Influence of the Rate of Nitrogen Fertilization on the Mineral Content of Winter Wheat in Ontario†

Bernie J. Zebarth,*,‡ C. James Warren,§ and Robert W. Sheard‖

Agriculture Canada Agassiz Research Station, P.O. Box 1000, Agassiz, British Columbia V0M 1A0, Canada, Department of Earth Sciences, University of Waterloo, Waterloo, Ontario N2L 3G1, Canada, and Department of Land Resource Science, University of Guelph, Guelph, Ontario N1G 2W1, Canada

The influence of the rate of N fertilization on the mineral content of winter wheat grown in Ontario, Canada, was studied. Treatments were main plots of wheat type, either soft white winter or hard red winter, and subplots of 40, 80, 120, 160, 200, or 240 kg of N ha^{-1}. Grain yield, yield components, nitrogen concentration, and grain ash concentration were determined for each subplot, and the concentrations of K, P, Ca, Mg, Fe, Zn, and Mn in the whole grain were determined for the 40 and 160 kg of N ha^{-1} treatments. Total ash concentration decreased with increasing N rate in a curvilinear fashion for both wheats and was negatively correlated with grain yield. Increasing the N application rate from 40 to 160 kg of N ha^{-1} resulted in small, <10%, decreases in the concentration of P and K but increased the concentrations of Ca, Mg, and Fe. Variation in grain mineral concentration as a result of N management was minor relative to variation in grain mineral concentration between local environments.

INTRODUCTION

There is an increasing trend in Ontario toward wheat production under intensive management systems. Whereas numerous studies have reported on the mineral concentration of wheat as influenced by geographical location, environment under which the crop was grown [i.e., Pomeranz and Dikeman (1983)], and wheat type or cultivar [i.e., Peterson et al. (1983)], few studies have reported on the influence of crop management practices on the ash or mineral concentration of wheat.

Wheat yield is more responsive to N fertilization when wheat is grown under more intensive management systems (Becker and Aufhammer, 1982). As a result, the influence of the use of high rates of N fertilization on the mineral concentration of wheat and the implications in terms of the nutrient content of wheat for human consumption are poorly understood. No consistent response of ash or mineral concentration of wheat to the rate of N fertilization has been obtained in previous studies (Ghanbari and Mameesh, 1971; Agarwal, 1976; Syltie and Dahnke, 1983).

The purpose of the present study was to determine the influence of the rate of N fertilization on the ash concentration in the grain of soft white wheat and hard red winter wheat, grown under an intensive cereal management system in Ontario, Canada.

MATERIALS AND METHODS

Field Methods. Two field trials, identified here as A and B, were conducted in 1988, and two field trials, identified as D and F, were conducted in 1989. Each trial used a split-plot arrangement of treatments replicated four times in a randomized complete block design. Main plots were wheat type, either soft white winter (SWW) wheat, cultivar Harus, or hard red winter (HRW) wheat, cultivar Monopol in 1988 and cultivar Absolvent in 1989. The 2 m × 6 m subplots received one of six rates of N fertilization: 40, 80, 120, 160, 200, or 240 kg of N ha^{-1}. The N was applied as a surface broadcast of ammonium nitrate, 75% at Zadok's growth stage (ZGS) 21–23 in mid-April and 25% at ZGS 32 in late May, using a precision fertilizer spreader. Grain yield and yield components, including number of grains per square meter and 1000-grain weight, were determined for each experimental unit.

Laboratory Methods. Subsamples of the grain were ground to pass a 1-mm screen in a UDY cyclone mill and oven-dried at 80 °C for the determination of grain ash concentration and Kjeldahl nitrogen for all treatments and the determination of grain concentrations of K, P, Ca, Mg, Fe, Mn, and Zn for the 40 and 160 kg of N ha^{-1} treatments.

Grain ash concentration was determined from the weight loss of a 3-g sample during combustion for 1 h at 220 °C, followed by 16 h at 570 °C.

A 0.20-g subsample of the grain was digested using a concentrated sulfuric acid–salicylic acid mixture with a K_2SO_4–Se catalyst and clarified with hydrogen peroxide during digestion at 370 °C (Thomas et al., 1967). Total nitrogen concentration in the digest was determined as NH_4^+ on an AutoAnalyser system.

Selected samples of the ground plant material were digested to determine elemental concentration using a mixture of three mineral acids in a microwave (2450 MHz) field. Between 0.25 and 0.30 g of oven-dried (60 °C) sample was weighed directly into 60-mL Teflon PFA containers. Five milliliters of concentrated HNO_3 was added to each container. The containers were then loosely covered and allowed to stand in a fume hood overnight. Containers then received 1 mL of concentrated HF and 2 mL of concentrated HCl and were swirled and digested, 24 at a time, in a CEM microwave oven (CEM Corp., Matthews, NC). Samples were treated at 650 W for 180 s, at 275 W for 360 s, and finally at 125 W for 240 s. The digested samples were cooled to room temperature, made up to a final volume of 20 mL with distilled water, and stored at 4 °C in sealed plastic containers until analysis. Digests were analyzed for total content of P, Mg, Ca, Mn, Zn, and Fe by inductively coupled plasma atomic emission spectrophotometry and for total content of K by atomic absorption spectrophotometry. The accuracy of the analytical technique was verified using NBS SRM 1575, pine needles, standard plant material. The accuracy of the methods used was good except for Fe, where the mean measured value (273 mg kg^{-1}) was higher than the NBS consensus value (185 ± 26 mg kg^{-1}).

The proportion of the total ash content of the grain made up by the mineral elements was calculated by converting from the elemental concentration to the concentration of the corresponding oxide (K_2O, P_2O_5, MgO, CaO, ZnO, F_2O_3, and Mn_2O_3) and dividing by the total ash content.

* Author to whom correspondence should be addressed.
† Contribution No. 436.
‡ Research scientist, Agriculture Canada.
§ Research associate, University of Waterloo.
‖ Professor, University of Guelph.

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Table I. Response of Grain Yield and Grain Ash Concentration of HRW and SWW Wheat to Rate of N Fertilization at Four Sites

wheat type	site	N rate, kg ha^{-1} 40	80	120	160	200	240	significance[c]
		Grain Yield, tonne ha^{-1} at 14.5% Moisture						
SWW	A	4.68	6.32	6.70	6.38	6.08	6.27	L[b] Q[b]
	B	4.23	4.59	5.60	4.82	5.82	5.36	L[b]
	D	4.93	5.64	5.71	5.72	5.73	5.87	L[b] Q[b]
	F	4.03	5.48	5.47	5.55	5.41	5.19	L[b] Q[b]
	mean	4.47	5.50	5.87	5.62	5.76	5.68	L[b] Q[b]
HRW	A	4.37	5.01	4.93	4.72	5.23	4.52	Q[b]
	B	3.23	3.64	4.17	4.34	4.39	4.38	L[b] Q[a]
	D	3.96	4.89	4.41	4.68	4.84	4.82	L[a] Q[a]
	F	4.09	4.84	5.13	4.65	4.40	4.32	Q[b]
	mean	3.91	4.59	4.66	4.59	4.72	4.52	L[b] Q[b]
		Ash Content, %						
SWW	A	1.72	1.66	1.58	1.54	1.55	1.52	L[b] Q[b]
	B	1.57	1.53	1.42	1.41	1.39	1.42	L[b] Q[b]
	D	1.90	1.89	1.87	1.86	1.84	1.82	L[a]
	F	1.91	1.85	1.85	1.81	1.82	1.84	L[b] Q[b]
	mean	1.78	1.72	1.67	1.66	1.65	1.64	L[b] Q[b]
HRW	A	1.79	1.74	1.74	1.73	1.72	1.75	L[a] Q[b]
	B	1.65	1.62	1.60	1.59	1.56	1.53	L[b]
	D	1.87	1.87	1.84	1.85	1.81	1.84	L[a]
	F	1.94	1.93	1.93	1.89	1.94	1.99	NS
	mean	1.81	1.79	1.77	1.76	1.76	1.77	L[b]

[a] $P < 0.05$. [b] $P < 0.01$. [c] L, linear; Q, quadratic; NS, nonsignificant.

Statistical analyses were performed using the general linear models procedure of SAS (SAS Institute Inc., 1990).

RESULTS

Grain yield increased with increased N fertilization for each cultivar in each experiment in either a linear or curvilinear fashion (Table I). On average, grain yield was increased 26 and 17% for SWW and HRW, respectively, when the N rate was increased from 40 to 160 kg of N ha^{-1}.

Except for the HRW wheat at site F, grain ash concentration, an indicator of grain mineral concentration, decreased with increasing N rate in either a linear or curvilinear fashion (Table I). Although the influence of N application rate on grain ash concentration was significant in seven of eight cases, the decrease in ash concentration with changing N rate was relatively minor, never exceeding 0.20% ash. Total mineral uptake, calculated as grain yield multiplied by grain ash content, was increased from 79.6 to 86.5 kg ha^{-1} for SWW wheat and from 70.8 to 80.8 kg ha^{-1} for HRW wheat when the N application rate was increased from 40 to 160 kg of N ha^{-1}. In general, the grain ash concentration varied more among sites, which reflects different environments, than among N application rates.

Grain ash concentation was negatively correlated with grain yield for three of four sites for each cultivar (Table II). Similarly, grain ash concentration was negatively correlated with grain per square meter and test weight for three of four sites for the SWW wheat but was not significantly correlated with grain per square meter and test weight at any sites for the HRW wheat. Although some significant correlations were observed between grain ash concentration and 1000-grain weight, there was no consistent pattern for those correlations.

A detailed analysis of the elemental concentration of the two wheats was performed at high and low rates of N fertilization to determine the influence of N application rate on the concentration of seven mineral elements in the grain and to determine which elements are responsible for the change in ash content with changing N rate. The 40 and 160 kg of N ha^{-1} treatments were chosen for the detailed chemical analyses because they encompass the rates over which N displayed its major influence on grain ash concentration and they represent the range of N application rates likely to be used for intensive cereal production in Ontario.

Table II. Correlation Coefficients between Grain Ash Concentration and Selected Wheat Parameters

wheat type	site	wheat parameters: grain yield	grain m^{-2}	1000-grain wt	test wt	N concn
SWW	A	NS[d]	NS	NS	−0.76[b]	−0.87[b]
	B	−0.62[b]	−0.54[b]	NS	−0.68[b]	−0.49[a]
	D	−0.38[c]	−0.50[a]	NS	NS	−0.68[b]
	F	−0.66[b]	−0.50[a]	0.40[c]	−0.43[a]	−0.64[b]
HRW	A	−0.57[b]	NS	0.42[a]	NS	NS
	B	−0.36[c]	NS	0.39[c]	NS	−0.40[c]
	D	NS	NS	−0.36[c]	NS	−0.41[c]
	F	−0.38[c]	NS	NS	NS	NS

[a] $P < 0.05$. [b] $P < 0.01$. [c] $P < 0.10$. [d] NS, nonsignificant correlation.

Nitrogen application rate had a significant ($P < 0.05$) influence on the concentration of five of the seven mineral elements in the wheat grain (Table III). Increased N application decreased the average K concentration by 7% and the average P concentration by 3%, increased the average Mg concentration by 2%, the average Ca concentration by 20%, and the average Fe concentration by 21%, but had no significant effect on Zn and Mn concentrations. An interaction between wheat type and N application rate was observed only for Ca: the increase in Ca concentration with increased N application was higher for the SWW than for the HRW wheat.

The proportion of the total ash content composed of the oxides of K and P was not significantly influenced by N rate (Table IV). A small but statistically significant increase in the proportion of the total ash content composed of magnesium, calcium, and iron oxides was determined at the higher N application rate.

DISCUSSION

Although small moisture deficits occurred due to dry climatic conditions in each year, the moisture deficits occurred late enough in the season to have minimal impact on crop growth and development. Neither lodging nor disease appeared to have a significant effect on crop growth and development. Therefore, given the large response in grain yield and grain N concentration to the increase in N fertilization and given that the applications of P and K were in excess of the crop requirement for the nutrients as indicated by soil testing, the growth of the crop can be assumed to be primarily limited by the availability of N.

While the total quantity of minerals in the grain increased with increased N application, the increase was less than the increase in grain dry matter, resulting in a negative correlation between grain yield and grain ash content. The increase in grain yield with increasing N application was primarily as a result of increased tillering for the SWW wheat and as a result of increased grains per head for the HRW wheat (unpublished data). It is likely, therefore, that the significant correlations observed between grain ash concentration and grain per square meter for the SWW wheat are expressions of the correlation between grain ash concentration and grain yield. No such relationships were observed, however, for the HRW wheat.

It is plausible, therefore, that the decreased grain ash concentration at increased N application rate is the result of a simple dilution effect: the increase in the amount of the nutrient taken up was less than the increase in grain

Table III. Mineral and Nitrogen Concentration of HRW and SWW Wheat As Influenced by the Rate of N Fertilization (on Dry Weight Basis, Averaged across Four Sites)

				mineral concn						
wheat type	N rate, kg ha^{-1}	N concn, %	ash concn, %	K, %	P, %	Mg, %	Ca, μg g^{-1}	Zn, μg g^{-1}	Fe, μg g^{-1}	Mn, μg g^{-1}
SWW	40	1.26	1.78	0.437	0.419	0.126	330	20.1	40.0	19.4
	160	1.74	1.66	0.407	0.399	0.129	432	23.1	50.3	20.7
HRW	40	1.49	1.81	0.435	0.432	0.134	343	25.2	54.4	22.7
	160	2.06	1.76	0.407	0.423	0.137	375	29.1	63.3	18.1

Statistical Significance (Values Are Mean Squares)

	N concn	ash concn (×10^{-3})	K (×10^{-3})	P (×10^{-3})	Mg (×10^{-3})	Ca	Zn	Fe	Mn
wheat type (A), 1 df	1.233[b]	82.4[b]	0.01	5.44[a]	1.069[b]	7744	484[a]	2998[b]	2
error A, 21 df	0.010	3.0	1.16	1.09	0.044	3510	81	109	56
N rate (B), 1 df	4.237[b]	109.1[b]	13.63[b]	3.45[a]	0.179[a]	71690[b]	189	1478[b]	46
A × B, 1 df	0.031[a]	17.6[b]	0.01	0.45	0.001	19670[b]	4	7	137
error B, 24 df	0.005	1.8	0.73	0.47	0.028	1081	144	87	52

[a] $P < 0.05$. [b] $P < 0.01$.

Table IV. Concentration and Statistical Significance of the Oxides of K, P, Mg, Ca, Zn, Fe, and Mn Expressed as a Percentage of the Total Ash Content of the Grain, As Influenced by N Rate for Two Wheats (Averaged across Four Sites)

		% of total ash						
wheat type	N rate, kg ha^{-1}	K_2O	P_2O_5	MgO	CaO	ZnO	Fe_2O_3	Mn_2O_3
SWW	40	29.8	54.1	11.8	2.6	0.14	0.33	0.16
	160	29.9	55.0	12.9	3.7	0.17	0.44	0.18
HRW	40	29.0	54.6	12.2	2.7	0.17	0.43	0.17
	160	28.3	54.8	12.9	3.0	0.21	0.52	0.15

Statistical Significance (Values Are Mean Squares)

	K_2O	P_2O_5	MgO	CaO	ZnO (×10^{-3})	Fe_2O_3 (×10^{-3})	Mn_2O_3 (×10^{-3})
wheat type (A), 1 df	25.57[a]	0.53	0.57	2.08[a]	15.6[a]	117.1[b]	1.1
error A, 21 df	4.48	20.73	0.62	0.26	3.9	6.3	3.6
N rate (B), 1 df	3.35	4.04	12.72[b]	7.57[b]	19.9	173.2[b]	0.2
A × B, 1 df	6.29	1.72	1.11	2.26[b]	1.8	1.8	10.6
error B, 23 df	4.23	10.27	0.39	0.09	8.9	5.2	3.4

[a] $P < 0.05$. [b] $P < 0.01$.

dry matter, resulting in a lower concentration. If this were true, one would anticipate that the concentration of most mineral elements either would be unaffected by N rate or would be decreased slightly at higher N rates. The fact that the concentrations of P, K, Zn, and Mn were all similar or lower at the high N rate than at the low N rate and the proportion of the total ash composed of the oxides of P, K, Zn, and Mn was independent of N rate would tend to support this idea.

In contrast, the increased concentrations of Ca, Mg, and Fe at the higher N rate are inconsistent with a simple dilution effect. Other studies on wheat have also reported increased N application to result in increased concentrations of Ca and Fe (Agarwal, 1976; Syltie and Dahnke, 1983).

The increased proportion of the total ash composed of the oxides of Ca, Mg, and Fe at higher N rates suggests some mechanism of enhanced nutrient uptake or enhanced nutrient availability. The mechanism responsible for the increased concentrations of Ca, Mg, and Fe at high N rates, however, is not known.

In terms of the overall nutrient content of the grain, the increased N rate resulted in small, <10%, decreases in the concentration of total ash and of P and K and in significant increases in Fe and Ca concentrations of the SWW wheat. Thus, no adverse effect of increased N fertilization on the mineral content of the grain was observed.

In addition, any variation in the mineral concentration of grain as a result of N management is likely minor relative to the variation in grain ash concentration resulting from grain production on different sites. It should be noted, however, that the decrease in the concentration of P and K at high N rates may have been larger if the amount of P and K applied was reduced.

LITERATURE CITED

Agarwal, M. M. Effect of fertilizer treatments on the grain quality of wheat. *Indian J. Agric. Res.* **1976**, *10*, 185–188.

Becker, F. A.; Aufhammer, W. Nitrogen fertilisation and methods of predicting the N requirements of winter wheat in the Federal Republic of Germany. In *Symposium on Fertilisers and Intensive Wheat Production in the EEC*; Fertiliser Society of London: London, 1982; pp 33–66.

Ghanbari, H. A.; Mameesh, M. S. Iron, zinc, manganese and copper content of semidwarf wheat varieties grown under different agronomic conditions. *Cereal Chem.* **1971**, *48*, 411–415.

Peterson, C. J.; Johnson, V. A.; Mattern, P. J. Evaluation of variation in mineral element concentrations in wheat flour and bran of different cultivars. *Cereal Chem.* **1983**, *60*, 450–455.

Pomeranz, Y.; Dikeman, E. Minerals and protein contents in hard red winter wheat flours. *Cereal Chem.* **1983**, *60*, 80–82.

SAS Institute Inc. *SAS/STAT User's Guide*, version 6, 4th ed.; SAS Institute: Cary, NC, 1990; Vol. 2.

Syltie, P. W.; Dahnke, W. C. Mineral and protein content, test weight, and yield variations of hard red spring wheat grain as influenced by fertilization and cultivar. *Qual. Plant. Plant Foods Hum. Nutr.* **1983**, *32*, 37–49.

Thomas, R. L.; Sheard, R. W.; Moyer, J. R. Comparison of conventional and automated procedures for nitrogen, phosphorous and potassium analysis of plant material using a single digestion. *Agron. J.* **1967**, *59*, 240–243.

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