



Long-term K fertilization effects on soil available K, grain yield, and plant K critical value in winter wheat

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Abstract This study takes advantage of Swiss long-term field experiments (> 30 yrs) with different K fertilization rates at three sites to (i) test the possibility to generalize linear relationships between K extracts (ammonium acetate, K-AA; ammonium acetate EDTA, K-AAE; water, K-H₂O; and water saturated with CO₂, K-CO₂), (ii) determine the K fertilization effect on soil exchangeable K, (iii) determine the K fertilization effect on shoot biomass and grain yield of winter wheat, (iv) analyze the possibility to derive a soil K critical value, and (v) determine a critical plant K (K_c) dilution curve as a function of shoot biomass (SB) using a “classical” and a Bayesian

method. Shoot biomass during the growing season, grain yield, and four soil extracts were measured in 2018 after more than 30 years with four to five rates of K fertilization. Unpublished data of soil K-AAE concentrations, and grain yield and K concentrations since the start of the experiments were also used to analyze the relationship between soil K-AAE and the cumulative K budget. The K-AA and K-AAE concentrations can be converted from one to the other [$K-AAE = 26.8 + (1.11 \times K-AA)$], while the relationship between K-H₂O and K-CO₂ depends on soil pH. The K-AAE concentrations were positively related to the cumulative K budget for K-AAE ranges from a minimal K concentration up to a K holding capacity that were specific to each site. The lack of K fertilization during several decades decreased shoot biomass in 2018 and grain yield over the course of the experiments at only one of the three sites. The K-AAE values corresponding to non-limiting soil K conditions at this site (50–75 mg K kg⁻¹) were close to the critical values previously reported but the large range suggests that more soil parameters should be taken into account to improve the accuracy of the fertilization guidelines. The Bayesian and “classical” methods used for estimating the K_c curve yielded similar results ($K_c = 58.21 \times SB^{-0.45}$) that should be confirmed in future studies under a range of pedoclimatic conditions along with the effect of other nutrients and wheat cultivars.

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Introduction

Of all essential mineral nutrients, potassium (K) is needed in second highest amounts by most agricultural crops and it is used for numerous physiological mechanisms (Prajapati and Modi 2012; Rawat et al. 2016). The ability of soils to provide enough K to meet crop demand is crucial to avoid yield losses. Soil total K content varies from 3 to 100 Mg K ha⁻¹ but only a fraction of total K, up to 2%, is readily available for crops (Schroeder 1978). Certain soils can satisfy crop K requirements whereas fertilization in other soils is a prerequisite to avoid K limitation (Swarup and Ghosh 1979; Swarup and Chhillar 1986; Edmeades et al. 2010). Investigating the long-term effect of contrasted rates of applied K on different soil types is crucial to identify the soil K reserves and soil conditions susceptible to K deficiency in crops.

Based on various chemical extractant strengths, soil K is traditionally divided into four pools that are in semi-equilibrium with each other (Blake et al. 1999; Madaras and Lipavský 2009). Total K (K-Tot), mainly composed of structural K, can be estimated using very strong extractants such as hydrofluoric acid (HF) or perchloric acid (HClO₄) (Potts 2012). Generally, structural K is not available for crops except for a tiny fraction due to mineral dissolution (Moody and Bell 2006). Non-exchangeable plus exchangeable K are commonly targeted with weaker extractants such as tetraphenylboron or nitric acid (Jalali 2006; Carey et al. 2011). Non-exchangeable K is partly fixed between clay sheets (Matthews and Beckett 1962; Moody and Bell 2006). The contribution of non-exchangeable K to crop nutrition increases as exchangeable K decreases and can be important especially for soils with high contents of 2:1 layered clay minerals (Barré et al. 2008; Damar et al. 2020). The availability of non-exchangeable K for crops is also influenced by the crystallographic properties of soil mineral particles (Bell et al. 2021). To measure only exchangeable K, numerous extractants are used, e.g., ammonium acetate (K-AA), ammonium acetate EDTA (K-AAE) in Switzerland, BaCl₂ – triethanolamine, NH₄Cl, or NaHCO₃ (NFX 31-108, ISO 13536) (Colwell 1965; Mc Lean and Watson 1985; FAL

2004). It consists in large part of adsorbed K on the surface of soil particles and it is classically considered as the main reserve of available K since it can represent more than 90% of the crop K uptake (Lalitha and Dhakshinamoorthy 2014). Exchangeable K can vary from a minimal value (Tabatabai and Hanway 1969; Rao and Rao 2000; Schneider et al. 2016) to a K holding capacity (Goulding et al. 2021), which are both specific to soil properties. Potassium in the soil solution is immediately available for crops (Schneider 2003) and can be analyzed with a K water extract (K-H₂O) or with K water acidified with CO₂ extract (K-CO₂) to simulate the effect of the soil solution, the latter being specific to Switzerland (FAL 2004; Stünzi 2007). The possibility to convert values obtained by different K extracts has repeatedly been studied (Qian et al. 1994; Bedi et al. 2002; Brennan and Bell 2013; Zhang et al. 2017), especially those estimating exchangeable K (Johnston and Goulding 1990; Moody and Bell 2006). However, considering the multitude of existing methods, there is still a lack of information on the correspondence of some of the methods, for example K-AAE or K-CO₂.

Bell et al. (2021) highlighted the weaknesses of this model with four pools. For instance, crop availability of K in the interlayer of various clay mineral types cannot be estimated through only one chemical extraction. In addition, structural K can also provide substantial amounts of available K in some cases (Sadusky et al. 1987). Therefore, Bell et al. (2021) proposed another model to classify the K pools based on soil physico-chemical mechanisms involved in holding K rather than on sequential chemical extraction methods. For instance, a “surface-adsorbed” pool is considered instead of the exchangeable K and K pools are classified according to their crystallographic properties. However, the chemical extraction of soil K and its interpretation are still in use since it remains the simplest and cheapest means to acquire a rough estimate of the soil K status, although the agronomic relevancy may be questionable.

The K withdrawn by roots from the soil solution is replenished at a rate regulated by the soil K buffer power, that is, “the soil capacity to resist a change in soil solution K concentration following removal or input of K to the soil–plant system” (Schneider et al. 2013). The soil K buffer power can be quantified using the quantity-to-intensity ratio (Beckett 1964a; Beckett et al. 1966). The quantity can be

approximated by the exchangeable K (e.g. K-AA; Beckett (1964b)). The intensity can be roughly approached using K-H₂O (Silberbush and Barber 1983). A more accurate calculation of the intensity ($aK/\sqrt{aCa+aMg}$) requires the measurement of the activity of K, Ca, and Mg in the soil solution (Beckett 1964b). Since the relationship between intensity and quantity is curvilinear, estimation of the K buffer power can be improved through sorption–desorption experiments that are time consuming (Schneider 1997).

Fertilization guidelines, considering indirectly the K buffer power and the non-exchangeable K, provide interpretation schemes of exchangeable soil K or K readily available taking soil texture into account (Mahler and Guy 2001; Gerwing and Gelderman 2005; Brennan and Bell 2013; Flisch et al. 2017; Denoroy et al. 2019). Usually, fertilization guidelines provide soil K critical values, i.e., values of a soil K extract indicating the limit between a soil K status considered deficient that requires fertilization and a soil K status considered sufficient that does not require K fertilization. Exchangeable K is frequently used to establish fertilization recommendations (He et al. 2012; Flisch et al. 2017; Goulding et al. 2021). However, the recommendations for K fertilization are poorly generalizable for different soil types. The relevance of using exchangeable K as indicator of the soil K status has to be investigated.

The most accurate method to quantify the crop nutrient status remains the analysis of the nutrient concentration in the crop biomass. The identification of a plant K critical value for a crop species is not simple since the K concentration decreases during plant growth (Baker and Tucker 1973). The difficulty can be overcome by using the critical dilution curve method, which was first developed to identify the critical N concentration of grasslands (Lemaire et al. 1984). The critical shoot concentration of a nutrient is modeled as a power function of the shoot biomass accumulation during plant growth. This gives a specific critical value for a given shoot biomass. For N (Greenwood et al. 1990; Justes et al. 1994; Colnenne et al. 1998; Debaeke et al. 2012) and P (Bélanger et al. 2015; Cadot et al. 2018; Soratto et al. 2020; Fontana et al. 2021), studies were carried out to develop critical dilution curves for many crop species. For K, critical dilution curves have so far only been investigated for vegetables and grasslands (Salette

and Huché 1991; Greenwood and Stone 1998; Gómez et al. 2019) whereas those for cereal or oilseed crops have never been investigated to our knowledge. While shoot K dynamics in wheat have repeatedly been studied in the context of ontogeny in pot experiments (Rao 1986; Brennan and Bolland 2007; Ma et al. 2013), they have, to our knowledge, never been related to biomass production. Hence, a critical K dilution (K_c) curve for wheat is still missing.

A critical dilution curve is usually modeled using data from an experimental design that includes a range of fertilization rates. Data from the lowest fertilization rate resulting in maximal shoot biomass production is used to fit the critical curve (Greenwood et al. 1990; Justes et al. 1994; Bélanger et al. 2015; Cadot et al. 2018). Classically, the modeling procedures have not considered the uncertainty of the threshold separating the limiting and the non-limiting nutritional conditions (Greenwood et al. 1990; Justes et al. 1994). Recently, a Bayesian method was developed to fit a critical curve directly from measurements of biomass and nutrient concentrations (Makowski et al. 2020). Data classification of nutrient limited vs non-limited is not required and the uncertainty is analyzed in a rigorous manner. A comparison between these two methods ("classical" vs. Bayesian) would allow us to evaluate the robustness of the parameters of the critical dilution curve to the statistical method.

This study takes advantage of long-term experiments established at three sites in Switzerland with contrasting pedo-climatic conditions to: (1) test the possibility to generalize linear relationships between results obtained by different extraction methods (K-AA, K-AAE, K-H₂O; and K-CO₂), (2) determine the K fertilization effect on soil exchangeable K, (3) determine the K fertilization effect on shoot biomass during the growing season and grain yield of winter wheat, (4) analyze the possibility to derive a soil K critical value and its stability across sites and years, and (5) determine a plant K_c curve using a "classical" and a Bayesian method.

Materials and methods

Field experiments

Long-term field experiments, established in Switzerland at Changins in 1971 and at Ellighausen and

Oensingen in 1989, were used to investigate the response of crop growth and yield, K uptake, and soil K availability to various rates of K fertilization over several decades (Table S1). The three sites differed in their soil characteristics. The Changins site had the highest clay content, and the highest concentrations of organic C (C-Org), total N (N-Tot), and total K (K-Tot) (Tables 1 and S1). The Ellighausen site had the lowest pH and the lowest K-Tot concentration, while the Oensingen site had the highest pH.

Potassium was applied as potassium sulfate (41.5% K) at Ellighausen and Oensingen, and as potassium chloride (49.8% K) at Changins. At Ellighausen and Oensingen, the four K fertilization treatments ranged between 0 and 5/3 of the theoretical crop K uptake with four levels: 0 K, 2/3 K, 3/3 K, and 5/3 K. All other nutrients were applied in adequate amounts according to the official Swiss fertilization guidelines (Sinaj et al. 2017). At Changins, the four K fertilization treatments were: (1) 0 K: no K and the amount of

the theoretical crop P uptake; (2) 3/3 K: the amounts of the theoretical crop K and P uptake; (3) K+: 166 kg K ha⁻¹ in addition to the theoretical crop K uptake and around the double of the crop P uptake; and (4) 0 K–0 P: no K and P. The 0 K treatment has only been implemented since 1985, while K was applied from the beginning of the experiment from 1971 until 1984. Only the 0 K and 3/3 K treatments were common to all three long-term experiments. The three long-term field experiments included four replicates of each K fertilization treatment in a randomized complete block design. The theoretical wheat K uptake was set at 66 kg K ha⁻¹ according to the recommendations of the official Swiss fertilization guidelines (Sinaj et al. 2017). Winter wheat was included in rotation with other crop species since the beginning of the long-term field experiments (Cadot et al. 2018; Hirte et al. 2021).

In October 2017, the winter wheat cultivar *cv Nara* was sown at Ellighausen and Oensingen and *cv Arina*

Table 1 Soil concentrations of total K extracted with hydrofluoric acid (K-Tot), exchangeable K extracted with acetate ammonium EDTA (K-AAE) or acetate ammonium (K-AA), and readily available K extracted with water acidified with CO₂ (K-CO₂) or water (K-H₂O) in the topsoil (0–0.2 m) and subsoil (0.2–0.5 m) in July 2018 following decades of different K fertilization treatments at three sites in Switzerland

Significant differences ($p < 0.05$) among treatments within sites are indicated by lowercase letters and among sites for the same treatments by uppercase letters. *0 P–0 K = no P and K fertilization; 0 K = no K fertilization; 2/3 K = 2/3 of the theoretical crop uptake and so on; K+ treatment was fertilized with 166 kg K ha⁻¹ in addition to the theoretical crop K uptake

Depth m	Site	Treatment*	K-Tot g kg ⁻¹	K-AAE mg kg ⁻¹	K-AA mg kg ⁻¹	K-CO ₂ mg kg ⁻¹	K-H ₂ O mg kg ⁻¹
0–0.2	Changins	0 P–0 K	19.2 b	172 b	128 b	4.7 b	8.7 b
		0 K	19.8 ab A	165 b A	127 b A	6.3 b	9.7 b A
		3/3 K	19.7 ab A	200 b A	155 b A	5.9 b B	9.1 b B
		K+	21.1 a A	551 a A	467 a A	26.3 a A	40.1 a A
	Ellighausen	0 K	10.4 C	76 c B	42 b B	3.3	5.5 b B
		2/3 K	10.5	96 b	57 b	4.4	7.1 b
		3/3 K	10.9 B	109 ab B	70 b B	6.0 B	10.0 ab A
		5/3 K	11.0 C	132 a C	110 a C	9.8 B	15.0 a B
	Oensingen	0 K	17.6 B	150 c A	106 c A	7.3 c	8.6 c AB
		2/3 K	18.2	163 c	121 c	9.5 bc	10.6 bc
		3/3 K	18.7 A	189 b A	144 b A	11.5 b A	12.6 b A
		5/3 K	18.9 B	237 a B	201 a B	17.7 a AB	18.9 a B
0.2–0.5	Changins	0 P–0 K	18.5	108	70 b	4.6	4.1
		0 K	19.8 A	128 A	76 ab A	3.6 A	2.0 A
		3/3 K	17.8 A	109 B	73 b B	4.4	1.7
		K+	18.9 A	152 A	102 a B	4.9	4.6 B
	Ellighausen	0 K	10.2 C	57 b B	37 b B	0.6 B	1.2 B
		2/3 K	10.8	62 ab	41 b	0.8	1.4
		3/3 K	11.1 C	74 ab C	53 ab C	2.5	3.6
		5/3 K	11.2 C	88 a C	67 a C	3.2	4.0 B
	Oensingen	0 K	13.4 B	106 b A	82 c A	3.4 b A	5.8 b A
		2/3 K	15.0	112 b	91 bc	4.4 ab	7.3 ab
		3/3 K	15.0 B	135 ab A	111 ab A	6.0 ab	9.3 ab
		5/3 K	16.7 B	152 a A	126 a A	7.2 a	11.5 a A

at Changins. According to the official Swiss fertilization guidelines, a total of 140 kg N ha⁻¹ of a mineral N fertilizer was split-applied on three occasions during the growing season (Sinaj et al. 2017). Soil management and plant protection were conducted according to the Swiss certification scheme Proof of Ecological Performance [Swiss Federal Council 2013; further details in Cadot et al. (2018), Hirte et al. (2021), and Fontana et al. (2021)].

Soil sampling and analyses

Following the wheat grain harvest in July 2018, five soil cores were sampled at depths of 0–0.2 m and 0.2–0.5 m. To avoid border effects and the contamination due to other K fertilization treatments in contiguous plots, soil samples were collected between 0.5 and 1.0 m from the plot delimitation and plots were separated by a 1-m wide buffer path. Soil moisture was particularly low at the time of sampling due to ongoing high temperature and low precipitation in the previous weeks. Plant residues were removed from the soil cores, which were mixed to prepare one composite sample per experimental unit. These composite samples were then air-dried and sieved at a 2-mm mesh size. For these samples, C-Org was determined by sulfochromic oxidation (NF ISO 14235) and N-Tot was measured using an elemental analyzer (Thermo, flash 2000, NF ISO 13878). Soil texture and pH were measured in line with the Swiss reference methods for soil analyses (FAL 2004). Total P (P-Tot) and K-Tot were measured after extraction of 0.25 g of soil with 5 ml of HF (40%) and 1.5 ml of HClO₄ (65%) using flame photometry and the molybdate colorimetric method, respectively (Murphy and Riley 1962) (NFX 31–147). Soil exchangeable K was estimated by two chemical extractions using either (i) ammonium acetate (K-AA) (NFX 31–108) or (ii) ammonium acetate and EDTA as a complexing agent at an acidic pH (K-AAE). Soil readily available K was also estimated by two methods using either (i) nanopure water at a ratio of 1:10 for 16 h (K-H₂O) or (ii) CO₂-saturated nanopure water at pH 3.5–4 and pCO₂ of 6 bars at a ratio of 1:2.5 for 1 h (K-CO₂). The Swiss reference methods for the determination of soil available K are K-AAE, K-H₂O, and K-CO₂ (FAL 2004).

In addition, soil samples were also collected since the beginning of the experiment at a depth of 0–0.2 m. Three to five soil cores per experimental

unit were sampled and prepared as described above. Only K-AAE was measured and the values were used to explore the relationship between annual values of the K-AAE concentration and the cumulative K budget.

K budget calculation

The annual K budget was calculated as the difference between K applied as fertilizer and annual K export (Tables S3–S7). The cumulative K budget was then calculated by summing up the annual K budgets. The annual K export was calculated by multiplying the grain yield by its K concentration for Changins. For Ellighausen and Oensingen, the straw biomass multiplied by its K concentration was also included in the calculation of the annual K export. Straw was exported at Ellighausen and Oensingen, resulting in more negative cumulative K budgets than at Changins, where straw was left on the field. The cumulative K budget included all crops in the rotation with winter wheat (Tables S3–S7). The annual K budget for the other crops was also calculated by taking into account the exported biomass and its K concentration.

Plant sampling and analyses

Shoot biomass of winter wheat was sampled on eight dates at Changins, Oensingen, and Ellighausen between the end of March and the end of May 2018. Shoot biomass was harvested using pruning shears at 20–30 mm above ground level in each experimental unit on a square area of 0.2 m² at Oensingen and Ellighausen, and of 0.5 m² at Changins. The developmental stages (CD) were then determined according to Meier (2018). The number of rows included within a square area varied sometimes due to the small-scale variability in topography. Therefore, the shoot biomass was calculated on a linear meter basis (i.e. g of DM per row meter). In July, grain yield of each experimental unit was harvested on an area of 29.5 m² at Changins, and of 8.88 m² at Oensingen and Ellighausen using a plot combine equipped with a scale. To determine the dry weight, the shoot biomass collected until the heading date and grain sub-samples were oven dried at 55 °C for 72 h. The dry shoot biomass and grain sub-samples were milled using a Retsch rotor mill. Total N was measured according to the Dumas method using an elemental

analyzer (Thermo, flash 2000) (NF ISO 13878) (Masson et al. 2010). Total K was determined by radial ICP-AES (Varian Vista RL Simultaneous or Varian 725ES Simultaneous) after incineration (480 °C for 5 h) and solubilization in HF (Masson et al. 2010). In addition, grain and straw were annually collected at harvest time since the beginning of the trials in order to measure grain yield, biomass production, and K concentration according to the procedures previously described.

Data analysis

All calculations and statistical analyses were performed in the R environment, versions 3.01 (R Core Team 2013) and 4.0.2 (R Core Team 2020). The relationships between the results obtained by two extractions methods of exchangeable soil K (K-AAE and K-AA) and between the two readily available soil K extracts (K-CO₂ and K-H₂O) were tested with linear regressions for each site using all experimental unit replicates. Confidence intervals (95%) of the slopes of the fitted linear regressions were determined using the *matplot* function (*graphics* package). When the confidence intervals obtained in the different sites did not overlap, the differences between sites was considered as significant and the site-specific linear regressions were used separately. In case of overlapping confidence intervals, site differences were considered non-significant and a common linear regression was fitted for the three sites together. Based on the fact that, for a given K-AA concentration, the K buffer power is greater in soil with lower K-H₂O concentration (Silberbush and Barber 1983; Schneider 2003),

$$\begin{aligned} \text{Plateau – linear – plateau : } Y &= Y_{\min} \text{ if } X < X_{t1}, \\ Y &= Y_{\min} + S * (X - X_{t1}) \text{ if } X_{t1} \leq X \leq X_{t2}, \\ \text{and } Y &= Y_{\max} \text{ if } X > X_{t2}, \text{ with } S = \frac{Y_{\max} - Y_{\min}}{X_{t2} - X_{t1}} \end{aligned} \quad (3)$$

the relationships between K-AA and K-H₂O concentrations for the three sites were compared to test if the K buffer power differed among sites. In case of non-overlapping confidence intervals, the K buffer powers of two sites were considered different.

The effects of the K fertilization treatments on extractable soil K (K-Tot, K-AAE, K-AA, K-CO₂, and K-H₂O) after the grain harvest, shoot biomass, shoot K concentration, and the K nutrition index

(KNI; for calculation see below) at each weekly sampling date, and grain yield and grain K concentration at harvest were tested by means of analysis of variance (ANOVA) and subsequent Tukey tests within sites using the function *tukeyHSD* (*stats* package). At Changins, the 0 P–0 K treatment was included for data analysis of extractable soil K but not for data analysis related to plant biomass, grain yield or shoot/grain K concentration since production was limited by P deficiency (Cadot et al. 2018).

To estimate the influence of long-term K fertilization on exchangeable K, the relationship between annual values of K-AAE concentration and the cumulative K budget was analyzed from the set of measurements taken since 1990. We tested if the linear relationship between K-AAE concentrations and the cumulative K budget was restricted by minimum and maximum concentrations of K-AAE, which would be related to a minimal value of exchangeable K (Tabatabai and Hanway 1969) and a maximal value, i.e. the K holding capacity (Goulding et al. 2021), respectively. These values would be reached below and beyond certain cumulative K budgets. To this end, linear-plateau, plateau-linear, and plateau-linear-plateau relationships were defined to relate the K-AAE concentration (Y) to the cumulative K budget (X):

$$\begin{aligned} \text{Linear – plateau : } Y &= Y_{\max} + S * (X - X_t) \text{ if } X < X_t, \\ \text{and } Y &= Y_{\max} \text{ otherwise} \end{aligned} \quad (1)$$

$$\begin{aligned} \text{Plateau – linear : } Y &= Y_{\min} \text{ if } X < X_t, \\ \text{and } Y &= Y_{\min} + S * (X - X_t) \text{ otherwise} \end{aligned} \quad (2)$$

where Y_{\min} and Y_{\max} are the minimum and maximum K-AAE concentration values, S is the slope of the linear part of the function, and X_{t1} and X_{t2} are two concentration thresholds. To account for the hierarchical data structure (serially replicated measurements), year (as factor) was included as a random effect on minimum and maximum plateaus. This also allowed for yearly variation in minimum and maximum K-AAE values. The three models were fitted for each

site separately with the *nlme* function of the *nlme* package. As the models are sensitive to starting values for the parameters, we did grid searches of plausible starting values (Ritz and Streibig 2008) and used the set of parameters that led to model convergence with the lowest Akaike Information Criterion (AIC) and a positive-definite variance–covariance matrix with meaningful standard deviations of the random effects. The function that returned the lowest AIC was eventually chosen for the respective site. To confirm that the assumptions of the models were realistic, nonparametric regressions (local regression implemented with the *loess* R function) were superimposed on the fitted curves to visually check whether lower and upper plateaus were apparent when using a non-parametric technique.

Limiting soil K conditions were estimated based on the outcome of the analysis of the K fertilization effect on shoot biomass measured in 2018 and/or on grain yields. The K fertilization treatments were considered as soil K-limited if, compared to the highest K fertilization rate (5/3 K or K+), shoot biomass decreased significantly ($p < 0.05$) in at least one of the weekly sampling dates in 2018 and/or grain yield decreased significantly in at least one year since the beginning of the experiments. In case of statistical significance, the non-limited K treatment with the lowest K fertilization rate was selected in order to test if a specific critical value for soil K can be derived across sites and years.

The critical K curve was estimated with data of shoot biomass and shoot K concentration. For each experimental unit, only the weekly sampling dates in 2018 with concomitant decrease in shoot K concentration and increase in shoot biomass compared to the previous sampling date were considered. The critical K concentration (K_c) was expressed as a power function of shoot biomass (SB):

$$K_c = a \times SB^b \quad (4)$$

where a and b are estimated parameters (Salette and Huché 1991). The K_c curve was estimated by both a “classical” method and a Bayesian method. The “classical” method, which has been widely used for establishing critical dilution curves, proceeds in two steps e.g. (Bélanger et al. 2015; Cadot et al. 2018; Soratto et al. 2020; Fontana et al. 2021): (1) the lowest non-limiting K fertilization rate for shoot biomass

was identified on each sampling date in 2018 as previously described for the limiting soil K conditions and (2) the K_c curve defined by Eq. (2) was fitted to the data of shoot biomass and K concentration for that non-limiting K fertilization rate by nonlinear least squares. Sites where K fertilization did not affect shoot biomass were not used to estimate the K_c curve. The Bayesian method, an approach more recently used for establishing critical dilution curves, relies on a hierarchical model that includes three levels (Makowski et al. 2020). In the first level, a linear-plus-plateau function is defined to relate shoot K concentration to shoot biomass on each sampling date. The second level describes the variability of the linear-plus-plateau parameters across sampling dates based on a probability distribution and defines the K_c curve as a function of these parameters. The third level describes prior knowledge related to parameter values. To limit the influence of priors on the results, the weakly informative priors defined by Makowski et al. (2020) were used. All the parameters are fitted in one step to the whole dataset using a Markov chain Monte Carlo algorithm [MCMC, *rjags* package, (Plummer, 2017)]. The algorithm was first run with three chains of 100,000 iterations. The posterior distribution of the individual slopes and the R^2 of the linear-plateau models were used to detect whether K limiting conditions occurred at each site. If not, the site was not considered to estimate the K_c curve. Convergence was checked using the Gelman-Rubin convergence diagnosis criteria and then the algorithm was run again with 100,000 additional iterations. The generated values were used to compute the median and 95% credibility intervals for the parameters of the K_c curve. The two methods were compared by testing whether the K_c curve obtained with the “classical” method fell within the 95% credibility interval calculated by the Bayesian method. Finally, a *KNI* was calculated for each weekly sampling date in 2018 and site as follows:

$$KNI(\%) = (K_{measured}/K_c) \times 100 \quad (5)$$

where $K_{measured}$ is the observed K concentration and K_c was calculated with the equation of the K_c curve obtained with the Bayesian approach using the same shoot biomass as observed for the $K_{measured}$ considered. For each K treatment, all *KNI* values were

averaged across sampling dates to obtain a global estimation of the K nutritional status.

As the K_c curve can be affected by the crop N status (Salette and Huché 1991), the N status in 2018 was assessed using the shoot N concentration and the critical N dilution curve of Justes et al. (1994) for winter wheat.

$$N_c = 53.5 \times SB^{-0.44}. \quad (6)$$

Results

Cumulative K budget and K-AAE values since the beginning of the experiments

Differences in cumulative K budget were observed between K fertilization treatments within each site (Fig. 1A). At Changins, the modification of K fertilization treatments in 1985 resulted in cumulative K budgets rather close for the 0 K and 3/3 K treatments whereas the 0 P–0 K treatment was the only one with a negative cumulative K budget. The lack of straw export at Changins resulted in a lower decrease in cumulative K budget for the 0 K treatment over the years compared to Ellighausen and Oensingen. In contrast to the cumulative K budget, the K-AAE values were more influenced by site conditions than by K fertilization treatments

(Fig. 1B). Overall, the K+ treatment at Changins resulted in the highest K-AAE value by far compared to the other sites and/or treatments.

Relationships between results obtained by different extraction methods of soil K

The two indicators of exchangeable K (K-AAE and K-AA), measured in the 0–0.2 m soil layer, were significantly related at each of the three sites (Fig. 2A). The confidence intervals of the three site-specific linear relationships between the K-AAE and K-AA concentrations fully overlapped, indicating that the relationship between these two indicators of exchangeable K was independent of site conditions. Hence, a general relationship was developed using the data from the three sites:

$$K\text{-AAE} = 26.8 + (1.11 \times K\text{-AA}) \quad (p < 0.001, \text{adj.}R^2 = 0.99) \quad (7)$$

The two indicators of readily available K (K-CO₂ and K-H₂O), measured in the 0–0.2 m soil layer, were linearly related at each of the three sites (Fig. 2B). The relationship between the K-H₂O and K-CO₂ concentrations, however, was influenced by site conditions as indicated by the lack of overlapping of the confidence intervals of the site-specific linear relationships. Therefore, no common linear relationship using data from the three sites was established.

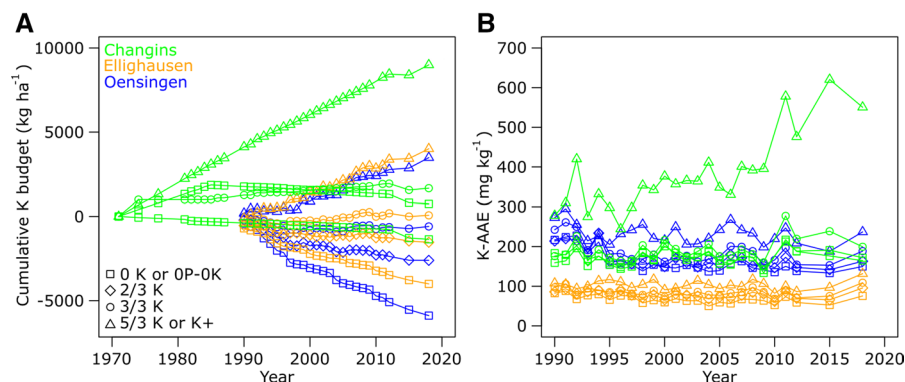


Fig. 1 Changes in cumulative K budget **A** and exchangeable K (K-AAE) concentrations at a 0–0.2 m depth **B** at Changins, Ellighausen, and Oensingen fertilized with various K applications since the setup of the experiments. 0 P–0 K=no P and K

fertilization; 0 K=no K fertilization; 2/3 K=2/3 of the theoretical crop uptake and so on; K+ treatment was fertilized with an additional 166 kg K ha⁻¹ to the theoretical crop K uptake

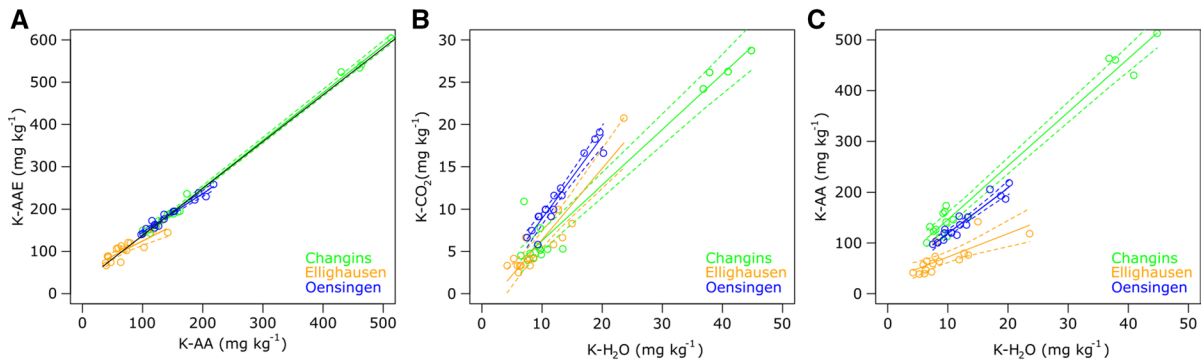


Fig. 2 Linear relationships between concentrations of K-AAE and K-AA **A**, K-CO₂ and K-H₂O **B**, and K-AA and K-H₂O **C** at a 0–0.2 m soil depth at Changins, Ellighausen, and Oensingen. Solid lines represent linear regressions and dashed lines indicate 95% confidence intervals. The black line **A** shows the linear regression established across sites

[K-AAE = 26.8 + (1.11 × K-AA), $p < 0.001$, adj $R^2 = 0.99$]. Relationships between K-AA and K-H₂O concentrations are used to compare the K buffer power among sites because the K buffer power is greater for a soil that has lower K-H₂O concentration for a given K-AA concentration (Silberbush and Barber 1983; Schneider 2003)

A significant relationship between K-AA and K-H₂O concentrations measured in the 0–0.2 m soil layer was found at each of the three sites (Fig. 2C) but the site-specific relationships differed as indicated by the lack of overlapping of the confidence intervals. The highest K buffer power among the three sites, as indicated by the low K-H₂O for a given K-AA, was observed at Changins, whereas the lowest was observed at Ellighausen.

In the 0.2–0.5 m soil layer, K fertilization increased the K-AAE and K-AA concentrations at Ellighausen and Oensingen, and the K-H₂O and K-CO₂ concentrations at Ellighausen only (Table 1). At Changins, only the K-AA concentration was increased in the K+ treatment whereas no significant effect was observed for the other soil K extracts.

Effect of K fertilization on extractable soil K

Relationships between cumulative K budget and soil exchangeable K

The K-Tot concentration in the 0–0.2 m soil layer was not affected by the long-term K fertilization, except at Changins where the K-Tot concentration differed between the 0 P-0 K and the K+ treatments (Table 1). At Ellighausen and Oensingen, concentrations of the four soil K extracts (K-AAE, K-AA, K-H₂O, and K-CO₂) generally increased with increasing K fertilization rates. In contrast, concentrations of the four soil K extracts differed only between the K+ treatment and the other K treatments at Changins. For the K-AAE concentration, the differences observed between K treatments within sites in 2018 were already present several years earlier (Fig. 1B). The variation in K-AAE concentrations across years was frequently greater than that among K fertilization treatments.

The plateau-linear function returned the lowest AIC for the relationship between cumulative K budget and K-AAE at Changins, while the plateau-linear-plateau relationship was most suitable for Ellighausen and Oensingen (Fig. 3; Table S2). Minimum K-AAE concentration was 185 mg K kg⁻¹ at Changins, corresponding to a minimum cumulative K budget of 3525 kg ha⁻¹. Minimum and maximum K-AAE concentrations were respectively 66 and 98 mg K kg⁻¹ at Ellighausen and 164 and 230 mg K kg⁻¹ at Oensingen. Those K-AAE concentrations corresponded to minimum and maximum cumulative K budgets of –1478 and 328 kg ha⁻¹ at Ellighausen and –724 and 103 kg ha⁻¹ at Oensingen.

The trends indicated by the nonparametric regressions were in line with both the plateau-linear model for Changins and the plateau-linear-plateau models for Ellighausen and Oensingen.

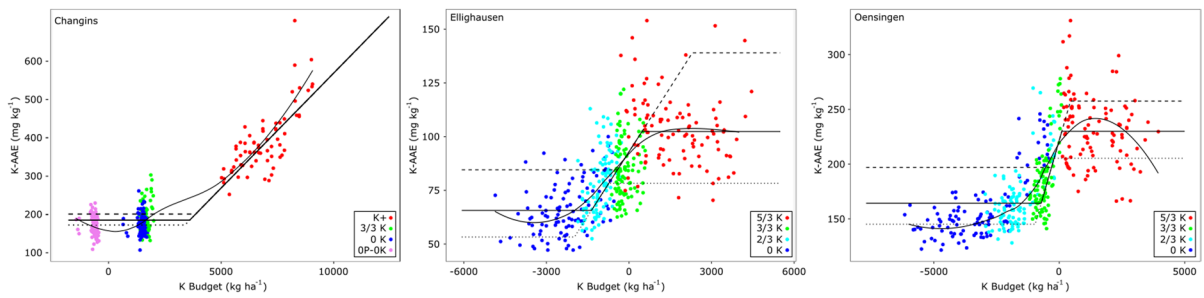


Fig. 3 Relationships between K-AAE concentrations and cumulative K budgets (i.e. shown separately on Fig. 1) at Changins [K-AAE = $101.5 + (0.04 \times \text{K-budget})$, $R^2 = 0.82$, for K-AAE concentrations from 181 to 432 mg kg^{-1} and cumulative K budgets from 1853 to 7694 kg ha^{-1}], Ellighausen [K-AAE = $92.9 + (0.02 \times \text{K-budget})$, $R^2 = 0.57$, for K-AAE concentrations from 66 to 102 mg kg^{-1} and cumulative K budgets from -1716 to 594 kg ha^{-1}], and Oensingen [K-AAE = $22.16 + (0.08 \times \text{K-budget})$, $R^2 = 0.71$, for K-AAE concentrations

ranging from 164 to 230 mg kg^{-1} and cumulative K budgets from -724 to 103 kg ha^{-1}]. Models considered the year of sampling as random factor and dashed lines illustrate the seasonal variation in minimum and maximum K-AAE. 0 K = no K fertilization; 2/3 K = 2/3 of the theoretical crop uptake and so on; K+ treatment was fertilized with an additional 166 kg K ha^{-1} to the theoretical crop K uptake. Additionally, nonlinear regressions (loess) were superimposed for each site (smooth curves)

Table 2 Shoot biomass at individual wheat developmental stages during wheat growth in 2018 following decades of different K fertilization treatments at three sites in Switzerland

Site	Treatment*	Shoot biomass (Mg DM ha ⁻¹)							
		**22–29	23–31	25–32	27–32	29–37	30–45	35–55	45–55
Changins	0 K	0.70 a	0.76 a	1.03 a	1.44 a	2.73 a	4.17 a	6.37 a	6.96 a
	3/3 K	0.64 a	0.78 a	1.07 a	1.59 a	2.87 a	4.44 a	6.09 a	8.61 a
	K+	0.79 a	0.86 a	1.25 a	1.75 a	3.33 a	5.32 a	6.85 a	8.76 a
Ellighausen	0 K	0.98 a	1.35 a	1.54 a	1.87 b	2.76 b	4.56 b	5.94 a	9.48 a
	2/3 K	1.03 a	1.31 a	1.63 a	2.39 ab	3.48 ab	5.50 ab	7.61 a	10.24 a
	3/3 K	1.09 a	1.41 a	1.91 a	2.54 a	3.98 a	6.54 a	7.87 a	11.94 a
Oensingen	0 K	1.12 a	1.57 a	1.98 a	2.76 a	4.04 a	6.24 a	7.63 a	10.69 a
	2/3 K	1.26 a	1.67 a	1.96 a	2.85 a	3.93 a	6.44 a	7.93 a	9.87 a
	3/3 K	1.21 a	1.63 a	2.09 a	2.78 a	4.06 a	6.26 a	7.48 a	9.22 a
	5/3 K	1.22 a	1.61 a	2.15 a	2.72 a	4.20 a	5.67 a	7.81 a	10.22 a

Different lowercase letters indicate significant differences ($p < 0.05$, ANOVA and Tukey test) between K fertilization treatments within a site and a range of developmental stages. *0 K = no K fertilization; 2/3 K = 2/3 of the theoretical crop uptake and so on; K+ treatment was fertilized with an additional 166 kg K ha^{-1} to the theoretical crop K uptake. **The developmental stages (CD) were determined according to Meier (2018)

Shoot biomass production of winter wheat

Maximum shoot biomass production varied substantially across sites from $8.8 \text{ Mg DM ha}^{-1}$ at Changins to $11.9 \text{ Mg DM ha}^{-1}$ at Ellighausen (Table 2). At Changins and Oensingen, shoot biomass between the developmental stages CD 22 and CD 55 was not affected by K fertilization. The 0 K treatment was therefore considered the non-limiting K treatment at

those two sites. At Ellighausen, only the 0 K treatment had a lower shoot biomass for the developmental stages CD 27 to CD 45 ($p < 0.05$, Table 2). This 0 K treatment was also the only treatment with a mean of relative shoot biomass production across sampling dates (0.79) less than 0.80. The 2/3 K treatment at Ellighausen was therefore considered the non-limiting K treatment with the lowest K fertilization rate.

Shoot K concentration

From the beginning of the growing season to the CD 27–32 developmental stages, the shoot K concentration increased up to 30–45 g K kg⁻¹ DM depending on site, corresponding to a shoot biomass of 1.7–3 Mg DM ha⁻¹ (Tables 2 and 3). For a greater shoot biomass, shoot K concentrations decreased and K dilution occurred. At Changins, shoot K concentrations increased from the 0 K to the K+ treatments for the developmental stages CD 22 to CD 55 ($p < 0.05$; Table 3). At Ellighausen, shoot K concentrations differed among treatments at all developmental stages ($p < 0.05$), most prominently between the 0 K and the other K treatments. At Oensingen, shoot K concentration increased from the 0 K to the 5/3 K treatments on the four sampling dates corresponding to developmental stages CD 27 to CD 55.

Shoot N status

At Ellighausen and Oensingen, no N deficiency occurred as indicated by shoot N concentrations generally greater than the critical N dilution curve proposed by Justes et al. (1994) (Fig. 4). In contrast,

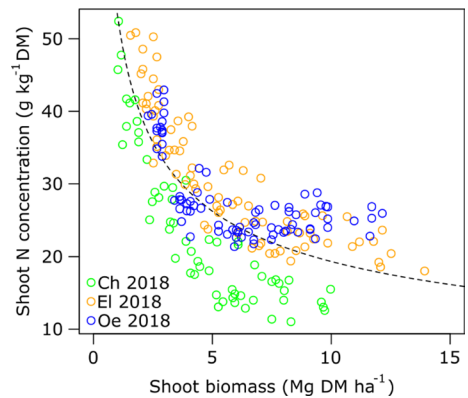


Fig. 4 Shoot N concentration as a function of wheat shoot biomass for all K treatments at Changins, Ellighausen, and Oensingen along with the critical N curve according to Justes et al. (1994) ($N_c = 53.5 \times SB^{-0.44}$ with N_c the critical N concentration and SB the shoot biomass, indicated by the dotted line)

values of shoot N concentration at Changins were generally lower than the critical N dilution curve, which indicates a N deficiency.

Table 3 Shoot K concentration at individual wheat developmental stages during wheat growth in 2018 following decades of different K fertilization treatments at three sites in Switzerland. The K nutrition index was calculated for each sampling

Site	Treatment*	Shoot K concentration (g kg ⁻¹ DM)								KNI (%)
		**22–29	23–31	25–32	27–32	29–37	30–45	35–55	45–55	
Changins	0 K	26.0 bc	29.5 c	27.6 c	30.2 b	30.9 c	24.4 b	19.4 b	16.8 a	74 b
	3/3 K	30.4 ab	34.0 bc	37.3 ab	39.8 ab	39.0 ab	28.9 b	21.6 b	17.7 a	88 b
	K+	33.0 a	41.6 a	44.9 a	48.8 a	46.4 a	34.5 a	26.4 a	21.8 a	115 a
Ellighausen	0 K	12.8 c	16.4 b	16.7 b	22.1 c	22.0 c	18.1 c	15.8 c	15.3 b	61 c
	2/3 K	22.7 b	25.9 a	33.2 a	37.2 b	32.6 b	25.9 b	21.4 b	20.1 a	95 b
	3/3 K	26.3 a	29.6 a	35.0 a	40.9 ab	36.6 a	29.5 a	23.1 ab	20.2 a	109 a
	5/3 K	25.5 ab	30.5 a	37.0 a	43.3 a	38.2 a	30.4 a	24.5 a	22.0 a	109 a
Oensingen	0 K	26.4 a	34.9 b	29.3 b	37.3 b	32.6 a	27.2 a	23.8 b	23.1 a	105 a
	2/3 K	26.4 a	38.1 ab	32.0 ab	39.0 ab	33.2 a	27.7 a	25.1 ab	23.7 a	109 a
	3/3 K	26.1 a	38.0 ab	31.5 ab	38.6 ab	31.8 a	27.4 a	24.4 ab	23.3 a	105 a
	5/3 K	27.4 a	39.6 a	34.8 a	41.0 a	34.7 a	28.4 a	26.4 a	25.1 a	113 a

Different lowercase letters indicate significant differences ($p < 0.05$, ANOVA and Tukey test) between K fertilization treatments within a site and a developmental stage. *0 K = no K fertilization; 2/3 K = 2/3 of the theoretical crop uptake and so on; K+ treatment was fertilized with an additional 166 kg K ha⁻¹ to the theoretical crop K uptake. **The developmental stages (CD) were determined according to Meier (2018)

date [KNI = $(K_{\text{measured}} / K_c) \times 100$ with K_c obtained through the Bayesian approach using data from Ellighausen] and averaged across all sampling dates for each treatment and site

Table 4 Grain yield of winter wheat in long-term field experiments with different K fertilization treatments at three sites in Switzerland

Year	Changins			Ellighausen				Oensingen			
	Treatment*			Treatment				Treatment			
	0 K	3/3 K	K+	0 K	2/3 K	3/3 K	5/3 K	0 K	2/3 K	3/3 K	5/3 K
1973	4.8 a	4.9 a	4.9 a								
1975	4.5 a	4.3 a	4.3 a								
1977	3.3 a	3.3 a	3.5 a								
1979	4.9 a	5.0 a	5.0 a								
1981	4.2 a	4.3 a	4.1 a								
1983	2.4 a	2.5 a	2.6 a								
1985	4.2 a	3.8 a	4.2 a								
1987	4.7 a	4.7 a	4.5 a								
1989	5.5 a	5.4 a	5.5 a	Beginning of the experiments							
1990	**	–	–	6.3 a	6.3 a	6.1 a	6.1 a	–	–	–	–
1991	6.4 a	6.5 a	6.3 a	–	–	–	–	–	–	–	–
1992	–	–	–	–	–	–	–	4.8 a	5.1 a	5.0 a	5.1 a
1993	3.8 a	3.6 a	3.7 a	–	–	–	–	–	–	–	–
1995	5.5 a	5.2 a	5.4 a	–	–	–	–	–	–	–	–
1997	4.1 a	4.1 a	3.8 a	–	–	–	–	–	–	–	–
1998	–	–	–	4.8 a	4.9 a	5.6 a	5.4 a	5.1 a	5.1 a	5.2 a	5.1 a
1999	4.6 a	4.8 a	4.8 a	–	–	–	–	–	–	–	–
2001	1.3 a	1.7 a	1.6 a	4.5 b	6.0 a	6.0 a	6.0 a	4.5 a	4.7 a	4.8 a	4.6 a
2003	4.6 a	4.6 a	4.4 a	–	–	–	–	–	–	–	–
2005	3.9 a	4.4 a	4.5 a	–	–	–	–	–	–	–	–
2007	6.5 a	6.2 a	6.4 a	–	–	–	–	–	–	–	–
2008	–	–	–	5.3 b	5.9 a	5.9 a	5.9 a	4.3 a	4.6 a	4.4 a	4.5 a
2009	4.2 a	4.2 a	4.0 a	–	–	–	–	–	–	–	–
2011	5.7 a	5.5 a	5.3 a	–	–	–	–	–	–	–	–
2013	5.9 a	6.4 a	7.0 a	4.5 a	5.2 a	5.6 a	5.4 a	4.5 a	4.6 a	4.3 a	4.2 a
2016	4.0 a	4.0 a	4.0 a	–	–	–	–	–	–	–	–
2018	4.8 a	4.9 a	4.9 a	4.8 a	5.2 a	4.8 a	5.2 a	4.2 a	4.0 a	3.9 a	4.1 a

Significant differences ($p < 0.05$) among treatments within sites and years are indicated by different lowercase letters. *0 K=no K fertilization; 2/3 K=2/3 of the theoretical crop uptake and so on; K+ treatment was fertilized with 166 kg K ha⁻¹ in addition to the theoretical crop K uptake
 **Winter wheat was grown in rotation with other crops. Grain and biomass yields and K concentrations of winter wheat and the other crops in the rotation are reported in supplementary material (Tables S3 to S7)

Grain yield

At Changins and Oensingen, grain yields were not influenced by several decades of contrasted K fertilization rates since the beginning of the experiment, including in 2018 (Table 4). At Ellighausen, although the grain yield was not affected by K fertilization in 2018, the 0 K treatment had lower grain yields than the 3/3 K treatment in 2001 and 2008. The grain K export was not affected by several decades of contrasted K fertilization rates in 2018 at the three sites (data not shown).

Limiting soil K conditions

Potassium fertilization affected shoot biomass at three development stages in 2018 along with grain yields in 2001 and 2008 only at Ellighausen (Table 2 and Table 4). The values of the four extracts (0–0.2 m soil layer) measured in 2018 from the 2/3 K treatment at that site were 96 mg K kg⁻¹ for K-AAE, 57 mg K kg⁻¹ for K-AA, 4.4 mg K kg⁻¹ for K-CO₂, and 7.1 mg K kg⁻¹ for K-H₂O (Table 1).

K_c curves

Because of the lack of a positive response to K fertilization at Changins and Oensingen, Ellighausen was the only site where a K_c curve could be estimated with the “classical” method. The 2/3 K treatment corresponded to the lowest fertilization rate for a maximum shoot biomass production at Ellighausen, and data points from that treatment were used to estimate the K_c curve:

$$K_c = 53.17 \times SB^{-0.42} \quad (8)$$

with K_c being the critical K concentration defined as a function of shoot biomass (SB).

For the Bayesian method, Gelman-Rubin convergence diagnosis criteria indicated that convergence was reached for K_c curves at the three sites (Supplementary Fig. S1). However, this method yielded linear plateau models with slopes probably null and R^2 generally null for Changins and Oensingen (Fig. S2), indicating that shoot biomass production was probably not limited by shoot K concentration. Therefore, data points from those two sites were not considered to estimate the K_c curve. At Ellighausen, the slopes of the linear-plateau relationships were most probably

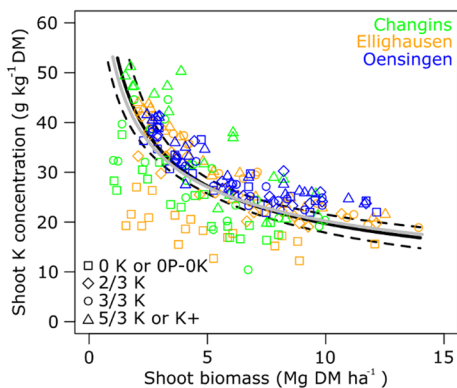


Fig. 5 Estimated K_c curves with the Bayesian approach (black, $K_c = 58.21 \times SB^{-0.45}$) and the “classical” approach (grey). For the Bayesian approach with data from Ellighausen, continuous and dashed lines (black) show the posterior median and the 95% credibility interval, respectively. For the “classical” approach, the K_c curve (grey) was estimated with data points from the 2/3 K treatment at Ellighausen, which corresponded to the lowest K rate that achieved the maximum shoot biomass accumulation. Data points for all K fertilization treatments and sites are presented

close to 1.0 and their R^2 ranged between 0.09 and 0.43 (Fig. S2). Data points from that site were used to estimate the K_c curve:

$$K_c = 58.21 \times SB^{-0.45} \quad (9)$$

The K_c curves obtained with both methods nearly overlapped (Fig. 5). The K_c curve from the “classical” method was within the confidence intervals of that from the Bayesian method, indicating that both methods yielded similar results. Based on the K_c curve estimated with the Bayesian method, the KNI ranged between 61 and 109% at Ellighausen, between 74 and 115% at Changins, and between 105 and 113% at Oensingen (Table 3).

Discussion

Converting soil K extracts and K buffer power across various site conditions

The K-AAE extract used in the Swiss fertilization guidelines (Flisch et al. 2017) to estimate the exchangeable K yielded slightly greater values than the K-AA extract, likely due to the K complexed by EDTA. However, the values can be converted from one to the other by using Eq. (5) as shown for the first time (Fig. 2).

Greater amounts of readily available K were extracted by K-H₂O than by K-CO₂ (Table 1) due to the lower soil-to-extractant ratio and the longer extraction time for K-H₂O (Stünzi 2007; Blanchet et al. 2017). The influence of site conditions on the relationship between the K-H₂O and K-CO₂ concentrations makes the conversion of extracted amounts of K between those methods not possible (Fig. 2). In a previous study, greater amounts of K were extracted with K-CO₂ than with K-H₂O for a similar soil-to-extractant ratio, except for acidic soils (i.e. pH=4.2–5), where water acidified with CO₂ did not influence the extractant pH (Stünzi 2007). Therefore, for a given K-H₂O concentration, the higher soil pH at Oensingen could explain the greater K-CO₂ concentration compared to that at Changins and Ellighausen (Fig. 2, Table S1). In addition, the CO₂ degassing during the filtration of the soil-extractant mixture for K-CO₂ (Stünzi 2007) may induce a slight bias since the extractant pH can be more or less affected

depending on the lag time required for filtration. These observations suggest that the K-CO₂ extract might be more influenced by soil properties than the K-H₂O extract. The K-H₂O method is more relevant internationally and, therefore, should be preferred. However, we cannot put forward any recommendation to measure readily available K for crops per se.

The linear relationships between K-H₂O and K-AA concentrations were different for each site ($p < 0.05$, Fig. 2), showing that values of both K extracts cannot be converted to one another independently of site conditions. At Changins, no relationship was observed if the K+ treatment was not considered (Fig. 2). The very high cumulative K budget for the K+ treatment greatly decreased the soil ability for K release and fixation due to a higher K-AA value compared to other K treatments [Fig. 2 and Schneider et al. (2013)]. The greater K buffer power observed at Changins compared to other sites likely results from the high clay content (Table S1) (Sharpley 1990; Schneider 2003). At Ellighausen and Oensingen, exchangeable K and readily available K were linearly related as already observed in another study for a coarse-textured soil (Halstead and Heeny 1959).

Long term K fertilization effect on soil exchangeable K

Long-term K fertilization increased exchangeable K (Table 1) as frequently observed (Blake et al. 1999; Lalitha and Dhakshinamoorthy 2014). Exchangeable K is typically less than 2% of total K (Lalitha and Dhakshinamoorthy 2014). This is in line with the range of the K-AA-to-K-Tot ratio that varied from around 0.5% for the 0 K treatment at the three sites to up to 2% for the K+ treatment at Changins (Table 1).

Variations in the cumulative K budgets affected the exchangeable K only at specific ranges that differed for each site (Fig. 3). The plateau-linear-plateau relationships indicated that, in the range of cumulative K budgets that linearly affected the exchangeable K, the soil K sorption sites were progressively saturated between a minimal K-AAE concentration (Tabatabai and Hanway 1969) and the K holding capacity (Goulding et al. 2021). Outside this range, the exchangeable K was not affected by variations in cumulative K budget, suggesting an influence of the non-exchangeable K pool when the minimal K-AAE concentration was reached. At Changins, the

K holding capacity remains to be determined for K budgets beyond 9000 kg ha⁻¹ since the nonparametric regression and the comparison between plateau-linear and plateau-linear-plateau models do not confirm that an upper plateau was reached (Fig. 3). At this site, the non-exchangeable K pool likely provided K for crops and the K fertilizer applied was probably fixed in this non-exchangeable K pool for cumulative K budgets less than ≈ 1850 kg K ha⁻¹ associated with the minimal K-AAE concentrations (Fig. 3). The contribution of the non-exchangeable K pool for crop nutrition was also demonstrated for a clay soil of the Canadian Shield when exchangeable K decreased (Damar et al. 2020). These observations can partly explain why relationships between the cumulative K budget and exchangeable K were not significant in all studies and are poorly generalizable (Buchholz et al. 2004; Fernández and Hoef 2009; Damar et al. 2020; Franzen et al. 2021). Future studies should test to what extent a specific soil texture or mineralogy affect the relationship between cumulative K budget and exchangeable K. Interestingly, the lowest K-AA concentration measured in 2018 at Changins (127 mg K kg⁻¹, Table 1) was close to the minimal K-AA concentration reported for another clay soil (≈ 120 mg K kg⁻¹; Franzen et al. 2021). However, data are scarce and, to our knowledge, this study is the first that determined the minimal K-AA concentration and the K holding capacity in the field for contrasting soil types.

At Ellighausen and Oensingen, the K-AA concentration in the 0.2–0.5 m soil layer in 2018 increased linearly with the K budget ($p < 0.001$, adj $R^2 = 0.95$ and 0.96, respectively) whereas K-AA was unchanged at Changins for 0 K–0 P, 0 K and 3/3 K (Table 1), despite notable differences in K budget.) This highlights a more limited K transfer from the 0–0.2 m soil layer to the 0.2–0.5 m soil layer at Changins compared to the other sites with a coarser texture, which is in line to previous studies (Rosolem et al. 2010; Mendes et al. 2016).

K fertilization effect on grain yield and aboveground biomass production

Differences in shoot biomass and grain yield were greater among sites than among the K fertilization treatments (Tables 2 and 4), as observed for long-term experiments on P fertilization established at the

same locations (Fontana et al. 2021). This highlights the differences of pedo-climatic conditions across the three long-term experiments. Although the K fertilization treatments resulted in a strong gradient of soil K (Table 1), the wheat grain yield was not affected in 2018 at the three sites. At Changins and Oensingen, the grain yield has never been decreased during 30 years of no K fertilization, while significant differences in wheat grain yield between the 0 K treatment and the other K treatments were observed in 2001 and 2008 at Ellighausen (Table 4). The lack of a significant effect of K fertilization in 2018 at Ellighausen might be due to a lower K demand compared to 2001 and 2008 when grain yields were 16% and 13% greater than in 2018, respectively. Ellighausen was also the only site where shoot biomass production was affected by K fertilization in 2018 (Table 2). As for P fertilization (Fontana et al. 2021), the response to K fertilization was greater for shoot biomass production than for grain yield, suggesting that the diagnostic of K deficiency or sufficiency performed on the shoot biomass during the growing season is a good early indicator of a potential yield loss. The lack of a long-term K fertilization effect on wheat grain yield was also reported for a large array of pedoclimatic conditions (Swarup and Ghosh 1979; Swarup and Chhillar 1986; Jouany et al. 1996; He et al. 2012; Zhao et al. 2014). However, grain yield and shoot biomass were affected by K fertilization, especially for coarse soil textures with very low exchangeable K (Damon and Rengel 2007), as observed at Ellighausen. Overall, our results underline that K fertilization might not be necessary for certain soil types.

Limiting soil K conditions for winter wheat

Because limiting soil K conditions were observed only at Ellighausen, the site with the greatest sand content and the lowest pH (Table S1), determining critical soil K values for a range of pedoclimatic conditions was not possible. At Ellighausen, the 2/3 K treatment was the lowest K rate that achieved maximum shoot biomass in 2018 along with maximum grain yields in 2001 and 2008 (Table 4). The K-AA value observed in 2018 (57 mg K kg⁻¹, Table 1) for that treatment is in the range of critical values of around 44.4 to 71.0 mg K kg⁻¹ reported for a large array of Australian soils using the Colwell extract that correspond to K-AA (Brennan and Bell 2013; Zhang

et al. 2017). Higher critical values for K-AA (82 and 91 mg K kg⁻¹) were reported for winter wheat grown under contrasted pedoclimatic conditions (Bedi et al. 2002; El Dessougi et al. 2002; He et al. 2012; Zhao et al. 2014; Zhang et al. 2017). This confirms that the soil K status obtained with a critical value of soil exchangeable K is strongly uncertain and varies with soil types.

The range of K-AAE values (62 to 96 mg K kg⁻¹ [corresponding to 31.7 and 62.3 mg K kg⁻¹ for K-AA according to Eq. (5)]) obtained since 2001 at Ellighausen for the 2/3 K treatment (Fig. 1B and Table S4) is representative of the uncertainty of critical K-AAE values that can be observed for specific soil conditions. This range partly overlapped that observed for the 0 K treatment for the same years [50–75 mg K kg⁻¹, corresponding to 20.9–43.4 mg K kg⁻¹ for K-AA according to Eq. (5)]. Overall, 64% of the K-AAE values observed for the 0 K treatment since 2001 were classified by the Swiss fertilization guidelines (Flisch et al. 2017; Sinaj et al. 2017) as “moderate” whereas 93% of the K-AAE values observed for the 2/3 K treatment were classified as “sufficient”. Although the K-AAE concentration of 64 mg K kg⁻¹ set as the limit between “sufficient” and “moderate” for winter wheat in the Swiss fertilization guidelines seems a reasonable trade-off, the uncertainty related to soil conditions during sampling can also lead to an incorrect diagnostic of soil K status. The variability of K-AAE concentrations across sampling years was sometimes greater than that between K treatments (Fig. 1), resulting also to an uncertainty around the minimal K-AAE concentration and K-AAE holding capacity (Fig. 3). Accordingly, the relationship between the cumulative K budget and exchangeable K can be different depending on the sampling year (Jouany et al. 1996), possibly due in a large part to the variability in the water content of the soil samples that can influence exchangeable K (Luebs et al. 1956). This highlights the limitation for setting a critical value based on exchangeable K, even for specific soil conditions.

Because the minimal level of exchangeable K is positively related to the cation exchange capacity (Schneider et al. 2016), a critical value based on exchangeable K could never be reached for soils with a high cation exchange capacity. For instance, the minimal K-AAE values observed at the sites with the highest clay content and soil pH (Changins and

Oensingen) are largely greater than the values of the “sufficient” K status indicated by the Swiss fertilization guidelines. In addition, the minimal K-AA values observed at these sites in 2018 (Table 1) are clearly greater than 100 mg K kg^{-1} , which was considered as a threshold beyond which K fertilization is not recommended for winter wheat cropped in the north China plain (He et al. 2012). Soil parent materials at Changins and Oensingen maintained very high values of exchangeable K, as already observed for Cambisoiil (Maiksteniene et al. 2008). At Oensingen, the higher values of iron from amorphous materials (i.e. oxalate extract) and soil pH (table S1) than at the other sites (Demaria et al. 2013) are consistent with high exchangeable K (Han et al. 2021). The clay-rich soil at Changins derives from a marly molasses, resulting in high smectite content (Gratier) which is supported by a noticeable soil shrinkage ($\approx 7\%$, unpublished data). This high smectite content assumed at Changins would be consistent with high exchangeable K (Sharpley 1989). This raises the question whether or not a soil having a minimal exchangeable K concentration greater than a certain critical value of exchangeable K for a given crop can be K limited. As the negative K balance that a soil can support corresponds to the amount of K provided by weathering (Simonsson et al. 2007), it should be formally tested in future studies to what extent K provided by weathering is linked to the minimal exchangeable K. Overall, our results show that exchangeable K or readily available K could be used as rough indicators that should be used with caution only for soils with a coarse texture with a low minimal concentration of exchangeable K but not for clay soils. The cutoff between these two extreme cases is unknown and should be investigated. To improve the soil K diagnostic, Sharpley (1989) proposed measurements of exchangeable and non-exchangeable K of soils previously sorted according to their clay type. Since the clay mineralogy is unknown for most agricultural land, the soil K diagnostic could be also improved by developing a fertilization guideline that would include the exchangeable K, the non-exchangeable K, and the K buffer power.

K_c curve and K status of winter wheat linked to soil K

The results obtained with a Bayesian and a “classical” method both confirmed that Ellighausen was the only site where a K_c curve could be estimated with an acceptable level of accuracy. The “classical” method resulted in a K_c curve ($K_c = 53.17 \times SB^{-0.42}$) that was similar to that obtained with the Bayesian method ($K_c = 58.21 \times SB^{-0.45}$, Fig. 5) as was also observed for the critical N dilution curve of spring wheat (Jégo et al. 2022). The KNI, calculated with the K_c curve based on the Bayesian method, was close to 98% (Table 3) for the 2/3 K treatment, the treatment that was used to estimate the K_c curve with the classical method. This result confirms that both methods were consistent in estimating critical K concentrations.

The highest KNI values (Table 3) provide an estimation of the maximal K luxury consumption of winter wheat (i.e. the maximal possible K concentration in wheat biomass), which is relatively low compared to carrots, peas, leeks, and red beet (Greenwood and Stone 1998). This small gap between the maximal luxury consumption and the critical shoot K concentration could make the K diagnostic more difficult for winter wheat. As this study presents the first K_c curve developed for winter wheat, it should be confirmed in future studies for contrasted pedoclimatic conditions. Considering the current state of knowledge, it is useful to compare the critical shoot K concentrations obtained at Ellighausen with those reported in previous studies. In a pot experiment, a plant K concentration of around $42\text{--}43 \text{ g kg}^{-1} \text{ DM}$ was identified as critical for wheat at the CD 30 developmental stage (Zhang et al. 2017). At a similar developmental stage, the shoot K concentration observed at Ellighausen for the 2/3 K treatment was $\approx 37 \text{ g kg}^{-1} \text{ DM}$, whereas values greater than $40 \text{ g kg}^{-1} \text{ DM}$ were observed only for the 5/3 K or K+ treatments (Table 3). Rao (1986) estimated a critical value 30 days after wheat sowing (corresponding roughly to the CD 30 developmental stage according to Zhang et al. (2017) of $38 \text{ g K kg}^{-1} \text{ DM}$, which is close to our critical value of $37 \text{ g kg}^{-1} \text{ DM}$ obtained at CD 30.

The KNI values (Table 3) ran parallel to the soil K concentrations at Ellighausen and Oensingen. The lowest KNI value was observed at Ellighausen for the 0 K treatment. It corresponded to the lowest values of exchangeable and readily available K among the three sites (Table 1 and Fig. 1) along with a reduction in biomass production. At Oensingen, the high KNI values for all K treatments are in line with the greater values of exchangeable and readily available K than at Ellighausen (Table 1). This suggests that the K uptake was not limited at Oensingen, even for the 0 K treatment that had reached the minimal level of exchangeable K (see previous discussion). Compared to the two other sites, the higher soil pH at Oensingen (Table S1) favored soil K mobilization and K uptake.

In contrast, the low KNI values observed at Changins for the 0 K and 3/3 K treatments (Table 3) indicate a lower K uptake than at the other sites and do not match the greater values of soil K extracts compared to those at Ellighausen and Oensingen (Table 1). This low K uptake may be explained by the greater K buffer power at Changins than at the other two sites (Fig. 2) (Sharpley 1990; Schneider 2003). At Changins, the increase in K fertilization decreased the K buffer power as demonstrated by the difference between the 0 K and K+ treatments (Schneider et al. 2013) and likely enhanced the K uptake. This would be consistent with the fact that differences between the 0 K and 3/3 K treatments were observed for KNI values (Table 3) but not for K extracts (Table 1). It is also possible that the K uptake might have been hampered at Changins by a N deficiency (Fig. 4), as already observed for wheat and other crop species (Swarup and Chhillar 1986; Salette and Huché 1991; Greenwood and Stone 1998). Similarly, the N deficiency decreased the shoot P dilution curve in spring wheat (Ziadi et al. 2008) and winter wheat at Changins compared to Ellighausen and Oensingen (Fontana et al. 2021). Alternatively, the lower K uptake at Changins could be due to the fact that a different cultivar of winter wheat was sown than at Ellighausen and Oensingen. The growth response to K fertilization and the wheat K use efficiency are known to differ depending on genotypes (Damon and Rengel 2007; Chachar et al. 2015). For potatoes, critical dilution curves of N and P were not affected by genotype in contrast to K_c curves (Gómez et al. 2019). Possibly, the K_c curve identified for Ellighausen might be generalizable only under certain conditions. At Changins,

the low KNI values did not correspond to a limitation of shoot biomass production since it was not affected by either the K fertilizer rate or the shoot K concentration (Table 2 and Fig. S2). Future studies are needed to test to what extent the K_c curve is cultivar-specific or site-specific and is affected by N uptake.

Conclusions

Among four K extracts investigated in this study, K-AAE concentrations can be converted into K-AA concentrations with a high level of confidence whereas the relationship between K-H₂O and K-CO₂ concentrations is likely influenced by soil pH. The effects of long-term K fertilization on exchangeable K and readily available K varied among sites, mostly because of their contrasting K buffer power. Exchangeable K responded to K fertilization only for specific ranges of the cumulative K budget, with site-specific variations in exchangeable K from a minimal level to the K holding capacity. Below the cumulative K budget corresponding to the minimal level of exchangeable K, K fertilization affected only K uptake but not the exchangeable K, suggesting a contribution of non-exchangeable K to crop K uptake.

After several decades of contrasted K fertilization rates, a K limitation occurred only at the site with the coarsest soil texture where the parent material weathering did not provide enough K to meet the winter wheat K demand. Soil K extracts measured at this site resulted in values generally in the range of soil K critical values previously reported, despite a strong annual variability likely due to moisture conditions during soil sampling. At the two other sites, grain yield over the course of the experiments and shoot biomass during the growing season in 2018 were not affected, showing that soil was not limited in K and suggesting that weathering provided enough K to meet the wheat K demand. Because the minimum level of exchangeable K was greater than the critical soil K values reported in the literature, it is unclear if winter wheat could ever be K-limited at those two sites. Exchangeable K can provide a rough estimate of soil K availability for winter wheat that should be used with caution only for soil types with a coarse texture. To generalize a method for assessing soil K availability for contrasted soil conditions, the

non-exchangeable K and the K buffer power should also be considered.

The Bayesian and “classical” methods used for estimating the K_c curve with data from the K-limited site yielded similar results. The plant K diagnosis could be a valuable option to diagnose the K status of winter wheat but future studies are necessary to confirm the K_c curve derived from these long-term experiments and to determine to what extent the K_c curve is influenced by wheat cultivars and the status of other nutrients.

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