Independent Quality Control of Fertilizing Residuals by Environment Québec

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ABSTRACT

An independent quality control done by the *ministère de l'Environnement* (MENV) demonstrates that all of the fertilizing residuals (FR) sampled in 2000 and 2001 respected the maximum limits for contaminant and pathogen content. The determination of category based on chemical contaminants (category C1 or C2) by the promoter was reliable or conservative in 96% of the cases. The determination of category based on pathogen levels (category P1, P2, or P3) by the promoters was reliable or conservative in at least 83% of the cases. In 17% of the cases, the samples taken by the MENV showed a P2 category, while the promoters claimed that their product was P1. This implies a possible underestimation of risk by the promoters. However, the pathogen exceedances beyond the P1 criteria for *E. coli* were relatively low, and the numbers measured were well below those typically found in manure. The fertilizing element content alleged by the promoters were also generally reliable for the purpose of determining agro-environmental spreading rates. However, the use of a complementary nitrogen fertilizer indicated to minimise the risk of over or under fertilization with certain FRs.

KEYWORDS

Biosolids, nitrogen, metals, pathogens, phosphorus, independant quality control

INTRODUCTION

Close to one million tons of fertilizing residuals (FR) are spread annually on the agricultural soils of Québec (Charbonneau et al., 2000). These are mainly comprised of paper mill, abattoir or municipal biosolids (sludge), cement kiln dust and wood ash.

In order to spread these FRs the promoters must generally obtain a certificate of authorization (CA) from the *ministère de l'Environnement* (MENV), except for commercial products that are certified by the *Bureau de normalisation du Québec* (BNQ) and used according to the instructions. Approximately ten percent of the FR tonnage applied in agriculture is certified by the BNQ. Other FR reclamation activities that are low risk may also be exempt from the CA requirement, such as the spreading of tree pruning debris in limited quantities (MENV, 2002).

When a promoter requests a CA from the MENV, he must supply laboratory analysis results indicating levels of contaminants, pathogens and fertilizing elements for the FR in question over the last 12 months. The average of these values are then compared to the levels permitted (MENV, 2002) to determine the quality category for chemical contaminants such as heavy metals (category C1 or C2) and pathogens (category P1, P2, or P3). The levels permitted for the C categories are derived mainly from the compost quality criteria of the CCME (1996). The criteria for the P categories are derived mainly from the American regulations for municipal biosolids (USEPA, 1993). The sampling and analyses methods, as well as the mandatory sampling frequency are established by the MENV (2002). The analyses must be performed by a laboratory that has been accredited by the *Centre d'expertise en analyse environnementale du Québec* (CEAEQ). The residual is also classified based on its odour (category O1, O2, or O3) by the MENV (2002). A residual that does not meet the base requirements (class C2-P3-O3) is considered "out of category" and cannot be spread on agricultural soils. To avoid over fertilization, which could lead to water contamination, the maximal spreading quantities are determined by the level of fertilizing elements, particularly nitrogen and phosphorus

The FR quality classification by the MENV supposes the following hypothesis:

- The analysis results and resulting classification provided by the promoter are reliable;
- The residuals that will be delivered to the farms in the near future (a few weeks to a few months) have levels of fertilizing elements and contaminants similar to the average value of the past 12 months.

The validity of these hypothesis implies the respects of the following conditions:

- The variability over time for contaminants and fertilizing elements is limited, for a given fertilizing element
- The sampling done by the promoter is adequate and not biased;
- The samples are analysed by reliable laboratories that have been accredited by the CEAEQ.

To ensure that the environmental criteria are respected, the regional offices of the MENV must regularly perform independent quality controls for the FR quality (MENV, 2002). A large scale control was carried out in 2000 and 2001 for most of the administrative regions of Québec. The specific objectives of this control were the following:

- Representatively sample of all the FR reclaimed under a CA;
- Determine if the C and P categories and levels of fertilizing elements alleged by the promoter in the CA application are reliable;
- Document, if applicable, certain sources of variation between the results observed by the MENV and those of the promoter.

A detailed analysis of the results of this quality control were the subject of an essay, done in the course of a masters degree in environment at the *Université de Sherbrooke* (Rioux, 2002). This article presents the main results that relate to the representativity of the sampling and the overall classification of FR, while giving more details on the results of the C and P categories and the levels of fertilizing elements.

MATERIALS AND METHODS

The sampling campaign was carried out under the supervision of the *Direction des politiques du secteur agricole* in collaboration with the participating regional offices. Each regional office selected at least two FRs representative of their administrative region. The sampling was done partly in 2000 and mainly in the summer of 2001. Employees of the MENV did the sampling, either at the plant or in the field (stored heaps), or both. The samples at the plant were generally taken at a single moment in time, rather than over an extended period. The sampled were sent to the CEAEQ for analysis. For certain FR, a sub-sample was sent to the promoter for analysis by his own laboratories.

Based on the results from the CEAEQ, the C and P categories were determined for each MENV sample, as described in the *Critères provisoires pour la valorisation des matières résiduelles fertilisantes* (CPVMRF) (MENV, 2002). We compared these to the categories alleged by the promoters in their CA applications, which are based on their own laboratory analysis results.

The results for each chemical, microbiological or agronomic parameters were analysed in greater detail in order to explore some of the sources of variation. To do this we used dispersion diagrams coupled with a linear regression analysis, using Microsoft Excel (Microsoft Corporation, 1997).

Analysis results beneath the detection limits were attributed half the detection limit value, except for dioxins and furans, for whom values of zero toxic equivalent (TEQ) were attributed on the analysis certificate from the CEAEQ. For more details on sampling, analysis, and data manipulations, refer to Rioux (2002).

Odour categories were not determined in this study.

RESULTS AND DISCUSSION

Representativity of the FR sampled

Twenty four FR and three compost were sampled by the MENV (Table 1). This represents approximately 30% of the 85 FR reclaimed in Québec under a CA (Charbonneau et al., 2000). Paper mill biosolids account for 62.5% of the samples, and correspond fairly well to the 70% paper mill biosolids that are spread in agriculture (by weight). Municipal biosolids are over represented, whereas abattoir biosolids and other agri-food residuals are under represented.

Table 1 Representativity of FR sampled in relation to all of the FR reclaimed in agriculture

Type of FR	Number sampled by the MENV in 2000-01	%	Number of FR indexed in 1999 (1)	%	Quantity spread in 1999 (1) t/year (wet weight)	%
Paper mill biosolids (2)	15	62.5	33	39	576 886	70.2
Municipal biosolids (3)	6	25	11	13	56 260	6.9
Abattoir biosolids and wastes	2	8.3	8	9.4	>30 789(4)	3.7
Other agri-food biosolids and residuals	0	0	4	4.7	19 567	2.4
Ash	0	0	16	18.8	45 457	5.5
Liming amendments(5)	0	0	1	1.2	14 059	1.7
Cement kiln dust	0	0	1	1.2	40 000	4.9
Other liming amendments (6)	1	4.2	4	4.7	5 617	0.68
Other FR	0	0	7	8	32 483	4
Total	24	100	85	100	821 118	100

(1) From Charbonneau *et al.* (2000)
(2) Including primary biosolids alone, secondary, or mixed.
(3) Including municipal biosolids that have been dehydrated or limed, as well as septic tank biosolids, alone or mixed with abattoir biosolids
(4) Underestimated
(5) Magnesium residuals (in French = residus magnésiens)
(6) Includes lime mud from paper mills and quick lime

Ashes are not represented at all, as no samples were taken. However, these residuals do not contain any pathogens, and are therefore considered to be P1 by default (MENV, 2001). The representation for other liming amendments is also almost nil. However, six of these residuals are currently certified by the BNQ, which necessarily means that they have undergone an independent quality control.

The three composts sampled do not appear on Table 1, because their representativity is difficult to establish due to a lack of data on the quantities reclaimed in agriculture (Charbonneau et al 2000).

In terms of geography, 11 regional offices participated in the project. This represents 85% of the administrative regions where there is significant agricultural reclamation. With the exception of ashes, the sampling is therefore fairly representative of the FRs reclaimed in agriculture in Québec under a CA.

Overall classification

C and P categories were established for 24 FR, including three composts (Figure 1). All of the FR sampled respected the basic requirements (class C2-P3): none were “out of category”. The class of excellent environmental quality (C1-P1) includes close to 30% of the FR sampled. Moreover, more than half of the FR sampled were in the C1 category, including 80% of the paper mill biosolids. This is quite similar to the findings of Charbonneau *et al.* (2001).

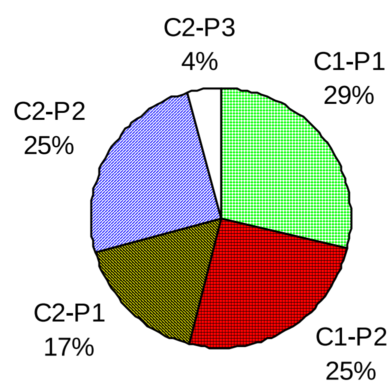

Figure 1 Classification of 24 fertilizing residuals sampled in Québec

Chemical contaminant (C) categories

In 20 cases out of 27 (74%), the C categories determined following MENV sampling correspond exactly to the category alleged by the promoter. In 5 cases out of 27, the MENV results indicated a C1 category while the promoter alleged a C2 category in the CA application.

For the paper mill biosolid #6, the sample taken at the mill indicated a C1 category, while the sample taken in the field indicated C2. This difference was due to the level of cadmium (5 mg/kg) and molybdenum (7 mg/kg). However, the category is C1 when both samples are averaged. We therefore consider this biosolid to be in the C1 category, as the categories are based on the average or median value of many samples (MENV, 2002) and the levels of cadmium and molybdenum are well below the C2 limits (10 mg Cd/kg and 20 mg Mo/kg).

According to the sampling done in 2000 and 2001, the C categories alleged by the promoters are therefore reliable or conservative in 96% of the cases (23/24). However, in the case of municipal biosolid #1, the risk was probably under estimated: the MENV sampled indicated a C2 category, while the promoter alleged a C1 category. However, the levels of arsenic (14 mg/kg) and selenium (2.8 mg/kg) responsible for classification difference, barely exceed the C1 limit (13 mg As/kg, and 2 mg Se/kg) and are 5 times less than the maximal limits permitted for the C2 category (75 mg As/Kg and 14 mg Se1/kg). Furthermore, for statistical reasons, we cannot definitely affirm that the municipal biosolid #1 surpasses the C1 criteria, because only one sample was taken and the exceedence of the C1 criteria is relatively low. Moreover, in 3 of 12 cases where the MENV took samples both at the plant and in the field, the results were divergent, which underlines the fact that there is a certain variability due to the sampling or the process which generates the residuals

Cadmium and selenium are the main elements responsible for a category difference between the promoter and the MENV, or between the two samples taken by the MENV. This difference could be due to variations over time of the contaminant levels for a given FR caused by, for example, differences in primary materials or processes. The data provided by the promoters for the CA (not published) show a variation over time for certain parameters. Another source of variation is attributable to the sampling method. However, this was not evaluated in this study.

A third source of variation is attributable to the laboratory. However, the regression analysis (Table 2) generally shows a good fit between the results of the promoter and the CEAEQ. Nickle has the best fit, arsenic the worst.

The number of samples is however too low to draw conclusions, notably because the regression may in certain cases be strongly influenced the a single outlying value. Moreover, in the case where a substance was not detected, arbitrarily giving a value of half the detection limit increases the variability for substances with low concentrations, such as selenium. In fact, the regression slope for selenium is negative. However, the variability due to very low concentrations does not have an impact on the accuracy of the C classification, and by extension on the environmental risk.

Table 2 Comparison between the values obtained by the promoters (x) and those obtained by the MENV (y) for a given sample

Parameters	n	Regression	r^2
Aluminium	4	y = 1.2 x - 62.1	0.99
Arsenic	6	y = -3.1 x + 1.8	0.04
Cadmium	6	y = 0.7 x + 1.3	0.28
Cobalt	6	y = 0.1 x + 1.2	0.34
Chrome	6	y = 0.5 x + 4.1	0.01
Copper	7	y = 0.9 x + 4.6	0.95
Iron	6	y = 1.0 x + 509.4	1.00
Mercury	6	y = 3.8 x + 0.0	0.70
Manganese	6	y = 1.7 x - 57.2	0.86
Molybdenum	6	y = 1.3 x - 0.4	0.81
Nickel	6	y = 1.0 x + 0.1	0.84
Lead	6	y = 4.3 x - 16.2	0.86
Selenium	6	y = -0.9 x + 0.7	0.30
Zinc	6	y = 0.3 x + 93.8	0.02
Dioxins and furans	4	y = 0.3 x + 0.3	0.25

Pathogen (P) categories

In 17 cases out of 23 (74%), the P categories alleged by the promoters and that observed by the MENV were identical (Table 3). In 2 of 23 cases, the MENV sampling indicated a P1 category (no spreading restriction based on pathogens) while the promoter alleged a P2 category (with spreading restrictions). Thus, the P category provided by the promoters was reliable or conservative on a risk basis in 19 of 23 cases (83%).

However, in four cases, the MENV sampling indicated P2 category, whereas the promoter alleged a P1 in the CA application. This is the case for paper mill biosolids #1, 5 and 12, as well as for compost #1. It is possible that in these cases, the promoter under estimated the risk (4 of 23 cases, 17%). This is more that the 4 % non conformity observed in the C categories (chemical contaminants). In practise, these four P2 FR may have been spread as P1 FR in crops where P2 residuals are forbidden (e.g. potatoes), or too close to wells. However, these four cases must be examined in detail before drawing such conclusions.

For the paper mill biosolids #1 and #5, the average of the two samples taken by the MENV (in the field and at the mill) gave the P2 category, whereas one of the two samples respected the P1 pathogen limits (Table 4). For the paper mill biosolids #12 and compost #1, the measured values of 4100 and 6400 MPN *E. coli* /g (d.w.), respectively, were higher than the P1 limit (1000 *E. coli*/g (d.w.)), but 400 times lower than the P2 limit (2 000 000 *E. coli*/g d.w.). The exceedance of the P1 criteria is thus relatively low.

Table 3 Conformity of the P_x classification alleged by the promoter in the CA application and those obtained by the MENV.

FR	P_x according to the CA	P_x according to the MENV sampling (at the plant and in the field)			
		At the plant	Divergent parameter	In the field	Divergent parameter
Paper mill biosolids					
# 1	P1	***P2***	***E. coli***	***P2***	***Salmonella***
# 2	P2	**P1**		**P1**	
# 3	P2	P2		**P1**	
# 5	P1	P1		***P2***	***E. coli***
# 6	P2	P2		P2	
# 7	P1	P1		---	
# 8	P1	P1		---	
# 9	P1	P1		---	
# 10	P1	P1		---	
# 11	P1	P1		---	
# 12	P1	***P2***	***E. coli***	---	
# 13	P2	P2		P2	
# 14	P1	P1		P1	
# 15	P1	P1		---	
Municipal biosolids					
# 1	P2	---		P2	
# 2	P2	P2		P2	
# 4	P2	P2		---	
# 5	P1	---		P1	
# 6	P2	---		P2	
Abattoir biosolids					
#1	P2	P2		P2	
# 2	P2	P2		---	
Composts					
# 1	P1	***P2***	***E. coli***	**---**	
# 2	P1	P1		---	

Note : The bold text highlights the samples where the P category obtained by the MENV differed from that alleged in the CA. The data that is both bold and italicised shows the cases where the environmental risk was probably underestimated by the promoter.

Table 4 Comparison between the P criteria for *E. coli* and *Salmonella* and the level in certain FR and farm residuals.

	E. coli **(MPN/ g dry weight)(1)**	***Salmonella*** **(MPN / 4 g dry weight)(1)**
P1 criteria	< 1000	< 3
P2 criteria (2)	< 2 000 000(2)	
Paper mill biosolid #1	18 (in field); > 5000 (at mill)2,3	3
Paper mill biosolid #5	296 (in field); > 5300 (at mill) 2,3	<1,5
Paper mill biosolid #12	4100	< 1,0
Compost #1	6400	< 1,5
Cattle manure (n=5)(4)	64 000; (min=235; max =285 000)	Detected in 100% of the cases
Liquid hog manure (n=6) (4)	1.5×10^7 ; (min=5×10^5; max =5×10^7)	Detected in 67% of the cases

(1) MPN: Most probable number
(2) The P2 limit for *E. coli* does not apply to paper mill biosolids that are reputed to be non contaminated by human fecal matter, as is the case for paper mill biosolids #1, 5 and 12.
(3) The actual *E. coli* content is not determined because the upper quantification limit was too low.
(4) n : number of sample analysed.

Moreover, if we comparewith the spreading of solid and liquid manures, the risk from FR spreading seems relatively low for the following reasons:

- Three of these FR do not contain *Salmonella*, as opposed to cattle and hog manure (Table 4).
- The *E. coli* content of the four FR in question are relatively low compared to that of solid and liquid manures (Table 4)
- According to Gauthier and Archibald (2001), among the *E. coli* analyzed as pathogen indicators in paper mill biosolids, no pathenogenic strains were observed, whereas it is well known that the pathenogenic strain O157:H7 may be present in cattle manure.
- The *E. coli* measured in the paper mill biosolids may be due to microbial regrowth (Chantal J. Beauchamp, personal communication). A high level of *E. coli* is not necessarily correlated with a significant fecal contamination of the FR.
- Even if the *E. coli* were shown to be indicators of fecal contamination, the spreading distance relative to wells (> 30 m for P1 residuals) would give a level of protection similar to that required for untreated solid and liquid manures when the FR was spread.
- For compost #1, the oxygen consumption rate of 50 mg O_2/kg organic matter/hour indicates a very mature compost (P1 criteria < 500 O_2/kg o.m./h). This suggests that an intensive composting occurred which theoretically brings about the destruction of pathogenic and other bacteria

Thus we can conclude that the P categories alleged by the promoter are conservative in at least 83% of the cases, and in 0 to 17% of the cases, the risk was possibly underestimated by the promoter. Nevertheless, the health risks implied by this classification error appears to be relatively low, compared to the risks from spreading of solid and liquid manures. However, following the present sampling campaign, the MENV decided on a preventative basis to re-categorize the paper mill biosolids #1 from P1 to P2. In the other cases where the P classification did not conform, the classification will be re-evaluated on a case by case basis by the regional offices.

Data was incomplete for four FR: paper mill biosolid #4, municipal biosolid #3 and compost #3. Consequently, they were not placed into P categories and do not appear in Table 3. This is because the upper quantification limit for *E. coli* was too low, and the detection limit too high for *Salmonella* in the case of certain samples containing less than 25% dry matter. The lime muds were not tested for pathogens as they are unlikely to be contaminated due to their mineral and alkaline nature.

Overall for paper mill biosolids, 64% (9 of 14) of the samples taken at the mill were P1, and 36% (5 of 14) were P2. This follows the study of Charbonneau et al. (2001) who states that the majority of paper mill biosolids were P1.

Agronomic parameters

Table 5 shows the main results for linear regression analyses for agronomic parameters. The comparison is made between the average value of the promoter in the CA application, on the X axis (theoretical value), and the sample taken at the mill by the MENV, following issuance of the CA, on the Y axis (observed value).

The dry matter (dryness) is an important parameter for calculating the spreading rate and thus respecting the agronomic recommendations based on the level of nitrogen and phosphorus or on the limits for trace elements (C2 residuals). The regression analysis shows a very strong coherence between the values given by the promoters and the MENV when we consider the r^2 value and the slope of the regression, which are close to one, and the ordinate which is close to 0 (Figure 2). This indicates that the values given by the promoters are reliable and that the dryness of the FRs vary little through time: the batches sampled by the MENV were produced after those analyzed for the CA application.

The agronomic spreading rate is often determined as a function of nitrogen needs, as this is the nutrient that has the most influence on plant production. In order to meet the plant needs and avoid excessive application, the agronomist must evaluate the nitrogen availability in the FR. This availability, or efficiency, is estimated as a function of the total nitrogen, the C/N ratio and the ammonia (NH_4). The data of the promoter and that observed the MENV are very coherent for the C/N ratio (Table 5). The relation is less good for total nitrogen, but is improved overall when the comparisons are made on a wet weight basis (Figure 3). It is even better if we retain only the paper mill biosolids on a wet weight basis ($y=1.04x + 308$, $r^2=0.79$). The linear relation is non existent for ammonia, even though the levels are

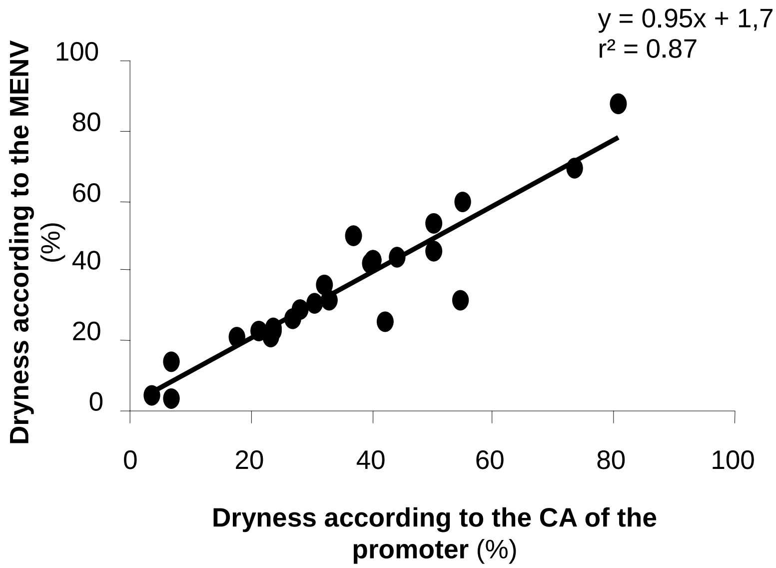

Figure 2 Dispersion diagram for dry matter (dryness) of fertilizing residuals

Table 5 Results of the regression analysis for agronomic parameters in the comparative analysis of data from the promoter (x) and those obtained by the MENV (y).

Parameters	n (1) =	Regression	r^2
Dry matter (w.w.)(2)	25	y = 0.95x + 1.7	0.87
N total (mg/kg, d.w.)(2)	24	y = 0.79x + 6851	0.66
N total (mg/kg, w.w.)	24	y = 0.91x + 935	0.67
N- NH_4 (mg/kg, d.w.)	24	y = 0.61x + 1947	0.09
P_2O_5 (mg/kg, d.w.)	25	y = 1.00x + 4141	0.57
P_2O_5 (mg/kg, w.w.)	25	y = 1.18x + 468	0.70
K_2O (mg/kg, d.w.)	25	y = 0.31x + 1010	0.30
Organic matter (%, d.w.)	23	y = 1.06x – 3.0	0.88
C/N	17	y = 0.69x + 5.0	0.89
C/N recalculated (2)	23	y = 0.77x + 2.4	0.91
pH	15	y = 0.57x + 3.3	0.44

(1) n = number of samples.
(2) w.w = wet weight; d.w. = dry weight
(3) C/N of the promoter recalculated based on the values for organic matter and total nitrogen written in the CA application.

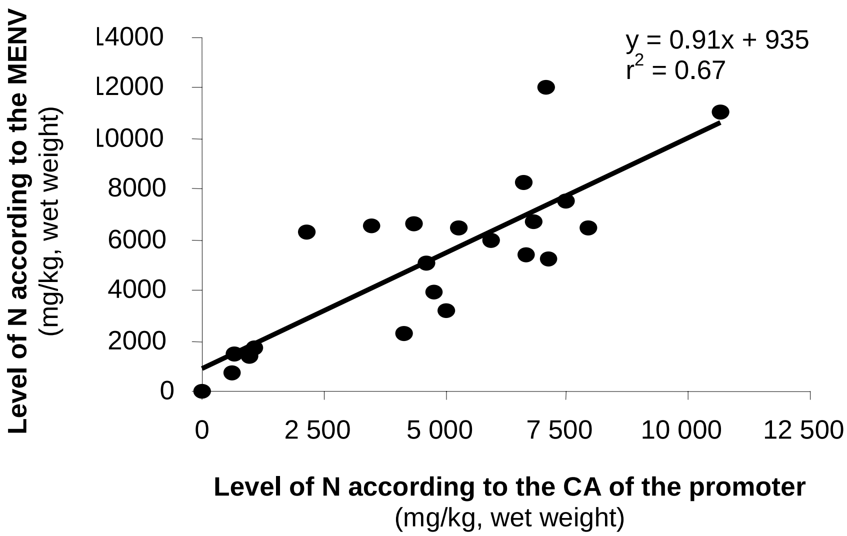

Figure 3 Dispersion diagram of total nitrogen in fertilizing residuals

relatively low. It is known that the level of ammonia can increase during storage following microbial activity, especially if the temperature in the heap remains high, which favours ammonification, and if the storage is prolonged (many months). The analysis of ammonia at the mill therefore does not give a valid result in this case.

In order to limit the impacts related to an under-estimation or overestimation of the availability of nitrogen, it is recommended that an important part of the needs of the plant (≥ 25%) be met by a mineral fertilizer that has a more predictable nitrogen content and availability. Otherwise, it is important for each promoter to analyses each FR lot, and base the agronomic recommendation on each lot, rather than on the 12 month average of many lots. Inversely, certain FRs may have a level of total N and ammonia that is more stable over time and thus do not require corrective action.

Phosphorus (P or P_2O_5) is also a major fertilizing element and often limits the permissible spreading rates. An excessive fertilization over many years provokes an accumulation of phosphorus in the soil and increases the risk of desorbtion from the soil and runoff into surface waters, causing eutrophisation and the consequent effects on aquatic fauna and human health (e.g. algae blooms). Analysing the data on a wet weight basis gives a fairly good relation, when we take into account the r^2 value and the slope of the regression, which are close to one, and the ordinate which is close to 0 (Figure 4).

Nevertheless, there may be an underestimation of the actual phosphorus content in many cases, which could cause over fertilization problems. However, over the long term, the over fertilization of one year may be compensated by the under fertilization of another year (where the level of P was over estimated). As for total nitrogen, the variability of total phosphorus may be due in part to the variability of lots over time. For example, the modification at the mill of the ratio of primary to secondary biosolids, for the case of mixed biosolids.

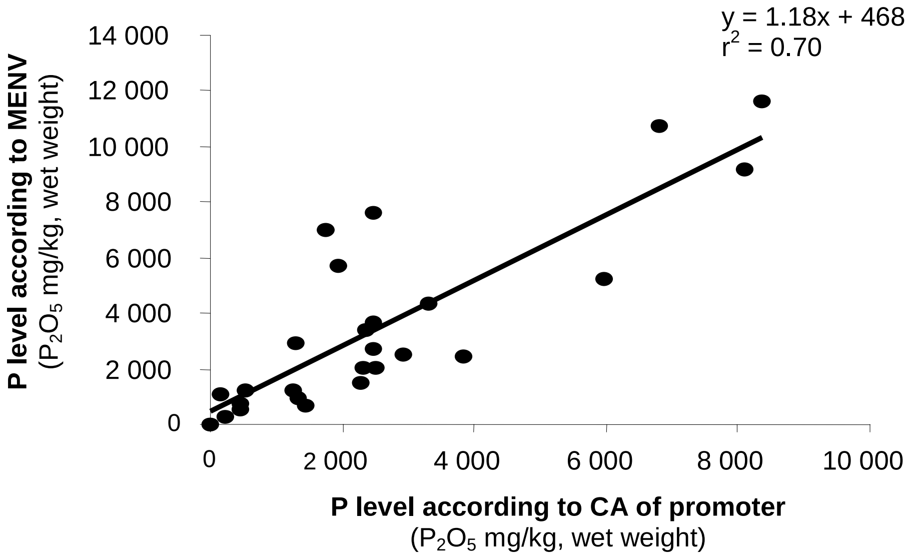

Figure 4 Dispersion diagram of total phosphorus in fertilizing residuals

As for potassium (K or K_2O), the third major element in fertilization, the linear relationship is weak (Table 5). This could be due to the low K levels in biosolids and their large variability over time, as potassium is soluble and often lost during the pressing of the sludges. On an environmental basis, there is no consequence to over or under estimating K, because it is generally not considered a contaminant. On an agronomic basis, the impacts of an over or under estimation are generally limited with biosolids as their levels are ofen very low. For liming amendments, such as ash and cement kiln dust, which have higher levels of K, an accurate assessment becomes more important in order to make a good agronomic recommendation.

As for organic matter, which permits the amelioration of the physical and chemical properties and biological activity of soils, the data from the MENV samples are very coherent with those given by the promoters (Table 5).

The pH indicates the acidic or basic character of a FR when it is spread on the soil. The relation between the values alleged by the promoters and the values observed by the MENV is only partially linear ($r^2 = 0.44$). However, the impact of an over or under estimation of the pH of a FR is not usually problematic for vegetable production. In fact, the quantities of FR used represent in most cases less than 1% of the soil mass in the cultivable zone. Moreover, it is the neutralising or acidifying power of the FR, and not the pH that will have the most influence on the soil. The pH has en environmental significance if the residual is being limed or acidified for pathogen or odour reduction. Retaining only those FR with a pH > 10 (as stated in the CA application), which presumably have been limed, the coherence between the data of the promoter and the MENV is low in two of three cases. However, the *E. coli* counts were very low, which suggest that the liming done by the promoter was in fact effective.

Generally, the variability observed for agronomic parameters, between the results of the MENV and the CA application, are not due to inter-laboratory variation. When the values obtained by the private laboratories chosen by the promoters are compared with those of the CEAEQ, for the same FR samples, the results are very coherent (Table 6). We can therefore consider that the laboratories used by the promoters give reliable results for the agronomic parameters.

Table 6 Regression analysis results for the agronomic parameters in a comparative analysis of the results obtained by the promoters (x) and those obtained by the MENV (y) for a same sample.

Parameters	n =	Regression	r^2
Dry matter (%)	7	y = 0.98x + 0.9	0.96
N total (mg/kg, d.w.)	7	y = 0.91x + 390	0.95
$N-NH_4$ (mg/kg, d.w.)	7	y = 0.82x + 120	0.96
P_2O_5 (mg/kg, d.w.)	7	y = 0.72x + 3740	0.97
K_2O (mg/kg, d.w.)	7	y = 1.12x + 154	0.88
Organic matter (mg/kg, d.w.)	7	y = 0.98x + 2.4	1.00
C/N	7	y = 1.0x + 1.2	1.00
pH	5	y = 0.98x + 0.2	0.98

We also compared, for 10 FR (mainly paper mill biosolids), the impact for sampling at the mill as compared to sampling in the heaps stored in the field (Table 7).

Table 7 Results of the regression analysis for agronomic parameters in a comparative analysis of data obtained from a sample taken at the mill (x) and that obtained from a field sample (y).

Parameters	n =	Regression	r^2
Dry matter (%)	11	y = 0.84x + 3.5	0.89
N total (mg/kg, d.w.)	10	y = 0.96x + 307	0.79
$N-NH_4$ (mg/kg, d.w.)	10	y = 0.79x + 3316	0.41
P_2O_5 (mg/kg, d.w.)	11	y = 1.2x – 2506	0.96
K_2O (mg/kg, d.w.)	11	y = 1.3x – 181	0.92
Organic matter (%, d.w.)	10	y = 0.98x – 2.6	0.91
C/N	10	y = 0.64x + 5.4	0.96
Neutralizing power (%, CCE)	6	y = 0.97x - 0.3	0.98
pH	8	y = 0.36x + 5.1	0.86

The results are generally very coherent, except for the case of ammonia and pH. This shows a variability that is generally limited between different lots for these 10 FR. This variability is lower that than which is noted in Table 4. For ammonia and pH, the difference between the two sample points (which represent different lots) may be explained partly by the biological activity. The micro-organisms mineralize the organic matter during storage, which generate acids, bases and NH_4. Thus, we can consider that, with exception, the sample point (field or mill) has little impact for agronomic parameters, as long as the sampling is representative.

CONCLUSIONS

This independent field control campaign of FR by the MENV has shown that all FR sampled in 2000 and 2001 respected the maximum limits for chemical contaminants and pathogens (C and P categories). The sampling was highly representative as it covered around 30% of the FR reclaimed under a CA in the majority of the administrative regions of Québec where FRs were reclaimed for agriculture. However, no ash was sampled.

The FR classification alleged by the promoter for the CA application was exact or conservative in 96% of the cases for chemical contaminants such as heavy metals and dioxins and furans (C1 and C2 categories). The analysis results for chemical contaminants provided by the promoters therefore seems to be conservative from an environmental point of view, even when we consider the numerous sources of possible variation.

The pathogen categories alleged by the promoter in the CA application were exact or conservative in at least 83% of the cases. In 17% of the cases, the samples taken by the MENV showed a P2 category, whereas the promoter alleged a P1 category. However, the exceedences of the P1 criteria for *E. coli* were low when we consider the normal variability of this parameter. Moreover, the content was well below that which is found in solid and liquid manures, which implies a low risk when compared to farm manures.

As for methodology, because of the high variability associated with *E. coli* analyses, the independent quality control should involve a minimum of two distinct composite samples. The promoters must ensure that the analyses are performed by laboratories accredited by the CEAEQ for the *E. coli* and *Salmonella* analyses.

For agronomic parameters such as nitrogen and phosphorus, the values given by the promoters, which are derived from the 12 month sampling period preceding the CA application, are reliable for predicting the fertilizing value for a FR that will be spread *a posteriori*. However, in the case of nitrogen, a complementary mineral fertilization of at least 25% of the needs of the plant is recommended, for certain FR, in order to lessen the agronomic or environmental impact of the temporal variability of nitrogen and the incertitude of mineralization rate of organic soil nitrogen.

The results of agronomic parameters for a given FR sample shows that the laboratories used by the promoters are reliable. This is not surprising as the MENV requires that the laboratories be accredited by the CEAEQ, when the accreditation domain exists. Finally, the sampling point (at the mill or in the field) generally has little impact on agronomic parameters, except those influenced by microbial activity such as pH and ammonia.

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