4	0.9	0.2	1.8	15	2.8	11
BS1-Fall	160	3.2	0.30	2.6	1.0	0.4	2.4	17	2.9	13
BS2-Fall	320	3.3	0.33	2.6	1.2	0.4	3.4	20	2.8	14
BS3-Fall	480	3.2	0.28	2.7	1.2	0.7	4.5	18	2.8	16
BS1-Spring	191	3.1	0.43	2.7	1.0	0.4	1.9	17	2.8	12
BS2-Spring	382	3.1	0.38	2.7	1.2	0.5	2.6	17	2.9	15
BS3-Spring	572	3.1	0.33	2.6	1.3	0.5	3.0	18	2.8	17
Significance		NS	**	NS	**	**	**	NS	NS	**
CV (%)		6	20	7	7	31	11	12	4	10
LSD (0.05)		0.27	0.107	0.27	0.12	0.19	0.41	3.1	0.17	1.9
<b>Contrasts</b>										
Chk vs. All		-	**	-	**	NS	**	-	-	**
Chk vs. AA		-	**	-	NS	NS	NS	-	-	NS
Fall vs. Spring		-	*	-	NS	NS	**	-	-	NS
AA vs. BS2		-	NS	-	**	*	**	-	-	**
Biosolids rate		-	NS	-	**	**	**	-	-	**

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> Early Flowering (Feekes 10.5)



## SUMMARY AND CONCLUSIONS

Flag leaf N concentrations can be reliably used as indicators of sufficient available N for maximum grain yields and low grain N concentrations, based on the responses observed in the present study. The relationship between flag leaf N concentrations and grain yields was consistent over the two years of this study, despite differences among the locations, maximum yields, and wheat variety. Sampling the flag leaf during the flowering growth stage produced the best correlation between flag leaf N and grain yields. Critical nutrient concentrations for the two research locations were found to be 3.5 to 3.7% N at heading, 3.0 to 3.2% N at flowering, and 2.8 to 3.0% N at milk development stages.

The relationships between flag leaf N concentrations and grain N were best described by quadratic regression at P-95 and linear regression at M-96. The grain N concentrations at P-95 were 1.6 to 1.8% N at the CNC. At M-96, the grain N concentrations were 1.6 to 1.7% N at the CNC.

Based on the significant responses to N fertilization, high  $R^2$  values, low CV values, and wide range of nutrient concentrations, flag leaf samples appear to be the best plant part to sample for determination of N. Coefficient of variation (CV) values for flag leaf S and Zn were less than or equal to those observed with the grain, and lower than those observed with the straw. Plant P concentrations (Flag leaf, Grain, straw) did not correlate with P application rates.

Future research at more field locations is needed to confirm the flag leaf N concentrations corresponding with maximum grain yield for dryland wheat fertilized with biosolids in the Pacific Northwest. The calibration of chlorophyll meter readings to the CNC would decrease sampling time and potentially save money.

There was a relationship between flag leaf N and grain N observed at both locations. The responses ranged from a quadratic (P-95) to a linear (M-96) response. There was general agreement between the predicted value (1.6-1.8%) and the observed value at the agronomic rate (1.6-1.7%). The CNC provided good approximation of grain N concentrations.

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## WINTER WHEAT RESPONSE TO NITROGEN, PHOSPHORUS, SULFUR, AND ZINC SUPPLIED BY MUNICIPAL BIOSOLIDS

### SUMMARY

For the two on-farm research locations in Sherman County, the agronomic rate for soft-white wheat production was found to be approximately 240 to 370 kg biosolids N/ha. Application of biosolids at these rates produced 90 to 95% of maximum grain yields, with no decrease in grain quality. Application rates above 370 kg biosolids N/ha did not increase grain yields, but increased grain N and plant tissue trace element concentrations. Biosolids applied at rates higher than 370 kg N/ha may increase production risks. Greater risks of lodging, grain shrivel, cheatgrass proliferation, high grain N concentrations, and increased availability of trace elements in the soil (which could lead to increased uptake) are associated with excessive biosolids application rates. For grain production, fall and spring-applied biosolids performed similarly. Increased biosolids application rates resulted in higher grain N concentrations and straw N concentrations, indicating that more than adequate N was supplied to the crop for maximum grain production.

The same general trend was observed at both locations with regards to available soil N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ). Biosolids provided 35 to 93 kg available N/ha for the first year after application. Biosolids application increased available soil N during the summer fallow and in the spring of the crop year at both locations. Biosolids application did not increase available soil N following crop harvest at either location. The slope of the regression line for each sampling date indicated that approximately 15-25% of the total biosolids N was recovered in available forms. Application time (fall vs. spring) did not affect the availability of total available N at either location.

Biomass (grain + straw) N uptake values increased with biosolids rate. Biomass N uptake was described by a linear regression model. The slope of the regression line indicated that approximately 10-11% of the total biosolids N was recovered in the above ground biomass the first year after application. Crop N uptake ranged from 45 to 90% (for all biosolids rates) of the available soil N recovered in the soil profile in the spring.

The crop N uptake averaged 90% for the anhydrous ammonia control. Biomass uptake efficiencies were higher than those reported in other studies. The higher uptake efficiency values in the present study are attributed to organic-N mineralization after spring soil sampling.

Biosolids application increased the Bray-1 P and DTPA extractable Zn soil test values in the surface (0-15 cm) sampling in the spring of the crop year (M-96) and following harvest of the soft-white winter wheat crop (P-95). Application of biosolids at the agronomic rate increased soil test P values, maintained or increased DTPA extractable Zn values, and increased extractable SO<sub>4</sub>-S (during fallow) over levels observed with the AA control. Soil samples collected during the fallow at M-96 revealed that approximately 14% of the total S applied was recovered as SO<sub>4</sub>-S. Surface (0-15 cm) soil testing revealed that there was no effect of biosolids on the amount of SO<sub>4</sub>-S recovered in the spring of the crop year (M-96) or following grain harvest (P-95).

Biomass (grain + straw) P, S, and Zn uptake values were not significant at P-95, but increased with biosolids application rates at M-96. The increased uptake values were associated with increased yields observed at M-96. Although biomass uptake values were increased with biosolids application rate at M-96, the amount of increase was small (average increases: 2.6 kg P/ha; 5.0 kg S/ha; 0.03 kg Zn/ha) in comparison to the amount applied (rates: 70 to 390 kg P/ha; 30 to 120 kg S/ha; 1.5 to 6.4 kg Zn/ha).

The effect of biosolids on surface (0-15 cm) soil pH and SMP buffer pH varied depending on sampling time. No effect on pH was observed following grain harvest (P-95). Sampling in the spring of the crop year (M-96) provided significant differences between treatments. Spring applied biosolids produced lower pH values than the fall applied biosolids at M-96). This difference was probably temporary and was attributed to the recent mineralization of organic-N, since no difference in SMP buffer pH was observed between the fall and spring applied biosolids. The agronomic rate of biosolids had no effect on EC values. The pH changes observed following biological activity, as evidenced by the different responses between sampling times (and locations). Agronomic rates of biosolids increased the availability of DTPA-extractable Cu (M-96). Small increases in hot water B (P-95) and DTPA-extractable Fe (M-96) were also observed. Analysis of the grain revealed no increase in elemental concentrations. Small increases in

the concentrations of some nutrients (K, Ca, Mg, and Mn) were observed with the straw. Although these increases were statistically significant, they were associated with the high rate of biosolids only.

The relationship between flag leaf N concentrations and grain yields was consistent over the two years, despite differences among the locations, maximum yields, and wheat variety. Sampling the flag leaf during the flowering growth stage produced the best correlation between flag leaf N and grain yields. Critical nutrient concentrations across the two field locations were 3.5 to 3.7% N at heading (Feekes 10 to 10.1), 3.0 to 3.2% N at flowering (Feekes 10.5 to 10.51), and 2.8 to 3.0% N at milk (Feekes 11.1).

The relationships between flag leaf N concentrations and grain N were best described by quadratic regression at P-95 and linear regression at M-96. The grain N concentrations at P-95 were 1.6 to 1.8% N at the CNC. At M-96, the grain N concentrations were 1.6 to 1.7% N at the CNC.

Based on the significant responses to N fertilization, high  $R^2$  values, low CV values, and wide range of nutrient concentrations, flag leaf samples appear to be the best plant part to sample for determination of N. Coefficient of variation (CV) values for flag leaf S and Zn were less than or equal to those observed with the grain, and lower than those observed with the straw. Plant P concentrations (Flag leaf, Grain, straw) did not correlate with P application rates.

Biosolids appeared to be a good nutrient source for winter wheat based on plant and soil responses. Future research focusing on long term effects of biosolids on residual soil fertility is needed. Future research at more field locations is needed to confirm the flag leaf N concentrations corresponding with maximum grain yield for dryland wheat fertilized with biosolids in the Pacific Northwest. The calibration of chlorophyll meter readings to the CNC would decrease sampling time and potentially save money.

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## APPENDICES

**Appendix A.** Monthly temperature (mean) and precipitation. Moro Experiment Station, Sherman County, OR.

<b>Date</b>	<b>Average Monthly Temperature</b>	<b>Monthly Precipitation</b>
	<sup>o</sup> C	cm
Nov-95	6.8	8.1
Dec-95	0.6	5.6
Jan-96	1.2	4.7
Feb-96	-0.3	6.2
Mar-96	5.5	1.7
Apr-96	9.0	4.0
May-96	10.4	3.7
Jun-96	15.5	0.9
Jul-96	21.9	0.4
Aug-96	20.0	0.1
Sep-96	14.4	1.4
Oct-96	9.8	3.9
Nov-96	3.7	6.7
Dec-96	1.1	10.6
Jan-97	0.3	4.0
Feb-97	2.8	2.1
Mar-97	6.1	3.3
Apr-97	8.1	3.2
May-97	14.4	1.4
Jun-97	15.7	1.4
Jul-97	19.1	0.3
Aug-97	21.5	1.4
Sep-97	16.9	1.2
Oct-97	9.8	4.1
Nov-97	5.1	1.7
Dec-97	1.3	0.7
Jan-98	1.3	6.3
Feb-98	4.1	3.3
Mar-98	6.0	2.6
Apr-98	8.2	1.7
May-98	11.5	8.0
Jun-98	15.8	0.7
Jul-98	22.7	0.7

**Appendix B.** Grain nutrient concentrations. Pinkerton Farm (P-95), Sherman County, OR. 1997.

Treatment	Biosolids Rate Mg/ha	Element									
		P	K	Ca	Mg	Na	Zn	Mn	Fe	B	Cu
		g/kg					mg/kg				
No fertilizer	0	3.2	4.7	0.43	1.3	0.19	18	48	40	3.8	2.6
AA	0	2.8	4.5	0.37	1.2	0.14	15	45	43	6.9	2.0
BS1-Fall	3.1	2.9	4.8	0.40	1.3	0.21	17	47	62	4.2	2.2
BS2-Fall	5.2	2.8	4.6	0.40	1.2	0.20	17	46	45	4.7	2.4
BS3-Fall	9.9	2.9	5.3	0.47	1.3	0.19	20	50	56	4.7	3.8
BS1-Spring	3.1	2.9	4.7	0.39	1.2	0.20	16	46	57	4.3	2.1
BS2-Spring	5.2	2.6	4.6	0.40	1.2	0.18	16	47	46	3.6	2.3
BS3-Spring	9.9	2.8	5.0	0.43	1.2	0.18	20	45	46	5.2	2.7
	Significance	NS	NS	NS	NS	NS	NS	NS	NS	NS	*
	CV (%)	7.5	8.1	8.8	6.2	65.3	19.1	9.9	26.7	38.6	21.3
	LSD (0.05)	0.04	0.07	0.01	0.01	0.02	5.80	8.08	23.07	3.17	0.93
<b>Contrasts</b>											
	No fertilizer vs. All	-	-	-	-	-	-	-	-	-	NS
	No fertilizer vs. AA	-	-	-	-	-	-	-	-	-	NS
	Fall vs. Spring	-	-	-	-	-	-	-	-	-	NS
	AA vs. BS1	-	-	-	-	-	-	-	-	-	NS
	AA vs. BS2	-	-	-	-	-	-	-	-	-	NS
	AA vs. BS3	-	-	-	-	-	-	-	-	-	**
	BS1 vs. BS2	-	-	-	-	-	-	-	-	-	NS
	BS2 vs. BS3	-	-	-	-	-	-	-	-	-	**
	BS1 vs. BS3	-	-	-	-	-	-	-	-	-	**

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

**Appendix C.** Grain nutrient concentrations. McClennan Farm (M-96), Sherman County, OR. 1998.

Treatment	Biosolids Rate Mg/ha	Element						
		P	K	Mg	Zn	Mn	Fe	Cu
		-----g/kg-----			-----mg/kg-----			
No fertilizer	0	3.3	3.4	0.9	17.5	45.5	43.3	7.2
AA	0	3.2	3.3	0.9	14.8	44.0	50.1	3.6
BS1-Fall	3.8	3.2	3.4	0.9	17.0	43.9	44.9	4.0
BS2-Fall	7.6	3.3	3.6	0.9	20.1	49.8	49.9	6.3
BS3-Fall	11.4	3.2	3.5	0.9	17.7	44.7	49.9	4.7
BS1-Spring	3.8	3.1	3.3	0.9	16.6	43.4	55.6	7.2
BS2-Spring	7.6	3.1	3.4	0.9	16.9	45.6	44.3	3.3
BS3-Spring	11.4	3.1	3.3	0.9	17.6	44.7	45.4	5.1
	Significance	NS	NS	NS	NS	NS	NS	NS
	CV (%)	5.83	5.29	7.26	12.09	7.51	15.95	47.47
	LSD (0.05)	0.03	0.03	0.01	3.05	5.00	11.24	3.64
	<b>Contrasts</b>							
	No fertilizer vs. All	-	-	-	-	-	-	-
	No fertilizer vs. AA	-	-	-	-	-	-	-
	Fall vs. Spring	-	-	-	-	-	-	-
	AA vs. BS1	-	-	-	-	-	-	-
	AA vs. BS2	-	-	-	-	-	-	-
	AA vs. BS3	-	-	-	-	-	-	-
	BS1 vs. BS2	-	-	-	-	-	-	-
	BS2 vs. BS3	-	-	-	-	-	-	-
	BS1 vs. BS3	-	-	-	-	-	-	-

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

<sup>a</sup> As, Cd, Cr, Pb, Hg, Mo, Ni, Se, B, and Na were below detection limits.

**Appendix D.** Straw nutrient concentrations. Pinkerton Farm (P-95), Sherman County, OR. 1997..

Treatment	Biosolids Rate Mg/ha	Element														
		P	K	Ca	Mg	Zn	Mn	Fe	Cu	B	Cd	Cr	Mo	Ni	Na	
		g/kg							mg/kg							
No fertilizer	0	0.23	7.4	1.3	0.55	2.5	57	80	2.2	2.6	0.53	0.40	0.47	5.2	318	
AA	0	0.28	8.7	1.5	0.66	5.4	81	107	1.6	3.4	0.63	0.66	0.84	1.1	218	
BS1-Fall	3.1	0.22	8.0	1.6	0.66	2.2	51	90	1.6	3.2	0.65	0.40	0.54	0.5	81	
BS2-Fall	5.2	0.25	8.9	1.7	0.71	2.7	58	99	3.8	3.7	0.68	0.41	0.38	0.8	275	
BS3-Fall	9.9	0.33	13.6	2.5	1.06	4.7	61	147	2.8	4.2	0.73	0.44	0.31	0.9	91	
BS1-Spring	3.1	0.22	8.9	1.6	0.68	2.4	52	91	5.6	3.1	0.65	0.40	0.37	0.9	80	
BS2-Spring	5.2	0.24	9.0	1.9	0.77	3.4	61	100	1.4	7.5	0.61	0.42	0.24	2.0	76	
BS3-Spring	9.9	0.39	13.9	2.4	1.11	5.4	62	116	3.2	4.3	0.54	0.45	1.08	0.4	147	
Significance		NS	**	**	**	NS	*	NS	NS	NS	NS	NS	NS	NS	NS	
CV (%)		31.4	16.1	13.5	17.2	55.5	13.5	26.0	101.5	56.0	22.6	36.7	105.9	184.2	108.8	
LSD (0.05)		0.02	0.28	0.04	0.02	3.49	14.27	47.13	4.89	3.92	0.25	0.29	0.98	4.73	306.38	
<b>Contrasts</b>																
No fertilizer vs. All		-	*	**	*	-	NS	-	-	-	-	-	-	-	-	
No fertilizer vs. AA		-	NS	NS	NS	-	**	-	-	-	-	-	-	-	-	
Fall vs. Spring		-	NS	NS	NS	-	NS	-	-	-	-	-	-	-	-	
AA vs. BS1		-	NS	NS	NS	-	**	-	-	-	-	-	-	-	-	
AA vs. BS2		-	NS	NS	NS	-	**	-	-	-	-	-	-	-	-	
AA vs. BS3		-	**	**	**	-	**	-	-	-	-	-	-	-	-	
BS1 vs. BS2		-	NS	NS	NS	-	NS	-	-	-	-	-	-	-	-	
BS2 vs. BS3		-	**	**	**	-	NS	-	-	-	-	-	-	-	-	
BS1 vs. BS3		-	**	**	**	-	NS	-	-	-	-	-	-	-	-	

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

**Appendix E.** Straw nutrient concentrations. McClennan Farm (M-96), Sherman County, OR. 1998.

Treatment	Biosolids Rate Mg/ha	Element									
		P	K	Ca	Mg	Zn	Mn	Fe	Cu	B	Na
		g/kg				mg/kg					
No fertilizer	0	0.50	10	1.2	0.85	2.8	74.3	65.3	1.2	9.6	42.3
AA	0	0.35	9.2	1.4	0.75	2.8	79.3	61.5	1.3	8.5	40.0
BS1-Fall	3.8	0.30	11	1.2	0.73	2.9	63.3	88.5	1.2	6.4	62.5
BS2-Fall	7.6	0.33	11	1.2	0.73	2.8	64.8	52.8	1.2	6.4	65.3
BS3-Fall	11.4	0.28	14	1.7	0.93	2.8	66.8	61.0	1.5	6.6	42.3
BS1-Spring	3.8	0.43	11	1.2	0.68	2.8	67.0	54.3	1.2	6.4	43.8
BS2-Spring	7.6	0.38	12	1.4	0.73	2.9	70.8	50.8	1.4	6.4	40.0
BS3-Spring	11.4	0.33	13	1.2	0.75	2.8	72.8	59.0	1.3	6.4	89.8
Significance		**	*	*	*	NS	NS	NS	NS	NS	NS
CV (%)		20.28	17.54	15.46	12.72	4.09	12.90	41.32	18.28	34.44	68.03
LSD (0.05)		0.01	0.29	0.03	0.01	0.17	13.25	37.45	0.34	3.59	53.24
<b>Contrasts</b>											
No fertilizer vs. All		**	NS	NS	NS	-	-	-	-	-	-
No fertilizer vs. AA		**	NS	NS	NS	-	-	-	-	-	-
Fall vs. Spring		*	NS	NS	NS	-	-	-	-	-	-
AA vs. BS1		NS	NS	NS	NS	-	-	-	-	-	-
AA vs. BS2		NS	NS	NS	NS	-	-	-	-	-	-
AA vs. BS3		NS	**	NS	NS	-	-	-	-	-	-
BS1 vs. BS2		NS	NS	NS	NS	-	-	-	-	-	-
BS2 vs. BS3		NS	NS	NS	*	-	-	-	-	-	-
BS1 vs. BS3		NS	*	**	*	-	-	-	-	-	-

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

**Appendix F.** Flag leaf tissue nutrient concentrations. Pinkerton Farm (P-95), Sherman County, OR. 1997.

Treatment	Biosolids Rate Mg/ha	Element, Flag Leaf Tissue (Flowering, Feekes 10.5-10.51)										
		P	K	Ca	Mg	Cr	Pb	Mn	Fe	Cu	B	Zn
		g/kg				mg/kg						
No fertilizer	0	1.85	7.20	4.85	2.85	0.46	0.80	86	92	5.2	1.6	11.4
AA	0	2.10	9.45	5.31	3.29	0.40	0.95	104	109	6.7	2.6	13.7
BS1-Fall	3.1	2.01	8.30	5.64	3.19	0.42	1.20	76	104	6.2	1.8	13.5
BS2-Fall	5.2	1.97	8.87	5.39	3.16	0.40	0.82	76	104	6.4	2.5	14.4
BS3-Fall	9.9	2.21	12.30	6.81	3.94	0.40	0.93	87	137	8.8	1.9	18.4
BS1-Spring	3.1	2.04	9.32	5.20	3.12	0.46	1.09	75	105	6.0	1.8	13.7
BS2-Spring	5.2	1.92	10.19	6.01	3.22	0.40	1.00	80	109	6.4	1.9	13.9
BS3-Spring	9.9	2.24	13.07	6.00	3.60	0.40	0.80	92	117	7.5	2.9	18.7
Significance		NS	**	NS	*	NS	NS	NS	*	**	NS	**
CV (%)		8.4	9.6	12.1	8.8	8.4	36.0	20.7	10.6	12.8	37.9	8.4
LSD (0.05)		0.03	0.16	0.12	0.05	0.06	0.60	30.59	20.33	1.51	1.41	2.17
<b>Contrasts</b>												
Chk vs. All		-	**	-	**	-	-	-	*	**	-	**
Chk vs. AA		-	*	-	NS	-	-	-	NS	*	-	*
Fall vs. Spring		-	*	-	NS	-	-	-	NS	NS	-	NS
AA vs. BS1		-	NS	-	NS	-	-	-	NS	NS	-	NS
AA vs. BS2		-	NS	-	NS	-	-	-	NS	NS	-	NS
AA vs. BS3		-	**	-	*	-	-	-	*	*	-	**
BS1 vs. BS2		-	NS	-	NS	-	-	-	NS	NS	-	NS
BS2 vs. BS3		-	**	-	**	-	-	-	**	**	-	**
BS1 vs. BS3		-	**	-	**	-	-	-	**	**	-	**

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.

**Appendix G.** Flag leaf tissue nutrient concentrations. McClannan Farm (M-96), Sherman County, OR. 1998.

Treatment	Biosolids Rate Mg/ha	Element, Flag Leaf Tissue (Early Flowering, Feekes 10.5)										
		P	K	Ca	Mg	Cd	Mn	Fe	B	Zn	Cu	
		g/kg				mg/kg						
No fertilizer	0	2.33	8.50	3.08	1.75	0.5	42.8	65.3	6.3	9.5	4.3	
AA	0	2.40	9.13	3.78	1.93	0.5	56.0	72.3	6.5	10.8	4.5	
BS1-Fall	3.8	2.55	10.30	4.10	2.18	0.5	56.3	72.5	6.3	12.5	5.3	
BS2-Fall	7.6	2.63	12.28	5.10	2.43	0.4	72.8	77.5	8.3	13.8	5.8	
BS3-Fall	11.4	2.65	14.28	6.18	2.70	0.4	83.0	86.5	6.8	15.5	6.3	
BS1-Spring	3.8	2.68	10.75	4.13	2.25	0.5	54.5	75.0	7.0	12.3	5.3	
BS2-Spring	7.6	2.73	13.45	5.38	2.55	0.5	81.0	80.5	7.3	14.8	5.8	
BS3-Spring	11.4	2.63	14.28	5.95	2.63	0.4	97.8	82.8	7.0	16.8	6.0	
Significance		NS	**	**	**	NS	**	**	NS	**	**	
CV (%)		7.11	6.32	9.84	8.94	11.29	14.33	5.93	12.20	9.65	10.93	
LSD (0.05)		0.03	0.11	0.07	0.03	0.08	14.33	6.67	1.24	1.88	0.86	
<b>Contrasts</b>												
Chk vs. All		-	**	**	**	-	**	**	-	**	**	
Chk vs. AA		-	NS	*	NS	-	NS	*	-	NS	NS	
Fall vs. Spring		-	NS	NS	NS	-	NS	NS	-	NS	NS	
AA vs. BS1		-	**	NS	*	-	NS	NS	-	*	*	
AA vs. BS2		-	**	**	**	-	**	*	-	**	**	
AA vs. BS3		-	**	**	**	-	**	**	-	**	**	
BS1 vs. BS2		-	**	**	*	-	**	*	-	**	NS	
BS2 vs. BS3		-	**	**	NS	-	*	*	-	**	NS	
BS1 vs. BS3		-	**	**	**	-	**	**	-	**	**	

\*, \*\* Significant at the 0.05 and 0.01 probability levels, respectively.