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# Temporal variability of soil fertility indicators and sampling periods in Québec

Hakima Chelabi, Lotfi Khiari, and Jacques Gallichand

**Abstract:** An inadequate soil sampling time leads to difficulties in interpreting soil tests, to incorrect recommendations for soil amendments and fertilizers, and to inappropriate environmental protection restrictions. Soil samples may be collected from agricultural fields before, during, or after the crop growth period. Since the time of soil sample collection can affect soil tests results, the objective of this study was to evaluate the effect of sampling time on measurements representativity of 15 fertility indicators in two fields located in La Pocatière (Québec, Canada). The soils were of fine (G1) and medium (G2) textural groups and were sampled weekly for 33 weeks per year during four years. Data analyses included descriptive statistics, time-series decomposition, and time autocorrelation function (ACF). Since results of these analyses showed a clear seasonal effect only for Mehlich-3 extracted phosphorus ( $P_{M3}$ ), soil phosphorus saturation index (SPS) for both G1 and G2 soils, and for  $pH_W$  for G1 only, we recommend that the sampling calendar should be restricted to the first five weeks of spring (until the end of May) and to the entire fall period (starting in early September). Also, the temporal autocorrelation was four weeks on average. This implies that, for an initial year, whichever date is chosen for the sampling, the following annual sampling should be done within a four-week time window (i.e., two weeks before until two weeks after the initial sampling date). Time series are an important element to consider in selecting a representative sampling period for soil fertility indicators.

*Key words:* temporal variability, soil test, soil sample representativity, sampling season.

**Résumé :** Un temps d'échantillonnage du sol inadéquat conduit à des difficultés d'interprétation des analyses de sols, à des recommandations incorrectes pour les amendements et les engrais et à des restrictions inappropriées de protection de l'environnement. Dans les champs agricoles, les échantillons de sol peuvent être prélevés avant, pendant ou après la saison de croissance. Puisque la période d'échantillonnage peut affecter les résultats, l'objectif de cette étude était d'évaluer l'effet du temps d'échantillonnage sur la représentativité des mesures de 15 indicateurs de fertilité dans deux champs situés à La Pocatière (Québec, Canada). Ces sols sont de texture fine (G1) et moyenne (G2) et ont été échantillonnés hebdomadairement pendant 33 semaines par année et durant quatre années. Les analyses des données comprenaient : des statistiques descriptives, la décomposition des séries chronologiques et la fonction d'autocorrélation temporelle (ACF). Puisque les résultats de ces analyses montrent un effet saisonnier clair seulement pour le phosphore extrait au Mehlich-3 ( $P_{M3}$ ), l'indice de saturation des sols en phosphore (SSP) dans les sols G1 et G2 et pour  $pH_W$  dans G1, on recommande que le calendrier d'échantillonnage soit limité aux cinq premières semaines du printemps (jusqu'à la fin mai) et à toute la période de l'automne (à partir du début septembre). Aussi, l'autocorrélation temporelle est de quatre semaines en moyenne. Ceci implique que, pour une année initiale, quelle que soit la date d'échantillonnage, l'échantillonnage annuel suivant devrait être fait dans une fenêtre de quatre semaines (i.e. à partir de deux semaines avant jusqu'à deux semaines après l'échantillonnage initial). Les séries chronologiques sont un outil important pour le choix d'une période représentative d'échantillonnage des sols.

*Mots-clés :* variabilité temporelle, analyse du sol, représentativité de l'échantillon de sol, saison d'échantillonnage.

## Introduction

Soil tests are essential for recommending crop fertilization and calcium amendment. These tests are

also required for protecting the environment (MDDELCC 2017). Some of these soil tests, such as pH, micronutrients, bioavailable phosphorus, potassium,

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and soil phosphorus saturation index (SPS), can vary considerably from season to season (Cameron et al. 1994). Several authors noted wide ranges of temporal variation for these soil tests. These ranges are measured by the standard deviation ratio (SDR) that can be as high as 23% for bioavailable phosphorus (Nyborg et al. 1992), 91.9% for soil organic matter (Dai et al. 2011), 54.64% for bioavailable potassium, and 43.8% for bioavailable Cu (Cameron et al. 1994). In Michigan, the seasonal variation of soil pH reported by Collins et al. (1970) was 0.8 pH units. CRAAQ (2010) defines three soil textural groups: G1 (fine: heavy clay, clay, silty clay, silty clay loam, clay loam, sandy clay, sandy clay loam), G2 (medium: loam, silt loam, silt), and G3 (coarse: sand, loamy sand, sandy loam). Because most soil fertility indicators are more stable in fine than in coarse textured soils, it is important to consider the soil group when studying temporal variation (Khiari 2014). In France, the COMIFER (2009) reported seasonal pH variations of 0.5, 0.7, and 1.0 for textural groups G1, G2, and G3, respectively. These wide variations are due to the biological activity, which tends to lower soil pH, and to heavy rainfall, which tends to raise it (Van Der Paauw 1962). Fertility indicator values may increase during one season and decrease the next one and vice versa (Lockman and Molloy 1984; Cameron et al. 1994). These temporal variations are often intra- and inter-annual and depend on cropping practices and soil management methods (Dai et al. 2011). This situation makes the agronomic and environmental diagnosis of soils difficult and challenges the recommendations for optimal rates of fertilizers and amendments, as in Québec recommendations are based solely on soil tests (CRAAQ 2010).

For the most commonly used soil chemical tests, namely pH in water ( $\text{pH}_W$ ), buffer pH ( $\text{pH}_{\text{SMP}}$ ), OM (organic matter), predicted cation exchange capacity ( $\text{CEC}_{\text{predicted}}$ ), and Mehlich-3 (P, K, Al, Ca, Mg, B, Cu, Zn, Mn, Fe, and SPS), Khiari (2014) reported that the temporal variation of these indicators is the most important factor in soil characterization in Québec. Khiari (2014) also found that the best sampling period in Québec is in spring as soon as the soils are dewatered. This is because most of the research related to fertilization and agri-environmental practices conducted in Québec has considered sampling in spring. However, this short spring period offers little flexibility since it also coincides with the start of intense work on the farm and in the fields. Most often, sampling is done in the summer or fall. Seldom do farmers or their advisors keep a sampling logbook that specifies the sampling date. Rather, their choice of period depends on arbitrary factors and the time available to the samplers or farmers (Khiari 2014).

Because the current choice of sampling time does not consider the temporal representativity of the collected samples, we hypothesized that sampling at other time periods of the year may show soil test results similar to spring sampling. The objective of this research is to use

time-series decomposition and autocorrelation to determine representative sampling time windows for different chemical soil tests in Québec.

## Materials and Methods

### Soil sampling

Two agricultural fields, labeled 38C and 47, were monitored. These two fields are in the region of La Pocatière, Québec, Canada ( $47^{\circ}21'21''\text{N}$ ,  $70^{\circ}01'45''\text{W}$  and  $47^{\circ}20'37''\text{N}$ ,  $70^{\circ}00'52''\text{W}$ ), and have a fine texture, noted G1 (38C: Kamouraska clay loam) and a medium texture, noted G2 (47: Saint André sandy loam). In this study, field 38C (2.1 ha) was seeded to spring wheat in a binary rotation with soybeans under a conventional 0.15 m deep fall tillage and spring harrowing. Yield levels were  $2.4\text{--}3.2\text{ Mg}\cdot\text{ha}^{-1}$  for wheat and  $1.5\text{--}2.2\text{ Mg}\cdot\text{ha}^{-1}$  for soybeans. This 38C field received only mineral fertilization of about 75 kg diammonium phosphate (DAP) and a 100 kg mixture of 80% ammonium nitrate and 20% calcium carbonate (CAN) at seeding (i.e.,  $40.5\text{ kg N}\cdot\text{ha}^{-1}$  and  $15\text{ kg P}\cdot\text{ha}^{-1}$ ) and  $48.6\text{--}59.4\text{ kg N}\cdot\text{ha}^{-1}$  as CAN at tillering. Field 47 (1.9 ha), on the other hand, was dedicated to research to test cultivars of different crops. It received crops of quinoa, barley, corn, and wheat from 2009 to 2012 under conventional 0.20 m deep fall tillage. During this period, field 47 received  $15\text{ m}^3$  of sheep manure in the fall of 2008 and  $23\text{ m}^3$  of swine manure in postemergence of corn (2011). As this field is highly concentrated in P and K, mineral fertilization was limited to a single application of nitrogen in postemergence. No liming was done during the duration of the sampling.

For both fields, soil samples were collected once a week for 33 weeks a year, from mid-April to early December during four years from 2009 to 2012. Since it is neither useful nor practical to collect soils while they are covered with snow, a 19 wk period from early December to mid-April was excluded from the weekly sampling. For each of the two fields, three equidistant soil locations, noted a, b, and c, were sampled along the middle of the field. Each location was clearly identified with flags that remained installed during the four years. Each flag identified the center of a five-meter radius circle. Consequently, 792 samples were collected ( $2\text{ sites} \times 3\text{ locations (a, b, and c)} \times 33\text{ weeks} \times 4\text{ years}$ ). For each location a, b, and c, five sub-samples were taken randomly by a 7 mm diameter Pro-Sonde probe (Khiari et al. 2014) at a 20 cm depth within the sampling circle to form a composite sample of 30 to 40 grams. Precautions were taken to avoid trampling the soil during soil sampling by using wooden planks suspended above the sampling area.

### Soil sample testing

Soil samples were tested for  $\text{pH}_W$  and  $\text{pH}_{\text{SMP}}$  according to the electrometric method of the CEAEQ (2003a). Soil texture was determined by the hydrometer method (Day 1965). The OM content was determined using the

loss on ignition method (CEAEQ 2003b). The soil samples were also tested for their contents in P, K, Ca, Mg, B, Cu, Zn, Fe, Mn and Al extractable at the Mehlich-3 common extractive (Mehlich 1984; CEAEQ 2014). These 10 elements were measured by plasma emission spectrophotometry (Varian model 725-ES ICP-OES, Torch Type: Radial; Australia Pty Ltd.). The cation exchange capacity was estimated by the following equation (CRAAQ 2010):

$$(1) \quad CEC_{estimated} (cmol_{[+]}kg^{-1}) = 9[7.5 - pH_{SMP}] + [K + Ca + Mg]_{M-III} (cmol_{[+]}kg^{-1})$$

Soil phosphorus saturation index (SPS) was calculated by (Khiari et al. 2000):

$$(2) \quad SPS_{\%} = \left[ \frac{P_{mgkg^{-1}}}{Al_{mgkg^{-1}}} \right]_{M-III}$$

Time-series analysis

Most series were complete, but for some properties, less than 1% of the observations were missing. To have complete time series, these missing values were interpolated by the `na.interp` function of the `imputeTS` package of the R software (Martitz 2021). The temporal variability of soil properties was expressed as the standard deviation ratio (SDR) and compared with the acceptable quality level for reproducibility (AQLR) of soil sample analyses. The AQLR is defined by the CEAEQ (2015) for analytical quality control of the accredited laboratories in Québec. When a soil test was not on the CEAEQ (2015) list, we used the AQLR of NAPT (2001). Of the 90-temporal series (2 sites  $\times$  3 localities  $\times$  15 fertility indicators), those with an  $SDR < AQLR$  were not considered problematic, that is, whatever the sampling time, the sample is considered representative. Time series with  $SDR > AQLR$  were decomposed using loess STL of the `stlplus` package from R software (Hafen 2016). Loess (STL) is an algorithm that has been developed to separate a time series into three components: seasonality, trend, and remainder. Of these three components, only seasonality was used to analyze periods of regularity and irregularity in soil testing reflecting the distribution of observations depending on the sampling period. As for the interannual trend, it only serves to detect whether soil tests may change within a four-year sampling cycle, after correcting for seasonality over the period 2009 to 2012. The third remaining component is a noise signal obtained after eliminating the two effects of seasonality and inter-annual trend and represents the random fluctuations. For the series with an  $SDR > AQLR$ , we did an autocorrelation analysis using the `acf` function of the R forecast package (Hyndman et al. 2021). The autocorrelogram shows the similarity between observations with different time lags. For the seasonal components with  $SDR > AQLR$ , we defined a lower limit of validity, noted  $LL_{validity}$  (eq. 3) and an upper limit of validity,

noted  $UL_{validity}$  (eq. 4). The AQLR was applied to a central value most representative of the soil tests in Québec, that is, the average calculated over the five weeks (W) of spring (S), noted  $\bar{X}_{5ws}$  (eq. 3 and Fig. 1).

$$(3) \quad LL_{validity}(X) = \bar{X}_{5ws}(1 - AQLR)$$

$$(4) \quad UL_{validity}(X) = \bar{X}_{5ws}(1 + AQLR)$$

These two limits allow the inclusion or exclusion of sampling periods. The average  $\bar{X}_{5ws}$  is taken over five weeks since all the fertilization grids and the agronomic and environmental critical thresholds developed in Québec have been designed considering a spring sampling from the third week of April to the end of May.

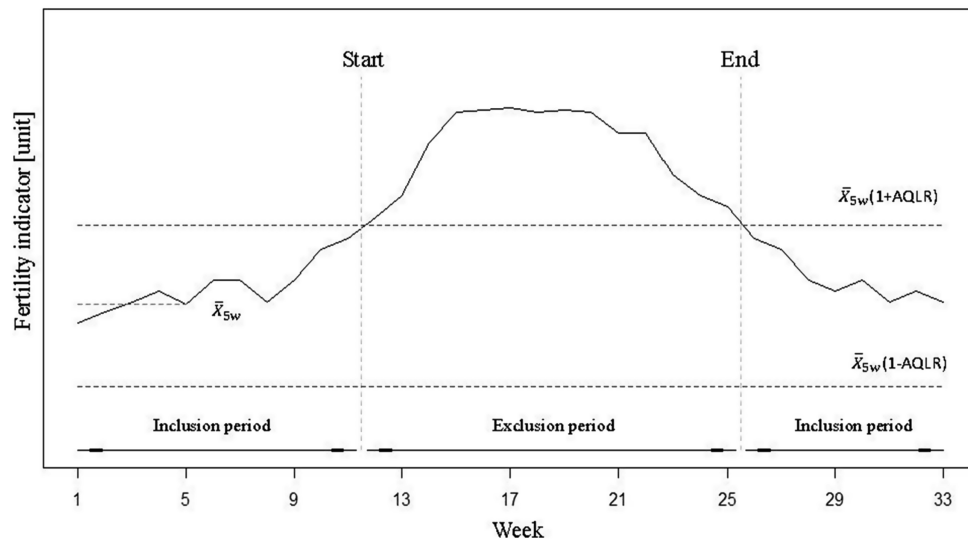
Fertility indicator values within the inclusion periods are considered stable and representative of spring sampling, those outside the inclusion periods are not representative of spring sampling and should be excluded from the sampling window. Thus, the exclusion period defined in Fig. 1 is a continuous range of weeks not recommended for soil sampling to limit the effect of summer on soil test values. Before using the results from the seasonality of soil tests, it is first necessary to make sure that these effects are present and can be considered. For one or two successive exceedance values, the phenomenon is considered sporadic and does not require the sampling windows to be modified. When seasonal or calendar effects cannot be identified in a time series, the time series is assumed to be deseasonalized (no seasonal effect) even if its SDR exceeds the AQLR. Whether sporadic or nonexistent ( $SDR < AQLR$ ), the series is free of a sampling calendar effect and no further analysis is required since samples can be taken any time from spring to fall.

## Results and Discussion

### Descriptive statistics of indicators

The summary results of SDR (Table 1) show that the overall seasonal variability of the 15 fertility indicators is large and ranges from 1.58% for  $pH_{SMP}$  to 54.97% for  $Cu_{M3}$ . As observed by Díaz-Ravifia et al. (1993), seasonal variations are more important for some. The five indicators  $pH_W$ ,  $pH_{SMP}$ ,  $P_{M3}$ , SPS, and  $Cu_{M3}$  showed much higher SDRs for the G1 soil than for the G2 soil. Clay appears to contribute to the high random and temporal variability of acidity, phosphorus, and some micronutrients, mainly  $Cu_{M3}$ . Turpault et al. (2008) explained this by the large specific surface areas of clay minerals to react with other soil elements to cause substantial changes over time. Also, the seasonal variability of OM and major nutrients (P, K, Ca, Mg) with SDRs of 9% to 24% is less than that of micronutrients with SDRs of 14% to 55%. Moreover, the two systems NAPT (2001) and CEAEQ (2015) allow more variation on micronutrients (10%–20%) than the other soil tests (5%–15%).

**Fig. 1.** Example of theoretical variation of an indicator bounded by the validity interval.  $\bar{x}_{5w}$  is the average of first five weeks (W) of spring. Acceptable quality level for reproducibility (AQLR) of soil sample analyses.



**Table 1.** Standard deviation ratio (SDR) of 15 fertility indicators for two soil types (G1 is fine texture; G2 is medium texture) and three locations (a, b, and c), compared with the acceptable quality level for reproducibility (AQLR) of two soil testing laboratory control programs for weekly sampling (from mid-April to early December) during four consecutive years.

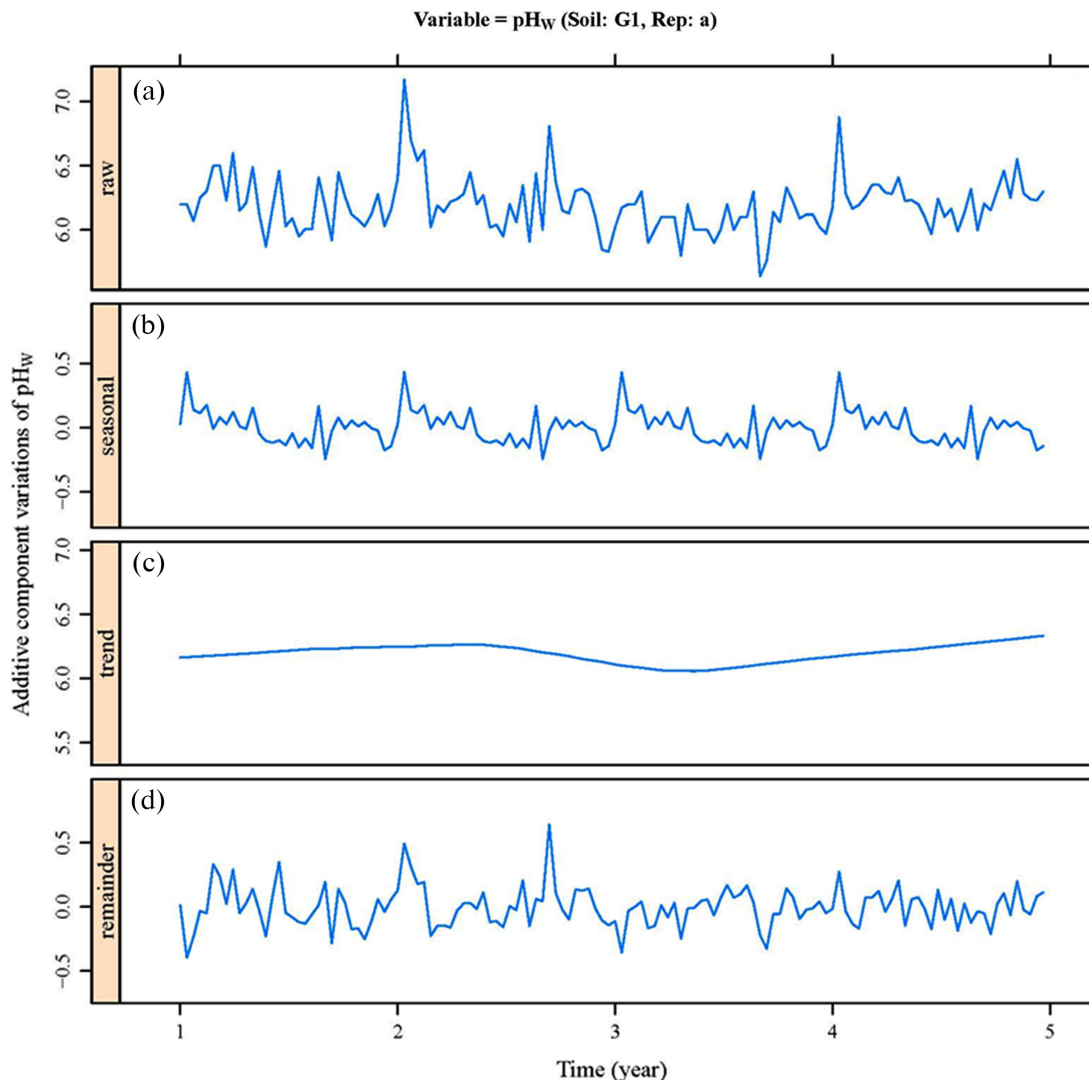
Soil test	Unit	AQLR (%)		SDR (%)					
		NAPT (2001)	CEAEQ (2015)	G1			G2		
				a	b	c	a	b	c
pH <sub>W</sub>	pH	0.8–1.2	3–4	3.6	4.2	4.2	2.4	2.8	3.0
pH <sub>SMP</sub>	pH	0.8–1.2	3	2.9	3.0	2.7	1.6	2.0	0.9
OM	g·kg <sup>-1</sup>	5–10	10	11	11	12	9	10	10
P <sub>M3</sub>	mg·kg <sup>-1</sup>	5–10	10	26	24	27	13	14	15
K <sub>M3</sub>	mg·kg <sup>-1</sup>	5–10	13	11	12	13	17	18	18
Ca <sub>M3</sub>	mg·kg <sup>-1</sup>	10–15	10	10	10	11	11	10	10
Mg <sub>M3</sub>	mg·kg <sup>-1</sup>	10–15	10–15	10	11	11	12	13	13
Al <sub>M3</sub>	mg·kg <sup>-1</sup>	—	10	8	10	10	9	9	9
SPS	%	—	—	30	29	33	12	12	13
CEC	cmol·kg <sup>-1</sup>	6–12	—	9	9	9	9	9	8
B <sub>M3</sub>	mg·kg <sup>-1</sup>	—	20	15	16	18	14	15	16
Mn <sub>M3</sub>	mg·kg <sup>-1</sup>	10–15	15–18	23	22	22	26	26	28
Cu <sub>M3</sub>	mg·kg <sup>-1</sup>	10–15	15–18	55	55	47	23	24	25
Zn <sub>M3</sub>	mg·kg <sup>-1</sup>	10–15	—	13	13	14	15	17	15
Fe <sub>M3</sub>	mg·kg <sup>-1</sup>	10–15	—	20	17	17	21	24	23

**Note:** NAPT: North American Proficiency Testing; CEAEQ, Centre d'expertise en analyse environnementale du Québec; OM, organic matter; M3, Mehlich-3 extraction method; SPS, soil phosphorus saturation index; CEC, cation exchange capacity.

Table 1 also shows that SDR values vary very little for a given field for all 15 fertility indicators. Even for Cu<sub>M3</sub>, the large SDR values are very close to one other with values of 55%, 55%, and 48%. Therefore, for these two fields, spatial heterogeneity does not seem to interfere with the temporal heterogeneity. For the two soil types, G1 and G2, only indicators pH<sub>SMP</sub>, Ca<sub>M3</sub>, Mg<sub>M3</sub>, Al<sub>M3</sub>, CEC<sub>predicted</sub>, and B<sub>M3</sub> are within the limits of the accepted

variation criteria of CEAEQ (2015). The other indicators, pH<sub>W</sub>, P<sub>M3</sub>, SPS, Mn<sub>M3</sub>, Cu<sub>M3</sub>, Zn<sub>M3</sub>, Fe<sub>M3</sub>, OM, and K<sub>M3</sub>, vary more and are above the CEAEQ (2015) limit for G1 and G2. For the soil acidity status, the SDR range of pH<sub>W</sub> is 2.4%–4.2%, a variation of 0.2–0.3 pH units, significantly lower than 0.8 units of the temporal variation reported by Colins et al. (1970). For phosphorus status, the SDR range is 13%–27% for P<sub>M3</sub> and 12%–33% for SPS,



**Fig. 2.** Additive decomposition of  $\text{pH}_W$  time series for soil G1 and replicate a. [Colour online.]

which upper limits are comparable to the 23% temporal SDR value for bioavailable phosphorus reported by Nyborg et al. (1992). The indicator  $\text{Cu}_{M3}$  has the highest SDR in the range of 23%–55%, with an upper limit is comparable to the temporal SDR of 43.8% for bioavailable Cu reported by Cameron et al. (1994). To define the optimal sampling range, only the latter indicators are considered. Consequently, nine of the 15 fertility indicators were analyzed with time series.

#### Seasonal variability of indicators

Figure 2 presents the time series of  $\text{pH}_W$ , for soil G1 and location a, and the additive components of seasonal, trend, and remainder. This seasonal component is representative of the three locations of field G1 (Table 2). During the four years, there is clear seasonal variation with four relatively high  $\text{pH}_W$  peaks, all corresponding to the same period in early spring (Fig. 2b). In mid-summer, between July and August, there is a  $\text{pH}_W$  trough

explained by increased biological activity. These seasonal variations in  $\text{pH}_W$  are related to temperature and humidity, which are periodic in nature. The results of this analysis show that the average  $\text{pH}_W$  does not show a clear increasing or decreasing inter-annual trend during the four years of study (Fig. 2c).

A total of 54 time series were analyzed, that is, the nine indicators selected from the previous section  $\times$  two soil types (G1 and G2)  $\times$  three locations (a, b, and c). These nine indicators were first assigned to one of three utility groups: (i) acidity and liming management indicator (pH); (ii) soil nutrient indicators other than phosphorus ( $\text{K}_{M3}$ , MO,  $\text{Mn}_{M3}$ ,  $\text{Zn}_{M3}$ ,  $\text{Cu}_{M3}$ , and  $\text{Fe}_{M3}$ ); and (iii) agro-environmental phosphorus management indicators  $\text{P}_{M3}$  and SPS.

For each utility group, only one indicator is presented to illustrate its seasonal variation. The three representative variables are  $\text{pH}_W$ ,  $\text{K}_{M3}$ , SPS and will be discussed in the following sections. For the other six

**Table 2.** Averages of fertility indicators, their types (Continuous, Sporadic, or Inexistent), their intervals of exceedance of validity limits, and their autocorrelation periods in the fine textured (G1) and medium textured (G2) soils.

Analysis	Rep	Average		Type of exceedance		Interval of exceedance (week)		ACF (week)	
		G1	G2	G1	G2	G1	G2	G1	G2
pH <sub>W</sub>	a	6.2	6.8	Continuous	Inexistent	14–23	NA	<b>2</b>	NA
	b	6.2	6.8			13–23		2	
	c	6.2	6.8			13–23		2	
OM (%)	a	4.6	7.7	Sporadic	Inexistent	NA	NA	NA	NA
	b	4.6	7.8						
	c	4.5	7.8						
P <sub>M3</sub> (mg·kg <sup>-1</sup> )	a	122	620	Continuous	Continuous	4–19	10–20	3	4
	b	123	610			5–19	9–23	4	4
	c	121	614			5–19	8–20	4	4
K <sub>M3</sub> (mg·kg <sup>-1</sup> )	a	589	636	Inexistent	Sporadic	NA	NA	NA	NA
	b	575	614						
	c	577	646						
SPS (%)	a	5.6	27.9	Continuous	Continuous	4–19	13–20	<b>5</b>	3
	b	5.8	27.8			4–19	14–20	4	4
	c	5.7	27.9			5–19	8–20	3	4
Mn <sub>M3</sub> (mg·kg <sup>-1</sup> )	a	58	91	Sporadic	Sporadic	NA	NA	NA	NA
	b	57	89						
	c	59	90						
Cu <sub>M3</sub> (mg·kg <sup>-1</sup> )	a	1.4	3.3	Continuous	Sporadic	11–24	NA	11	NA
	b	1.5	3.4			11–23		8	
	c	1.4	3.5			11–24		10	
Zn <sub>M3</sub> (mg·kg <sup>-1</sup> )	a	3.4	6.6	Inexistent	Sporadic	NA	NA	NA	NA
	b	3.4	6.6						
	c	3.4	6.5						
Fe <sub>M3</sub> (mg·kg <sup>-1</sup> )	a	406	240	Inexistent	Sporadic	NA	NA	NA	NA
	b	394	238						
	c	398	243						

**Note:** Inexistent ( $SDR \leq AQLR$ , see Table 1), so the time series is considered deseasonalized. Sporadic, if the lower validity limit ( $LL_{\text{validity}}$ ) or the upper validity limit ( $UL_{\text{validity}}$ ) are exceeded by only one or two successive weeks, so the time series is considered deseasonalized. Continuous, if  $LL_{\text{validity}}$  and  $UL_{\text{validity}}$  are exceeded for at least 3 successive weeks, so the time series is seasonalized (a seasonal effect is detected). ACF, autocorrelation of fertility indicators expressed in weeks; NA, not applicable if type of exceedance is Inexistent or Sporadic (deseasonalized). Values in bold are as shown on Fig. 3–4 for pH, K<sub>M3</sub>, and SPS, respectively.

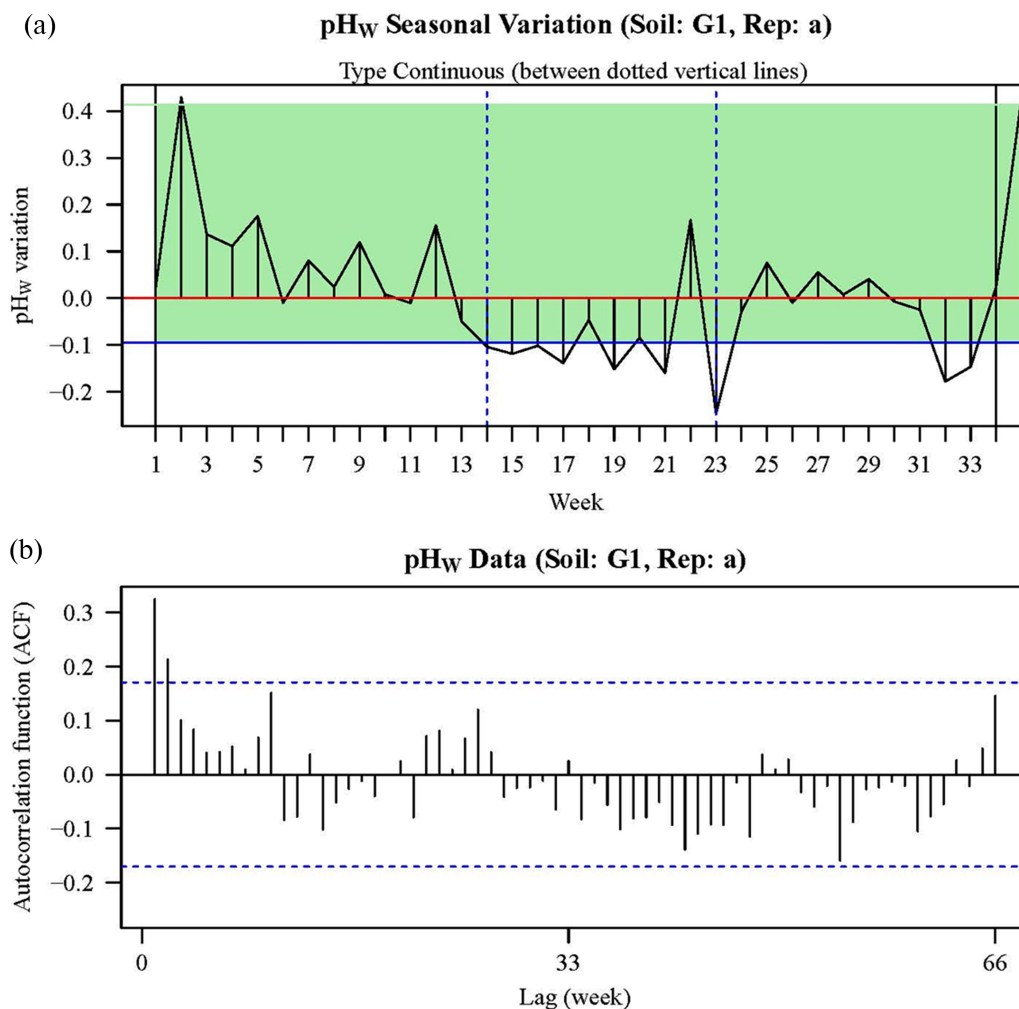
indicators (P<sub>M3</sub>, OM, Mn<sub>M3</sub>, Zn<sub>M3</sub>, Cu<sub>M3</sub>, and Fe<sub>M3</sub>), the resulting observations are summarized in Table 2. Analyses of the weekly data show a gradual change in the indicator values, but with occasional sharp fluctuations, as shown in Fig. 3 for pH<sub>W</sub>, in Fig. 4 for K<sub>M3</sub>, and in Fig. 5 for SPS. The seasonal component is the baseline on which a validity interval for each indicator is applied, as explained in Fig. 1. The trend and remainder series are not required for this study.

#### Variability of acidity diagnostic and liming management indicators

When considering the NAPT (2001) criteria of AQLR, both indicators (pH<sub>W</sub> and pH<sub>SMP</sub>) would have unacceptable

variations for both soil types G1 and G2 (Table 1). The NAPT (2001) system is significantly more stringent for the diagnosis of acidity, with pH tolerances of 0.8%–1.2%, compared with the CEAEQ (2015) system, which has three times greater tolerances: 3.0%–4.0% of pH unit (Table 1). The NAPT (2001) system provides greater accuracy for soil quality control. Therefore, there would be less risk of misinterpretation of active acidity (pH<sub>W</sub>), and especially of lime requirements, based on pH<sub>SMP</sub>. However, our results are discussed in relation to the criteria of CEAEQ (2015) because it is mandatory in Québec. Based on CEAEQ (2015) criteria, the pH<sub>SMP</sub> does not raise any problem of temporal variability for either G1 or G2 soil.

**Fig. 3.** Time series showing a seasonal pattern of  $\text{pH}_W$  variation (upper part) over a 33 wk cycle per year, followed by the autocorrelation function (lower part) of  $\text{pH}_W$  (correlation versus lag). [Colour online.]

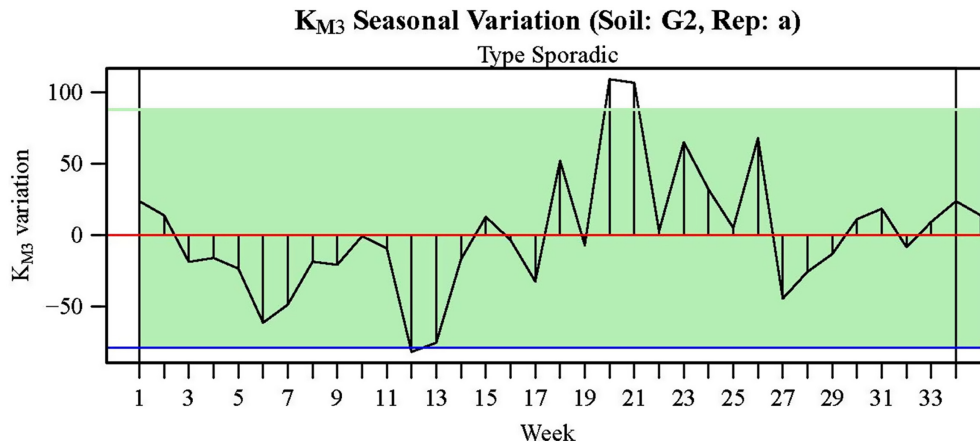


However, Lemire et al. (2005) raise the problem of accuracy in the  $\text{pH}_{\text{SMP}}$  measurement method, whose accepted tolerance in Québec is already high, that is,  $\pm 0.2 \text{ pH}_{\text{SMP}}$  units, which would result in an error of estimated lime requirements of  $\pm 2.5 \text{ t}\cdot\text{ha}^{-1}$ . In its official website, the MAPAQ (2021) mentions that this error is unpredictable and difficult to consider when making lime recommendations. On the other hand, for  $\text{pH}_W$ , the CEAEQ (2015) system detects variations that exceed the AQLR limit (Table 1). As shown in Fig. 3 and Table 2,  $\text{pH}_W$  decreases below the AQLR limit in the period from weeks 14 and 23, i.e., from July to October. Other studies, such as those by Collins et al. (1970) and Hoskinson et al. (1999), also obtained the same trends of significant decreases in soil  $\text{pH}_W$  in the summer to early fall. The range of nonacceptable  $\text{pH}_W$  variation is therefore an average of 10 consecutive weeks, starting in July for G1 soil (Table 2). It is therefore a type of continuous exceedance of the AQLR limit. This drop in  $\text{pH}_W$  values below the AQLR limit is likely due to climatic conditions during the summer and early fall. This 10-week period is the least suitable for sampling because low  $\text{pH}_W$

values will lead to poor diagnosis and poor decision-making regarding liming actions. On the other hand, fall and early spring are the best times to sample. Their variations are both similar, nonsignificant and within the acceptable fluctuation according to criteria of CEAEQ (2015). This is consistent with the interpretation grids of soil acidity obtained in Québec from liming tests (Tran and Van Lierop 1982) where sampling was carried out during this period of the year. Since  $\text{pH}_W$  is the most widely used acidity indicator, it must necessarily be associated with a period for sampling, that is, before July, or in the fall, starting in October. Figure 3 also shows the ACF autocorrelation function reflecting the degree of similarity in  $\text{pH}_W$  between consecutive weekly samples. Exceeding the critical limits, that is, the two dotted horizontal lines in Fig. 3, derived from the ACF function, shows autocorrelations significantly different from zero ( $p < 0.05$ ) only for lags of one and two consecutive weeks. In other words, regardless of the sampling period, the statistical similarity of  $\text{pH}_W$  over time is only maintained for a maximum time lag of two weeks. This low autocorrelation



**Fig. 4.** Time series showing a seasonal pattern of potassium Mehlich-3 ( $K_{M3}$ ) variation ( $\text{mg}\cdot\text{kg}^{-1}$ ) over a 33-week cycle per year. [Colour online.]



is due to the logarithmic pH scale, which decreases the amplitude of the variation in active acidity and makes repeated measurements over time less autocorrelated. For the other two locations (b and c), the pattern is the same as that in Fig. 3 (not shown, but the main observations are summarized in Table 2) and the autocorrelation is also  $\leq 2$ . Despite the low reliability of  $\text{pH}_W$  and  $\text{pH}_{\text{SMP}}$ , they are still the only investigative and diagnostic tools for the acidity of agricultural soils in Québec, Canada, and in several U.S. states. Since the 1980s, Follett and Follett (1980) have suggested adding other indicators such as soil texture, clay type, organic matter content, exchangeable aluminum, and cation exchange capacity to better diagnose acidic soils and assess their lime requirement more accurately.

#### Variation in diagnostic indicators of soil nutrients

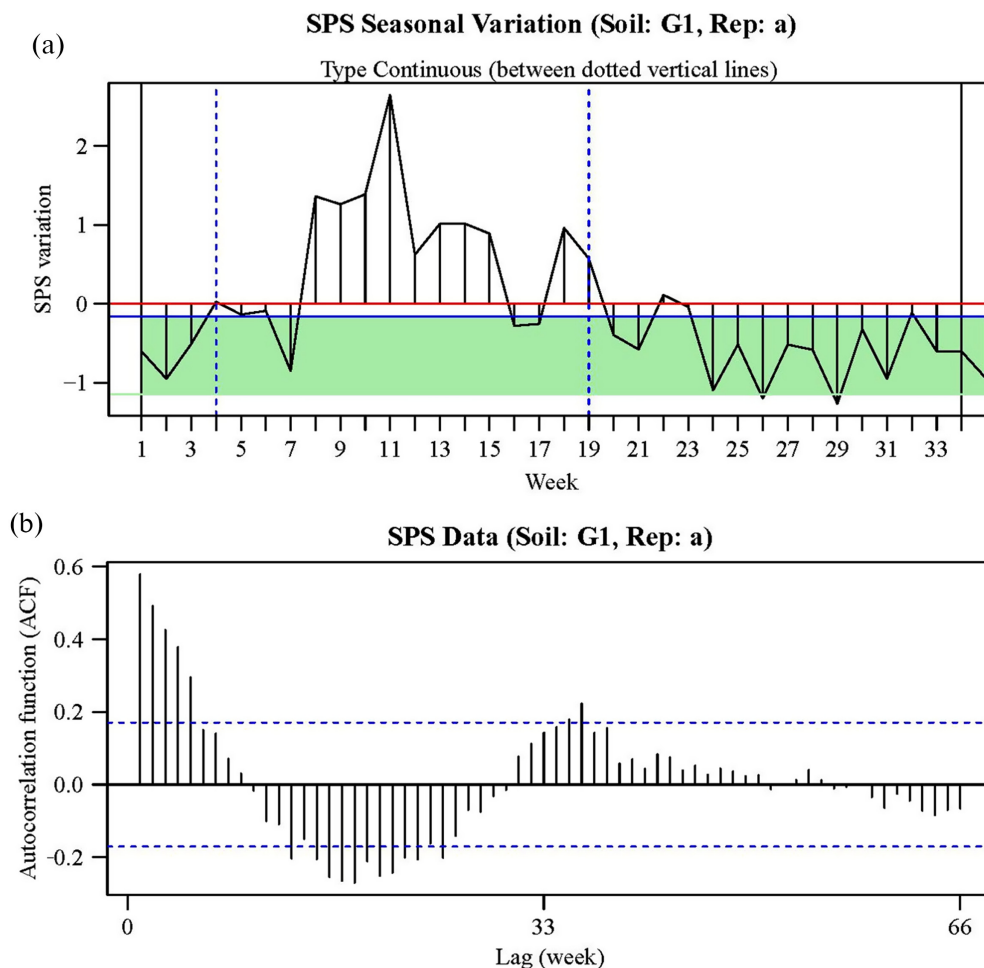
Five soil fertility indicators ( $\text{Ca}_{M3}$ ,  $\text{Mg}_{M3}$ ,  $\text{Al}_{M3}$ ,  $\text{CEC}_{\text{predicted}}$ ,  $\text{B}_{M3}$ ) were not affected by seasonality since their SDRs were  $\leq$  AQLRs (Table 1). However, six indicators ( $K_{M3}$ , OM,  $\text{Mn}_{M3}$ ,  $\text{Zn}_{M3}$ ,  $\text{Cu}_{M3}$ ,  $\text{Fe}_{M3}$ ) had a  $\text{SDR} \geq$  AQLR for G1 and (or) G2 soils. A representative case is shown in Fig. 4 for  $K_{M3}$  in soil G2 at location a. Result summary is shown in Table 2 for all six indicators, for both soils G1 and G2 and for all three locations (a, b, and c). Figure 4 shows a range of variation of  $K_{M3}$  that is not acceptable or below the AQLR limit because of three values, one isolated at week 12 and two at week 20 and 21. This variation of  $100 \text{ mg K}\cdot\text{kg}^{-1}$  between week 12 (summer) and weeks 20 and 21 (early autumn) is very important. The causes and mechanisms leading to this situation remain unclear due to the complex environment of the crops (Hoskinson et al. 1999). For this group of indicators, only  $\text{Cu}_{M3}$  showed a significant and continuous exceedance in the fine textured soil, G1, compared with the average of the five weeks of spring (Table 2). However, this  $\text{Cu}_{M3}$ , which has the greatest seasonal variation with SDRs of 47%–55% (Table 1), also shows that it is the least concentrated element, at

$1.4\text{--}1.5 \text{ mg Cu}\cdot\text{kg}^{-1}$ . These extremely low values make estimates of the concentration of  $\text{Cu}_{M3}$  usually less reliable than the measurement of the other indicators. Therefore, none of the five indicators, pre-examined by descriptive statistics ( $\text{Ca}_{M3}$ ,  $\text{Mg}_{M3}$ ,  $\text{Al}_{M3}$ , CEC, and  $\text{B}_{M3}$ ), and none of the six indicators examined by time-series decomposition ( $K_{M3}$ , OM,  $\text{Mn}_{M3}$ ,  $\text{Zn}_{M3}$ ,  $\text{Cu}_{M3}$ , and  $\text{Fe}_{M3}$ ) should be subject to a representative sampling calendar effect.

#### Variability of agro-environmental diagnostic indicators

The two indicators,  $\text{P}_{M3}$  and SPS, are used to prescribe phosphorus rates (CRAAQ 2010) and to prevent pollution and eutrophication of surrounding surface waters (MDDELCC 2017). The average contents of  $\text{P}_{M3}$  available phosphorus for soils G1 and G2 are  $122$  and  $617 \text{ mg P}\cdot\text{kg}^{-1}$ , respectively, and those  $\bar{X}_{5\text{ws}}$  are  $110$  and  $571 \text{ mg P}\cdot\text{kg}^{-1}$ , respectively (Table 2). The two latter averages indicate soils with high phosphate fertility, even for P-demanding crops, since they exceed the agronomic critical thresholds of  $90 \text{ mg P}_{M3}\cdot\text{kg}^{-1}$  (CRAAQ 2010; Khiari et al. 2000). The SPS averages of the G1 and G2 soils are, respectively, 5.7% and 27.9%, and the  $\bar{X}_{5\text{ws}}$  values are 5.0 and 26.6%. These  $\bar{X}_{5\text{ws}}$  values show that the fine-textured G1 soil is clearly below the environmental critical thresholds of 7.6% (MDDELCC 2017) and 8% (CRAAQ 2010). However, soil G2 is supersaturated in phosphorus and greatly exceeds the environmental critical threshold of 11% (CRAAQ 2010). As an example, the seasonal component of the SPS time series is shown in Fig. 5 for soil G1 and location a. Results are shown in Table 2 for both indicators, the two soils and the three locations. Figure 5 shows quasi-periodic oscillations characterized by alternating periods of SPS stability within the lower  $\text{LL}_{\text{validity}}$  and upper  $\text{UL}_{\text{validity}}$  limits (eqs. 3 and 4) and periods of increases in SPS above  $\text{UL}_{\text{validity}}$  (above the blue line). In Fig. 5, SPS is characterized by a long period of 16 weeks, between week 4 and 19, where the SPS values exceed  $\text{UL}_{\text{validity}}$ . These high SPS values cause an

**Fig. 5.** Time series showing a seasonal pattern of soil phosphorus saturation (SPS) variation (*a*; upper figure) over a 33 wk cycle per year, followed by the autocorrelation function (*b*; lower figure) of SPS (correlation versus lag). [Colour online.]



environmental risk of soil P saturation (CRAAQ 2010) going from a lower medium risk class ( $4.0 \leq \text{SPS} < 6.5$ ) in spring, to a higher medium risk class ( $6.5 \leq \text{SPS} < 8.0$ ) in summer for 30% of the samples, and up to a high-risk class ( $8.0 \leq \text{SPS} < 14$ ) for 14% of the samples. When compared with the critical value of 7.6%, above which the regulation (MDDELCC 2017) considers the risk of diffusion of P to surface water to be high, this results in an 18% exceedance. To select sampling windows for collecting representative data, period of high risk must be avoided. The twelve graphs of the agro-environmental indicators: two indicators (SPS and  $P_{M3}$ )  $\times$  two soils (G1 and G2)  $\times$  three locations (a, b, and c) showed patterns of seasonality similar to those of Fig. 5. For field G1, the six seasonal time series resulted in nonrepresentative sampling windows between weeks 4 and 19 (Table 2). In Québec (CRAAQ 2010) sampling should be done in the spring (weeks 0 to 5). Outside this interval, SPS is likely to vary considerably with an upward trend up to week 19 and then a return to the spring values during the fall (Fig. 5). During summer, soils warm up, which stimulate the biological activity and make phosphorous more

available (Habibiandekordi et al. 2015). For the G1 field, spring SPS values of 5.4 (Table 2) are not problematic and can increase by 2.5 % by week 11 (Fig. 5) and exceeds the environmental threshold of 7.6 %, established by the regulation (MDDELCC 2017) for soils with a clay content  $>30\%$ . For SPS values above the 7.6% threshold, the phosphate fertilization strategy must aim at reducing soil saturation below the threshold. A sample taken during the wrong time period may under-estimate phosphate fertilization amounts. An over-estimation of SPS would falsely reduce the areas available for manure application, and force farmers to rent land to dispose of their manure, resulting in extra time and costs. For the G2 medium textured field, SPS of almost 28% above the environmental threshold of 13% (MDDELCC 2017). In Table 2, when comparing the intervals of exceedance for  $P_{M3}$  and SPS, we observe an early seasonal variation starting in week 4 or 5 for G1 soil. But for G2 soil, it is shifted between weeks 8 and 14. Since the G2 soil is much more saturated with phosphorus than the G1 soil, this could explain such a delay for the seasonal effect of  $P_{M3}$  and SSP. Fig. 5b shows significant autocorrelations

for five consecutive weeks. Therefore, regardless of the sampling period, the statistical similarity of SPS across time is only guaranteed for a maximum of 5 wk. From one sampling cycle to another, it is therefore more consistent and representative to always sample at the same time within 5 weeks of each other. Locations b and c yielded a pattern similar to that of Fig. 5b (not shown), and almost the same 3–5 wk intervals where the SPS measures are significantly autocorrelated (Table 2).

## Conclusion and Recommendations

This study, although limited to two sites in Québec, shows the usefulness of analyzing the variability and temporal autocorrelation of soil indicators to obtain a better representativity of soil sampling periods for characterizing agro-environmental diagnosis indicators. The main conclusions are as follows: (i) descriptive statistics showed that among the 15 agro-environmental diagnostic indicators,  $\text{pH}_W$ , OM, and Mehlich-3 (P, K, Mn, Cu, Zn, Fe and SPS) varied significantly and were above the limits of the variation allowed by CEAEQ (2015); (ii) time variation of these nine indicators were decomposed to extract the seasonal component that showed a clear effect for only three soil indicators ( $\text{pH}_W$ ,  $P_{M3}$ , and SPS). For these three indicators, the temporal representativity is ensured only when a window of 12–15 successive weeks of summer is excluded. Therefore, for these three indicators, sampling should be done either during the first five weeks of spring or in early September. Finally, autocorrelation graphs showed that time series of  $\text{pH}_W$ ,  $P_{M3}$ , and SPS are not random, but show a temporal persistence of up to four weeks. Therefore, regardless of the sampling period initially chosen, soil sampling be done within a four-week window.

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