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**ASSESSMENT OF THE AGRICULTURAL VALUE  
OF SUGAR REFINERY BY-PRODUCTS**

by  
Luc Massicotte

A thesis submitted to the faculty of Graduated Studies  
and Research in partial fulfilment of the  
requirements for the degree of the  
Master of Science

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## ABSTRACT

M.Sc.

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Natural  
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The sugar refinery process used by Lantic Sugar Ltd generates three by-products having characteristics that give them potential as soil amendments or fertilizers, particularly as a phosphorous and calcium source. Laboratory and a field trials were conducted in order to examine the changes in agronomic properties of soil produced by the application of these residues.

During the laboratory experiment, the by-products examined were spend bone char (SBC), filter-press mud (FPM), clarification scum (SCU) and a compost (COM) produced using FPM and SCU, where as in a field experiment, COM, SBC and a mixture (MIX) made of FPM and SCU, were compared to a commercial fertilizer (TSP) and non-treated soils.

The laboratory experiment revealed that the use of by-products causes changes in the concentrations of extractable nutrients and pH, although soil type influences such a behaviour. SCU, SBC and COM, in that order, significantly increased extractable P and Ca in soils. FPM did not increase macro-elements in the soil, but rather stimulated enzymatic and microbial activity. A high level of P fixation was observed in soils treated with SCU and COM. All residues stimulated soil respiration and caused slight immobilization of soil mineral N. The study indicates that the residues, alone or in combination with other materials, have significant potential as soil amendments.

The orthic humic gleysol of clay texture and low pH soil conditions in which the field experiment was conducted resulted in high P fixation of all the applied residues. Contrasts analysis showed that TSP behaved as the soils unamended P for all nutrient concentrations in tissues over two cropping seasons (1993 and 1994), on two crops,

namely wheat (*Triticum aestivum*, L.) and corn (*Zea mays*, L.). Treatments (residues at different rates of application) did not significantly increase the Ca levels in COM plots nor did they increase the wet aggregate stability of soil under either crop.

Organic matter (OM) in both soils under both crops was increased by the use of MIX and COM. COM increased the OM content of the soil more efficiently than TSP. Two COM applications created net N immobilization in soil of corn plots and significantly increased N content in both corn tissues (grain and stover) over controls. MIX applied at three times the recommended  $P_2O_5$  rate significantly increased soil Mellich III P versus control plots. P concentration in corn stover decreased as opposed to that of treatment TSP applied at one fold the recommended rate for P, whereas the opposite was observed in wheat grain.

SBC applied at the recommended  $P_2O_5$  rate raised phosphorus concentration in the soil and the grain of corn over TSP at the end of 1993. A residual effect of P from SBC application appeared in soil, while its cumulative effect was superior to that of the control.

Extractable P was higher in some MIX over TSP treatments at the end of the second cropping season, indicating that the residue is less subject to fixation than soluble P fertilizer. These results suggests a review of the recommendations for phosphorus application rates under the actual conditions.

Due to the constraints during the field trials, the results for P uptake as well as N, P and K crop concentration should be used with reserve. The results demonstrated, however that the use of sugar refinery by-products, improves certain soil characteristics, particularly with respect to extractable P and OM.

## RÉSUMÉ

M.Sc.

Luc Massicotte

Natural  
Resource Sciences

Au cours du procédé de raffinage du sucre, Sucre Lantic ltée génère trois sous-produits dont les caractéristiques leur confèrent un potentiel comme amendement ou fertilisant des sols, particulièrement en tant que source phosphorée et calcique. Des essais en laboratoire et en champ ont été réalisés afin d'examiner les changements sur les propriétés agronomiques des sols suite à l'apport de ces résidus.

Les essais en laboratoire examinés ont été les poussières de noir animale (SBC), les boues de filtre-presse (FPM), les écumes de clarification (SCU) du procédé de raffinage, ainsi qu'un compost (COM) produit à partir des FPM et des SCU. Les essais au champ ont donné lieu à l'observation d'un mélange composé de FPM et de SCU, appelé MIX, du COM et des SBC, lesquels ont été comparés à un fertilisant commercial, le triple superphosphate (TSP), ainsi que des parcelles n'ayant reçu aucune source phosphorée.

Les expériences en laboratoire ont permis d'observer les changements qu'entraînent l'apport des résidus sur les concentrations en éléments nutritifs extractibles du sol et des variations de pH, en plus d'apprécier l'influence du type de sol sur ces comportements. FPM n'a pas permis de rehausser les macro-éléments du sol, mais a contribué à stimuler l'activité enzymatique et microbienne du sol. Un haut niveau de fixation du phosphore (P) a été enregistré dans les parcelles traitées au SCU et COM. Tous les résidus ont stimulé la respiration des sols et ont causé une légère immobilisation de l'azote minérale. L'étude a démontré que les résidus, appliqués seul ou en combinaison avec d'autres matériaux, possèdent un potentiel comme amendement des sols.

Les essais au champ se sont déroulés dans des sols de type gleysol orthique et humique à texture argileuse et à bas pH. Les résultats ont donné lieu à un haut niveau de fixation du P pour chacun des résidus appliqués au sol. L'analyse par contraste a démontré que

les parcelles traitées au TSP ne se sont pas démarquées significativement aux parcelles témoins (non-amendées) pour chacun des éléments nutritifs analysés au terme des deux années d'expérimentation, pour les deux cultures en présence, soit le blé (*Triticum aestivum*, L.) et le maïs (*Zea mays*, L.).

La teneur en matière organique (MO) du sol a été augmentée dans les cultures par l'apport de MIX et COM. La teneur en MO du sol a été augmenté de façon plus significative dans les sols amendés par COM par rapport aux sols ayant reçu du TSP. Une seconde application de COM a créé une immobilisation nette de N dans les sols pour les parcelles de maïs et a augmenté significativement la teneur de N dans les grains et les tiges de maïs. MIX appliqué au sol à un taux équivalent à trois fois le taux recommandé de  $P_2O_5$ , a rehaussé significativement la concentration en P Mellich III du sol par rapport aux parcelles témoins. La concentration en P dans les tiges de maïs a diminué par rapport à TSP1, alors que la situation inverse s'est produite dans les grains de blé.

SBC appliqué à une fois le taux en  $P_2O_5$ , a augmenté la concentration en P du sol et dans les grains de maïs versus TSP au terme de la saison 1993. L'effet résiduelle de P suite à l'apport de SBC appliqué au sol a été observé, alors que l'effet cumulatif est apparu supérieur aux parcelles non-amendées.

Le P extractible du sol a été plus élevé dans certains traitements de MIX que dans TSP à la fin de la saison de croissance 1994, ce qui indique que le sous-produit est moins sujet à la fixation que le fertilisant soluble. Ces résultats suggèrent qu'il y a lieu de revoir les recommandations relatifs aux taux d'application de P dans les conditions en présence.

En raison des contraintes vécues au cours des essais au champ, les résultats devront être utilisés avec réserves, particulièrement en ce qui a trait au P assimilé et aux concentrations de N, P et K analysés dans les tissus. Ces résultats ont démontrés, par

ailleurs que l'utilisation des sous-produits du raffinage du sucre améliore certaines caractéristiques du sol, particulièrement le P extractible et la MO.



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## TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT .....	i
RÉSUMÉ .....	iii
MANUSCRIPTS AND AUTHORSHIP .....	v
TABLE OF CONTENTS .....	vii
LIST OF TABLES .....	xi
LIST OF FIGURES .....	xiii
LIST OF APPENDIX TABLES .....	xiv
INTRODUCTION AND OBJECTIVES .....	1
<b>CHAPTER 1.</b>	
<b>LITERATURE REVIEW .....</b>	<b>3</b>
1.0 PHOSPHORUS AND SOIL NUTRIENT AVAILABILITY .....	
1.1 Phosphorus Transformation .....	4
1.2 Soil Phosphorus Availability .....	4
1.2.1 Soil Clay .....	5
1.2.2 Sorption, Desorption and P Precipitation .....	5
1.2.3 Soil pH and Added P .....	6
1.2.4 Soil P and OM .....	7
1.3 Soil P and its Relationship with Plant Growth .....	7
1.3.1 Plant P Uptake .....	7
1.3.2 P Source Determination .....	8
1.3.3 Soil P Tests .....	8
1.4 Soil Microbial Activity in Relation to Phosphorus and Organic Sources Application .....	9
1.4.1 Phosphatase Activity .....	9
1.4.2 Soil Respiration and CO <sub>2</sub> Evolution .....	10

## TABLE OF CONTENTS (con't)

	<u>Page</u>
2.0 COMPOST AS SOIL AMENDMENT . . . . .	11
3.0 LANTIC REFINING PROCESS AND BY-PRODUCTS GENERATED .	14
3.1 Lantic Refining Process . . . . .	14
3.2 Lantic By-products and their Potential as Soil Amendment or Fertilizer . . . . .	16
3.2.1 Spent Bone Char (SBC) . . . . .	16
3.2.2 Clarification Scum (SCU) . . . . .	17
3.2.3 Filter-Press Mud (FPM) . . . . .	17
REFERENCES . . . . .	19
 <b>CHAPTER 2.</b>	
<b>LABORATORY ASSESSMENT OF SUGAR REFINING BY-PRODUCTS ON AVAILABLE SOIL NUTRIENT, pH AND MICROBIAL ACTIVITY . . . .</b>	<b>28</b>
ABSTRACT . . . . .	29
RÉSUMÉ . . . . .	30
INTRODUCTION . . . . .	31
MATERIAL AND METHODS . . . . .	32
Series 1: Destructive soil sampling . . . . .	34
Series 2: CO <sub>2</sub> evolution . . . . .	36
Statistical analysis and calculations . . . . .	37

## TABLE OF CONTENTS (con't)

	<u>Page</u>
RESULTS . . . . .	37
Soil Chemical Properties . . . . .	37
Evolution of CO <sub>2</sub> . . . . .	40
Phosphatase Activity . . . . .	43
DISCUSSION . . . . .	43
Residues as phosphorus sources . . . . .	43
Residue effects on microbial activity . . . . .	46
Residue effects on soil cations and pH . . . . .	47
CONCLUSION . . . . .	48
REFERENCES . . . . .	49
CONNECTING SECTION . . . . .	52
<b>CHAPTER 3.</b>	
<b>AGRONOMIC VALUE OF SUGAR REFINING BY-PRODUCTS AND THEIR</b>	
<b>COMPOST ON ACID CLAY SOIL . . . . .</b>	<b>53</b>
ABSTRACT . . . . .	54
RÉSUMÉ . . . . .	56
INTRODUCTION . . . . .	58

## TABLE OF CONTENTS (con't)

	<u>Page</u>
MATERIAL AND METHODS . . . . .	65
Soil sampling and analysis. . . . .	67
Tissue sampling and analysis . . . . .	68
Statistical analysis . . . . .	69
RESULTS AND DISCUSSION . . . . .	70
Constraints on interpretation . . . . .	70
Soil physical and chemical characteristics . . . . .	71
Phosphorus . . . . .	75
Calcium and Magnesium . . . . .	75
Potassium . . . . .	79
Organic Matter . . . . .	79
Soil pH . . . . .	83
Nitrogen . . . . .	84
Wet Aggregate Stability . . . . .	86
Crop tissue results . . . . .	91
Nitrogen . . . . .	91
Phosphorus and Potassium . . . . .	92
P uptake . . . . .	93
CONCLUSION . . . . .	97
REFERENCES . . . . .	100
CHAPTER 4.	
GENERAL CONCLUSION AND RECOMMENDATIONS . . . . .	105

## LIST OF TABLES

	<u>Page</u>
 <b>CHAPTER 2</b>	
Table 1	Chemical analysis of sugar refining by-products and their compost (dry matter basis) . . . . . 33
Table 2	Some initial soil characteristics (0-20 cm) for Ste-Rosalie and Chicot . . . . . 35
Table 3	Significance of extractable P, K, Ca, Mg, NH <sub>4</sub> , NO <sub>3</sub> and pH for the destructive sampling analysis . . . . . 38
Table 4	Melhich III-extractable, P, K, Ca, Mg, KCl-extractable NH <sub>4</sub> , NO <sub>3</sub> and pH in extractant from two incubated soil types . . . . . 39
Table 5	Fixation of Melhich III extractable phosphorus after 27 days of soil incubation . . . . . 41
Table 6	Carbon added with residues and percentage of added C consumed for CO <sub>2</sub> production during the 27 days incubation in 100g of soil . . . . . 42
Table 7	Soil acid and alkaline phosphatase activity after 6, 13 and 27 days of incubation . . . . . 44
 <b>CHAPTER 3</b>	
Table 1	Chemical analysis of sugar refining by-products and their compost (dry matter basis) . . . . . 64
Table 2	Probability of treatment effects on soil moisture, P, K, Ca, Mg, NH <sub>4</sub> -N, NO <sub>3</sub> -N, OM, pH and wet aggregate stability of soil wheat plots (type III) . . . . . 72

	<b>LIST OF TABLES (cont'd)</b>	<u>Page</u>
Table 3	Probability of treatment effects on soil moisture, P, K, Ca, Mg, NH <sub>4</sub> -N, NO <sub>3</sub> -N, OM, pH and wet aggregate stability (type III) . . . . .	73
Table 4	Probability of treatment effects on wheat grain N, P, K, Yield and P uptake (type III) . . . . .	74
Table 5	Probability of treatment effects on corn grain N, P, K, Yield and P uptake (type III) . . . . .	76
Table 6	Probability of treatment effects on corn stover N, P, K, Yield and P uptake (type III) . . . . .	77
Table 7	Probability of treatment effects on contrasts comparing soil wheat over time of sampling for each sugar refinery by-products . . . . .	80
Table 8	Probability of treatment effects on contrasts comparing soil corn over time of sampling for each sugar refinery by-products . . . . .	81
Table 9	Probability of treatment effects on contrasts comparing wheat grain over time of sampling for each sugar refinery by-products . . . . .	87
Table 10	Probability of treatment effects on contrasts comparing corn grain over time of sampling for each sugar refinery by-products . . . . .	88



Table 11	Probability of treatment effects on contrasts comparing corn stover over time of sampling for each sugar refinery by-products . . . . .	89
Table 12	Significance of contrasts for wet aggregate stability in 1994, series c and d combined . . . . .	94

## LIST OF FIGURES

### Page

### CHAPTER 3

Figure 1.	Level of soil extractable P and Ca (Melhich III) influenced by sugar refinery by-products and TSP in wheat and corn soils in 1993 and 1994 . . . . .	78
Figure 2.	Level of soil organic matter and soil pH influenced by sugar refinery by-products and TSP in wheat and corn soils in 1993 and 1994 . . . . .	82
Figure 3.	Level of soil extractable $\text{NH}_4\text{-N}$ and $\text{NO}_3\text{-N}$ influenced by sugar refinery by-products and TSP in wheat and corn soils in 1993 and 1994 . . . . .	85
Figure 4.	Wet aggregate stability (%) influenced by sugar refinery by-products and TSP in wheat and corn soils in 1994 . . . . .	90
Figure 5.	Wheat grain P uptake in 1993 and 1994 . . . . .	95
Figure 6.	Corn grain and stover P uptake in 1993 and 1994 . . . . .	96

## LIST OF APPENDIX TABLES

	<u>Page</u>
<b>Appendix</b> . . . . .	110
Table A-1    Extractable P, Ca and organic C provided by sugar refining residues for the incubation of soil analysis . . . . .	111
<b>Appendix B</b> . . . . .	112
Table B-1    Quantity of sugar refinery by-products applied as P source . . . . .	113
Table B-2    Chemical and physical soil analysis of wheat plots . . . . .	114
Table B-3    Chemical and physical soil analysis of corn plots . . . . .	115
Table B-4    Wheat N, P and K content, Yield and P uptake . . . . .	116
Table B-5    Corn N, P, K, Yield and P uptake . . . . .	117

## INTRODUCTION AND OBJECTIVES

Lantic Sugar Limited operates in Montréal a refinery producing refined sugar from raw sugar. The process generates three types of residues with widely differing properties, hence their impact on soil properties are expected to differ. One of them, clarification scum, resulting from phosphatation, has a high organic content and is also rich in terms of total P and Ca. The spent bone charcoal contains a high concentration of total P and Ca. Filter-press mud, made up mostly of diatomaceous earth and clarified liquor impurities as a considerable silica content (Vigneux, J. et al., 1992). Significant contents of phosphorus, calcium, organic matter and/or silica in residues would lead us to believe that they have potential as soil amendments, fertilizers or as a way to improve soil microbial activity under agricultural production.

Clarification scum and filter-press mud in compost mixed with carbon rich material, such as leaves, shavings and cuttings may well prove to be a worthwhile method of managing such residues when field conditions allows direct spreading (Dick and McCoy, 1993). One should however pay particular attention to the C:N ratio resulting from these composted materials in order to not reduce soil nitrate (N'Dayegamiye and Angers, 1993).

The present project is aims at:

- 1- assessing of the use of these sugar refinery by-products as soil amendment and/or P fertilizer source, since P level in the residues is high;
- 2- determining of potential beneficial effects to plant growth;
- 3- an evaluation of composts produced from these materials as soil amendments;
- 4- and developing of recommendations for the use of these residues as suggested by the results obtained in this project.

In order to attain the above objectives, the thesis comprises four chapters.

Chapter 1, *The literature review* is divided into two sections. In section 1, it introduces phosphorus cycling and transformation, their use in agriculture and their behaviour as in crop production management. Section 2 presents a general review of the sugar refining process, the chemical additives and the expected behaviour of each by-product relative to chemical additions, preliminary analysis and previous experiments mentioned in the literature.

In chapter 2, *A laboratory assessment of sugar refining by-products on available soil nutrients, pH and microbial activity* is presented. Each by-product and compost from SCU and FPM is evaluated with regard to its potential to enhance soil macronutrient status, microbiological activity as well as to predict field growth response.

Chapter 3, *Agronomic value of sugar refining by-products and their compost on acid clay soil conditions* studies each refining residue under field conditions. Effects on plant growth (wheat and corn) and related impacts on soil properties are assessed over two (2) year experiments.

Chapter 4 presents the *General Conclusion and recommendations*, including the general behaviour of the residues, the limitations to application of the results of this study and avenues for future research.

## **CHAPTER 1. LITERATURE REVIEW**

## **1.0 PHOSPHORUS AND SOIL NUTRIENT AVAILABILITY**

### **1.1 Phosphorus Transformation**

Phosphorus (P) is an essential macro-element that is involved in many vital life processes such as photosynthesis in plants and energy transformations in all forms of life (Sanyal and De Datta, 1991). For plant-soil-microbiological relationships, the P cycle includes uptake of soil P by plants, recycling from plants and plant residues, biological turnover through mineralization-immobilization, fixation reactions with clay and oxides of iron (Fe) and aluminum (Al) and solubilization of mineral phosphates by microorganisms (Stevenson, 1986). Stewart (1981, as reported by Stevenson, 1986) proposed a P cycle where organic and inorganic forms are partitioned into pools, based on P availability to plants.

The P content of soils in their natural state varies from 0.01% (in sandstones) to 0.2% (in limestones) on a dry-weight basis (Tisdale et al., 1993). Soil P can vary widely in its capacity to supply P to crops because only a small fraction of total P is in available form (Brady, 1990; Stevenson, 1986; Tisdale et al, 1993). Unlike nitrogen which can be recycled to soil by fixation from air, P must be replenished from external sources to supply available P needed by crops (Sanyal and DeDatta, 1991).

### **1.2 Soil Phosphorus Availability**

Several physical and chemical soil properties of a given soil have an effect on solubility of phosphorus and, therefore affect the concentration of soluble P, its availability to plants and the ability of plants to take up the element when a P source is applied to the soil. The important properties and processes controlling P availability include clay content and type, pH, organic matter (OM), P sorption, desorption and P precipitation.

### 1.2.1 Soil Clay

The influence of clay content (Tisdale et al., 1993) is of primary importance for P availability. Evidence that natural availability of P is lower in clay than in coarser soils has been observed by many authors (Sanyal and DeDatta, 1991) using simple Langmuir equations. Sanyal et al. (1990) conducted a study showing that P sorption per unit weight of clay or OM increased with a decrease in clay and OM content. They found that Langmuir adsorption maximum values were reached when clay content was higher than 60%. To explain this observation, the authors proposed easier access of P to clay surfaces than on larger particles. So, soils with a high clay content are known to fix much more P than soils that have a small proportion of clay. Cole and Stephenson (1959) have demonstrated that P solubility drops more when dicalcium phosphate is applied on clay soils as compared to loam type soils.

### 1.2.2 Sorption, Desorption and P Precipitation

P adsorption by soil clay particles is the main mechanism rendering P in soil unavailable to crops (Magdoff and Barlett, 1985). Until recently, desorption of soil P already adsorbed to clay, was considered as irreversible (Okajima et al, 1983). However, Kuo et al (1988) proposed that the recovery of sorbed P in the form of available P is strongly related to P sorption capacity of soil, rather than to the buffering capacity of soil clay particles as previously thought. This important feature could corroborate the results obtained by Barrow (1983), where the author concluded that diffusive migration of initially adsorbed P beneath the absorbing surface of soil particles is a slow process leading to readsorption, hence the apparent irreversibility of P desorption. Willett et al. (1988) attributed this slow process to the possibility that migration of P to the surface of soil particles at sorption sites, rather than penetration of P into crystal lattice, decreases accessibility within the aggregates.

Precipitation of P as Fe, Al or Ca-phosphate is another process that causes P loss from the solution phase, making it unavailable to plants by causing accumulation of substances in a new bulk-solid phase (Sanyal and DeDatta, 1991). According to Lin et al. (1983b), adsorption mechanisms are of more importance at low P concentration whereas P

precipitation occurs at higher concentrations in soil. It is difficult to separate precipitation from adsorption of P, but it can be performed by indirect methods. Xie et al. (1993) proposed the Gibbs equation to describe P adsorption-desorption reaction in the soil in order to separate P sorption from Ca precipitation. These researchers used ammonium lignosulfonate, a by-product from the pulp and paper industry as a substrate where changes in energy of P adsorption-desorption were assessed. Many authors, such as Sibbesen (1981) have proposed equations used to describe various adsorption isotherms for soil P adsorption into the soil.

### 1.2.3 Soil pH and Added P

Ryden and Syers (1977) concluded that P added to acid soil had the tendency to be adsorbed due to hydroxy oxides of iron (Fe) and aluminium (Al) and to 1:1 clay layers, particularly with low soil pH. It has been reported (Sample, 1980) that low pH soils and recently fertilized soils with high-phosphate concentrations, would initiate dissolution of clay particles and allow the precipitation or crystallization of P compounds by reaction with soil components. This process is slow and easily disturbed by P uptake by plants, diffusion or adsorption of soluble P by surface-active aluminium hydroxides and iron present in soil (Sample, 1980).

Le Mare and Leon (1989) have studied P desorption and adsorption on several limed soil types. Migration of available and labile P was found to be enriched by exchangeable P released from soil desorption sites. Oxalate-extractable aluminum strongly correlated with P exchange, and liming was found to lower the buffering capacity of soil and cause higher P concentration in solution.

Phosphate source material may increase, decrease or have no effect on soil pH (Shuman, 1988), depending primarily on soil characteristics (Barrow, 1985), field conditions where seasonal variations in pH could account for the amount of soluble P in soil (Kuo and Jellum, 1987) and the amount and type of P source (Barrow, 1987).



Xie and Mackenzie (1993) assessed mechanisms of soil pH decrement with the use of phosphate. These authors concluded that P application to non-alkaline temperate soils with high exchangeable or soluble Ca may decrease soil pH, where a  $H^+$  releasing process is involved. This suggested that under Québec agricultural soil conditions, P addition could lower soil pH and affect micronutrient availability.

#### 1.2.4 Soil P and OM

The agricultural importance of soil organic matter (OM) in the nutrient cycling and stabilisation of soil structure has been reported many authors (Angers et al., 1993; Powlson et al., 1987). According to Khasaweh et al. (1980), a phosphate fertilization source applied in a matrix with a high level of organic matter enhances the availability of P from the medium, by creating an orthophosphate compound easier to assimilate by the roots. The authors argue that the presence of organic matter would be an important factor in replacing  $H_2PO_4^-$  in the adsorption site and forms a protective coating limiting the adsorption of P to the clay particles.

### 1.3 Soil P and its Relationship with Plant Growth

#### 1.3.1 Plant P Uptake

Raven and Hossner (1994) compared the ability of several equations describing P desorption kinetics from different soils to predict plant growth and P uptake. Their results indicate that initial and final amounts of desorbed P were closely related to relative plant growth and P uptake. Soil P desorption rates generally had soil-specific relationships with growth of corn and P uptake and did not seem to limit the P nutrition to plants.

On the other hand, Ravana et al. (1992) simulated the soil-root interface in an experimental incubator. Their results indicated that P uptake in  $CaCO_3$  induced incubator increased with the reaction time in the soil using P sources applied prior to the experiment. By their radial flow simulating system, the research workers were able to observe the migration of P in a direction opposite to the acidity.

### 1.3.2 P Source Determination

The standard laboratory method used to evaluate the availability of P in fertilizers consists in measuring the quantity of P soluble in water or in a citrate solution (Stevenson, 1986 and Sikora et al, 1989), and normally reporting P by their content of  $P_2O_5$  (Brady, 1990).

However, one cannot necessarily establish a close correlation between the measurement of laboratory solubility and the availability evaluated by agronomic trials with plants grown in a greenhouse or in the field (Destain et al, 1986 and Pierzynski and Logan, 1993). In particular, the relationship between extractable P and plant uptake may be poor if the solubility of an organic fertilizer is compared to chemical P fertilizer, such as calcium diphosphate ( $Ca(H_2PO_4)_2 \cdot H_2O$ ) (Barker et al, 1989), or compost with triple superphosphate (McCoy et al., 1986).

Phosphorus availability to plants varies with the following factors: conditions of the soil as well as its pH (Mnkeni and MacKenzie, 1988); tilling of soil (Dick, 1983); cultural methods as no-tillage and crop rotation (Pierzynski and Logan, 1993); thermic units and water inputs (Liang et al., 1993); the level of phosphorus in the soil and the application of a phosphorated source (Mnkeni and MacKenzie, 1985; Lemunyon and Gilbert, 1993), the nature of the plant and its ability to take up different types of phosphates (McVickar and Walker, 1978). It has been noted that P fertilizer applied to soil could accumulate as residual P and become available in later years, depending on soil type and crop yield (Ridley and Tayakepisuthe, 1974).

Read et al. (1973) reported that crop utilization of inorganic P fertilizer is often less than 20 to 25%, whereas only 2 and 11% of P applied as compost and TSP, respectively was taken up by corn (McCoy et al., 1986).

### 1.3.3 Soil P Tests

After its incorporation into the soil, a substantial part of soluble P is reduced to an insoluble form as a result of adsorption and precipitation (Sanyal and DeDatta, 1991).

More than twenty analytical methods have been proposed (Kamprath and Watson, 1980) to determine available P. Some researchers proposed to compare the value of soil test P to P uptake of crops (Olsen et al., 1983) or to compare their value for different soils having different physical and chemical properties and their relationship to soil P uptake by plant roots (Aquino and Hason, 1984). The method offering the best correlations and now recognized as representative of conditions in Québec is that of Melhich III extraction (Melhich, 1984 in AFEQ, 1990).

#### **1.4 Soil Microbial Activity in Relation to Phosphorus and Organic Sources Application**

##### **1.4.1 Phosphatase Activity**

As it has been reported by Frankenberger and Dick (1983), soil enzymes can be used as a satisfactory index to determine microbial activity. The above authors have suggested that alkaline phosphatase, an enzyme that is entirely derived from the soil microbial population is one of those. Visser and Parkinson (1992) demonstrated that enzymatic activity in the soil environment, which originated mainly from microbial activity and decomposition, is highly correlated to soil quality.

Phosphomonoesterases are enzymes that catalyze the reactions that involve the transformation, in the presence of  $H_2O$ , of phosphate ester into  $R-OH$  and  $PO_4^{3-}$  (soluble phosphate) which is the final stage in the conversion of organic P into soluble available P (Kuprevich and Shcherbakova, 1971). The differences in the level of enzymatic activity among soils are caused by many phenomena. Tabatabai and Dick (1979) demonstrated that pyrophosphatase activity is highly related (correlation  $r^2 = 0.90$ ) with organic C in soil where initial soil pH is below 7. The factors that influence the intensity of the biological process are soil types and the clay content (Juma and Tabatabai, 1977), soil management and temperature (Angers et al, 1993), the level of crop residues (Perucci et al., 1988), the organic matter content in the soil (Dalal, 1982 and Kremer, 1994) and soil pH (Tabatabai, 1982).

More generally, soil enzyme activity is mainly related to organic C content (Dalal, 1982; Harrison, 1983). It would not be surprising therefore to observe that soil enrichment with organic matter promotes phosphatase-producing organisms. However, such behaviour of soil enzyme activity would result in varying growth rates depending on organic material origins: crop residues and herbicides (Perucci et al., 1988) and crop management practices (Kremer, 1994; Doran, 1980; Angers et al, 1993).

#### 1.4.2 Soil Respiration and CO<sub>2</sub> Evolution

Energy sources that are provided by OM after residue application are completely and rapidly transformed into microbial tissue and CO<sub>2</sub> (Stevenson, 1986). Rate of decomposition and so CO<sub>2</sub> liberated in soil to enhance mineralization of substrate as CO<sub>2</sub> is intimately related to microbial activity.

CO<sub>2</sub> production is the result of respiration or oxidation processes (Brooks et al., 1983). Carbon dioxide concentrations are required for absorption, precipitation-dissolution reactions and transport of nutrient in solution. Under anaerobic conditions, the dominant effect of CO<sub>2</sub> concentration on the chemistry of redox sensitive elements such as Fe, Mn, As and Se have been observed to create adverse effects on plant growth (Simunek and Suarez, 1993).

Many investigations have been made in order to determine the best model to predict carbon dioxide transport and production into the soil root zone, but all based on water content, temperature and nutrient status under different conditions (agricultural practices, soil types, volatile organic concentration, salinity and so on) (Glinski and Stepniewski, 1985). Understanding CO<sub>2</sub> evolution is of importance when we look at the nutrient and carbon cycling to determine the intrinsic value of residues applied to soil (Simunek and Suarez, 1993).

## 2.0 COMPOST AS SOIL AMENDMENT

Literature on compost and its use as an organic amendment is abundant. The majority deals, however, with composts produced from sewage sludge, yard trimmings or, to a lesser extent, composts made of municipal solid waste (MSW). Goldstein and Steuteville (1994) surveyed composting units in the United States, and recorded 350 and 20 composting facilities of sewage sludge and MSW respectively. There is very little information on composting of industrial by-products.

"Le Conseil Canadien sur le Compostage" (Environment Canada, 1995) had 30 compost projects on record in Québec (excluding yard trimming), of which 11 were dedicated in part or exclusively to composting industrial by-products. Industry appears to be adopting composting more and more as a means to reduce waste volumes in an environmentally sound manner.

Considering specifically composting of sugar refinery residues, the first project on record was by Krum (1953) who examined the citric solubility of P in compost produced with filter cake and phosphate rock. Acting rather empirically, researchers found that in a compost situation, high concentrations of calcium ions from filter cake and the solubility of the calcium compounds from phosphorus in the rock phosphate was reduced.

More recently, in France, clarification scum was used with the bagasse in a sugar refinery and other fibrous residues to produce high quality compost, adequate for the production of mushrooms on a commercial scale (Zamparini, 1987).

According to Dick and McCoy (1993), compost can influence soil fertility, including soil pH and the supply of mineral nutrient elements, enhance moisture content, temperature and biotic factors. The same authors specify that in temperate regions, the mineralization reaction is much slower and a portion of organic matter generally becomes stabilized as soil humus, possibly several years after application.

As reported by numerous authors (Perucci, 1990; Epstein et al, 1976), mature compost has high populations of microorganisms and its application to the soil acts as a source of food for microorganisms already present in the soil. Furthermore, Perucci (1990) has demonstrated that ammonifiers and nitrifiers in soil are stimulated by the addition of MSW compost, causing microbial biomass carbon, nitrogen and phosphorus to increase over a 12 month incubation period in a soil treated with 2.5 % (w/w) MSW compost. However, the span of time to reach the maximum level of activity varies with the elements present. The same author reports significant differences in soil enzyme activities in soil amended with compost in comparison with soils treated with commercial fertilizers.

Taylor et al. (1978) found that most of the phosphorus remains in an inorganic form during composting, where its availability depends less on OM mineralization than fixation reactions. This is supported by Mazur et al. (1983) who found that available soil P, determined by anion exchange resin, increased by 57% by the addition of MSW compost.

Several experiments were carried out with a view to enrich compost with rock phosphate under aerobic conditions (Single and Yadah, 1986). The process enhances the solubility of phosphorus due to the increased of temperature during composting, especially when materials of low natural P availability are added (Mathur et al., 1987). On the other hand, phosphorus uptake by corn increased only in the absence a phosphorated source in the study by Mazur et al. (1983).

Martins and Kowald (1988) concluded that compost addition to soil decreased crop yield due to development of N deficiency caused by excessive N demand of soil microorganisms degrading the added compost. These authors suggested that, when applied to soil, mature humified compost continues to be degraded by microorganisms, releasing mineral nutrients through mineralization.

Several problems have been encountered, however, by the use of compost as sole mineral nutrient source for crop growth (Dick and McCoy, 1993). When compost is not fully

mature, its use as soil fertilizer creates a demand for nutrients as the organic components break down to maturity. Composts are diluted and unbalanced sources of N, P and K to meet crop requirements compared to fertilizer sources, which may allow a deficiency of one or more nutrient, such as reported by McCoy et al. (1986). Generally, when sewage sludge compost is used to meet crop N requirements, an excess of P is applied to soil.

Compost from yard trimmings, as well as from bark residues, contains phenolic polymer substances such as lignin that resist to biological decay (Crawford et al, 1990). Whether applied as fresh or composted tree clippings, no inhibitory effects on potato growth were found under field conditions (Beauchemin et al, 1992). On the other hand, phenolic materials are known to inhibit nitrification ( $\text{NH}_4\text{-N}$  to  $\text{NO}_3\text{-N}$ ) in agricultural systems (Azhar et al., 1986) and may bind with the enzyme urease (Al-Kanani et al., 1990). It also has been proposed that organic material, could stimulate denitrification (Firestone, 1982) by acting as an electron donor as well as by creating anaerobic microsites (Parsons et al, 1991). However, nitrification can be inhibited by amendments. On sludge-compost amended soils, McClung et al. (1983) realized that nitrapyrin inhibited soil nitrification, as suggested by unchanged  $\text{NO}_3$  concentration and increased  $\text{NH}_4^+$  ions.

Sikora et al. (1983) also reported that if only 25 to 40 % of total P would be available in primary physico-chemical dephosphatation sludge compost, the amount of P applied would still be substantially in excess for grasses forage crops. Finally, the release of nutrients from compost may not correspond to the time when crop demand is greatest (Epstein et al., 1976). As previously mentioned, P from compost needs a certain time to become soluble and thus available.

This aspect of mineral availability has been studied by O'keefe et al. (1986). These authors noted that the addition of OM to soil, particularly in the form of compost, results in an increase of mineralization of N. Sewage sludge compost application (Epstein et al., 1976) evoked the evidence that micro-elements become more available by chelating the

minerals, making them soluble and giving them access to the root system by better diffusion.

Among the physical changes provided by sewage sludge compost, it was noted that wet aggregate stability could be enhanced by a relatively small quantity (70 t<sub>m</sub> ha<sup>-1</sup>) in clay soil, short of OM (Avnimelech and Cohen, 1988), but where much greater quantities (230 t<sub>m</sub> ha<sup>-1</sup>) are required by soils short of OM, but with a sandy loam texture.

### **3.0 LANTIC REFINING PROCESS AND BY-PRODUCTS GENERATED**

In order to establish a link between the residues evaluated in the present project and their potential use, this section presents a literature review dealing with the main steps of sugar refining and the main components thereof.

#### **3.1 Lantic Refining Process**

Lantic Sugar imports raw sugar from countries producing sugar cane in order to transform it into white granulated sugar (solids) or liquid sugar, in its Montréal refinery (Vigneux et al., 1992).

The sugar refining process comprises five steps: affinage (preliminary refining), clarification, filtration, bleaching and finally crystallization and preparation. The previously mentioned residues are all generated before the last step. The filtration process of sugar liquors varies from one refinery to another. Most of the experiments dealing with agricultural use of refinery by-products were conducted with residues that were originated from carbonatation, where a mixture of calcium hydroxide and carbonic gas is used in the process (Philipps and Jauhri, 1984; Prasad, 1976; Kuong et al., 1988). Their make up however has not always been clearly defined, and the terms "press mud", "filter-press cake", "scum", "milo", "miala", "filter mud", "mill mud" are used in the literature interchangeably to designate residues coming sometimes from different



processes, the make up of which varies from one region or one country to another (Alexander, 1972).

The first step in the refining process is called affinage or wash, which consists mainly in the separation and removal of the molasses coating covering the surface of raw sugar crystals (Chen, 1985). Liquors resulting from this first steps have a slight acidity and contain a certain amount of insoluble matter. The liquors are clarified either by adding phosphoric acid ( $P_2O_5$ ), hydrated lime ( $Ca(OH)_2$ ) and a polymer, Taloflote, or by induction heating causing the precipitation of solubles and colloidal impurities (Chen, 1985). The process is referred to as phosphatation, taking into account the large quantities of phosphoric acid used during the process.

The addition of phosphoric with large amounts of lime neutralizes the organic acids found in raw sugar forming calcium phosphate (Paturau, 1989). The addition of cations also favours the creation of ionic links between molecules of the same charge allowing coagulation and precipitation of several organic substances, other than sugars, including a large part of gums, albumins and pectins (Chen, 1985).

A heavy flocculent precipitate brings down a substantial part of suspended or colloidal matter. Rising bubbles of injected air bring up particles they run into. The process creates a layer of foam on the surface of the liquor, called clarification scum (SCU). An anionic polymer of high molecular weight (Taloflote) is added during phosphatation to enhance the clarification process (Barton, 1977; Chen, 1985). Before being piped into the main clarifier, secondary liquors go through preliminary treatment in a secondary clarifier where ammonium dimethyl dialkyl chloride (ALCHEM IC08) is added to the dissolved sugar solids base. This cationic polymer is an aid to the formation of primary floc (personal communication, Lantic, 1993).

Scums are later unsugared by dilution followed by reclarification, before being finally piped into dehydration equipment giving them a sticky and pasty texture similar to that of chemical wastewater sludges.

After phosphatation, liquors are piped into a filter-press where, under pressure, calcinated diatomaceous earth is used in the presence of alkali (Celatom) to facilitate speed and increase the effectiveness and performance of the filtration process (Chen, 1985). Diatomaceous earth is also used in different industries such as breweries (Fisher, 1992; Bridge, 1987). Diatomaceous earth, kielselguhr or kielselgur, is non metallic mineral made up of the skeletons of aquatic unicellular microscopic plants called diatoms. In its natural state, diatomaceous earth is made up mostly of amorphous silica (non-crystalline silicium dioxide) (Lewin, C.J., 1961). The mixture of earth, retained particles and Alfa floc, an inert filtering aid made of cellulose (Vigneux, J et al., 1992) is use in small quantities to enhance filtration, are all components of filter-press (FPM).

Bone char is used as for final purification purposes. Bone char is the most used adsorbant to bleach and purify liquors in the refining of sugar (Chou and Hanson, 1971) and must be replaced on a monthly basis. Bone black is manufactured by crushing (3.35 mm) and heating to 1300°F (Chen, 1985) bones of selected animals. Bone char is used almost exclusively in the sugar refining industry, even though it has been a subject of research to other uses for it, such as drinking water treatment (Christoffersen et al, 1991).

### **3.2 Lantic By-products and their Potential as Soil Amendment or Fertilizer**

#### **3.2.1 Spent Bone Char (SBC)**

According to an exhaustive study by Gupta (1973), calcination of bones and rock phosphate greatly enhances the solubility of phosphorus compared to non-calcinated bone and rock phosphate powder. The author explains the phenomenon as a chemical change in the phosphated fertilizers. Non-calcinated bones contain hydroxyapatite ( $3\text{Ca}_3(\text{PO}_4)_2 \cdot (\text{Ca}(\text{OH})_2)$  apatite carbonate ( $3\text{Ca}_3(\text{PO}_4)_2(\text{CaCO}_3)$ ). High temperatures during the calcination process may have caused the dissociation of apatite carbonate particles, producing carbonate and tricalcium phosphate. This could explain both the greater solubility of calcinated bone and the fact that solution, coming from calcinated bone show a more alkaline pH than obtained with non calcinated bones (fresh or steamed), which remain relatively neutral even when extracted at 40°C. Since phosphate solubility

increases with increasing extraction temperatures, the incorporation of SBC in compost operations could further increase the solubility of P, taking into account that compost temperatures could reach 40 to 70°C (Haug, 1980) under aerobic composting conditions. That heat from compost increases the availability of phosphorus of low natural availability, such as rock phosphate, has already been demonstrated (Bagar, et al, 1985; Mathur et al, 1987).

### 3.2.2 Clarification Scum (SCU)

Kuong et al. (1988), experimenting in six (6) different zones of Mauritania, have found, as have other authors such as Prasad (1976), Cooper and Abu Idris (1980), that phosphate carried in sugar refinery residues was more beneficial to sugar cane plantations than P carried by an equivalent quantity of  $P_2O_5$  from superphosphate. On the other hand, the C:N ratio of scum is often high ( $> 20$ ) in sugar cane (Alexander, 1972; Wood, 1981) or beet mills (Bogdonava et al, 1969) with the result that nitrogen carried by scum is not necessarily available for plant growth.

In the case of the Montréal plant, clarification scum contains considerable N, P and C for a net C:N ratio of 21:1. We can expect that the application of that matter will enhance soil microbial activity, improve soil physical properties and act as P fertilizer.

### 3.2.3 Filter-Press Mud (FPM)

No specific mention was found in literature dealing with the agricultural use of FPM produced by filtration of sugar in a process using diatomaceous earth as a filtration agent. On the other hand, research was carried out in Germany during the 1980's on the spreading of filtration muds produced by breweries in the fields (Hodenberg et al, 1987; Schildbach, 1990). Schildbach (1990) reports a moderate yield improvement of corn grown in a greenhouse experiment, presumably caused its porous structure and low density ( $< 0,5 \text{ kg m}^{-3}$ ). An increased yield was noted in comparison with the controls, in spite of the fact that diatomaceous earth contained no nutrients. The author believes that the increase could be partly attributed to an improvement in the physical conditions of the soil. Adding diatomaceous earth to sandy soil, less subject to physical deficiencies,

would therefore have no effect on yield. On the other hand, adding large quantities of diatomaceous earth on the surface of the soil could prove harmful to germination of some plants because of its tendency to form an excessibility damp layer on the surface of the soil (Hodenberg et al., 1987). According to the same authors, water retained in that structure would be readily available to plant and would increase the water retention capacity of heavy soils.

Silica, in the form of  $\text{SiO}_2$ , is present in soil in very large quantities (from 60 to 90 %, Brady, 1990). Nevertheless, minute quantities of the acid form of silicate,  $\text{Si}(\text{OH})_4$  are available to plants. The concentration of  $\text{Si}(\text{OH})_4$  is still lower when soil pH is low due to the presence of large quantities of iron and aluminium hydroxides (Russell, 1973). Depending of soil type, iron and aluminium hydroxides could control both silica and phosphorus solubility. Then, because of its high silica content, diatomaceous earth added to the soil could increase soil P availability (Russell, 1973).

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**CHAPTER 2. LABORATORY ASSESSMENT OF SUGAR REFINING BY-  
PRODUCTS ON AVAILABLE SOIL NUTRIENT, pH AND MICROBIAL  
ACTIVITY**

## ABSTRACT

Refinery residues present a waste disposal problem for the sugar refining industry but these materials often have high phosphorus and calcium contents and thus have the potential for use as soil amendments or fertilizers. This experiment compared three residues, spend bone char (SBC), filter-press mud (FPM), clarification scum (SCU), and a compost (COM) produced using FPM and SCU, in terms of their effects on extractable nutrients, pH, soil respiration and phosphatase activity in two soils in a laboratory incubation. SCU, SBC and COM significantly increased extractable P and Ca and pH in the soils. Recovery of added extractable P was low in SCU and COM indicating fixation whereas recovery of P added in SBC was 100 - 200% higher than expected, depending on the soil. FPM did not increase extractable nutrient concentrations but enhanced phosphatase activity. All residues stimulated soil respiration and caused slight immobilization of soil mineral N. The study indicates that the residues, alone or in combination with other materials, have significant potential as soil amendments.

Key words: Sugar refinery by-products, Phosphorus, Soil respiration, Phosphatase, Compost, Spent bone char, Filter-press mud, Clarification scum.

## RÉSUMÉ

Les résidus issus du raffinage du sucre présentent un problème de disposition pour l'industrie sucrière. Pourtant, ces matériaux possèdent souvent des teneurs élevées en phosphore et en calcium, ce qui leur confère un potentiel en tant que fertilisant ou amendement des sols. Au cours de cette expérience, l'effet sur les éléments extractibles du sol, le pH, l'activité microbienne et la phosphatase des poussières de noir animales " Spent bone char (SBC)", des boues de filtre-presse "filter press mud (FPM)", des écumes de clarification "clarification scum (SCU)", et un compost (COM) produit à partir des deux derniers résidus ont été comparés dans deux sols, en condition d'incubation. Dans l'ordre, SCU, SBC et COM ont augmenté significativement les concentrations de P et Ca, ainsi que le pH du sol. La récupération de P suite aux apports de SCU et COM au sol a été faible, indiquant la fixation de cet élément. Par ailleurs, la récupération de P dans les contenants ayant été amendés au SBC a été de l'ordre de 100 à 200% supérieur aux prévisions, selon les types de sol. Les FPM n'ont pas augmenté le contenu des macro-éléments du sol, mais ont contribué plutôt à stimuler l'activité de la phosphatase. Tout les résidus ont stimulé la respiration des sols et ont causé une légère immobilisation du N minéral du sol. Cette étude indique que tous ces résidus, appliqué seul ou en combinaison avec d'autres matériaux ont un potentiel comme amendement des sols.

Mots-clés: Sous-produits du raffinage du sucre, C évoluant comme du CO<sub>2</sub>, Phosphatase, Compost, Poussière de noir animale, Boues de filtre-presse, Écumes de clarification.



## INTRODUCTION

The major inputs to the sugar refining process used by Lantic Sugar Ltd. of Montréal, Quebec, include hydrated lime, phosphoric acid, diatomaceous earth, black bone char, ammonia and organic polymers. Some of these materials are used as nutrient sources in agriculture or in the production of agricultural chemicals and consequently refinery wastes may have potential for use as soil amendments or fertilizers. The wastes consist of reaction products, and spent filter residues along with impurities from the raw sugar, and come in three forms, namely clarification scum, filter-press mud and spent bone char, produced at different stages in the processing. Scum and bone char are particularly high in phosphate (Chen, 1985), and have been shown, in experiments carried out in Mauritania, to constitute an amendment of higher value to sugar cane farming than the equivalent superphosphate application (Kuong et al. 1988).

**Clarification scum (SCU)** is the result of primary and secondary clarification by the phosphatation process. It is the product of multiple desugaring treatments of sugar liquors, where the addition of phosphoric acid ( $H_3PO_4$ ), hydrated lime ( $Ca(OH)_2$ ), a flocculent (an anionic polyacrilamide) and a cationic surfactant (an ammonium salt) are the major chemical components used during the treatment. Clarification scum contains a significant amount of organic matter (over 43%) and phosphate materials.

The residues of subsequent clarification by filtration are composed principally of diatomaceous earth. When the filtration capacity of diatomaceous earth is depleted, the mixture forms the waste material called **filter press mud (FPM)**. The agronomic interest in diatomaceous earth comes from its silica content. Long term experiments carried out more than twenty years ago in England (Russell, 1973) showed that adding silica would help increase phosphorous in solution by removing phosphated ions of Fe and Al hydroxides, particularly in soils with near neutral pH and low in phosphorous content.

Calcinated bone char is an absorbent used to bleach and purify liquors during sugar refining. After a period of use, bone char becomes exhausted because of accumulation

of organic matter and ash (Chou and Hansen, 1971). It is designated **spent bone charcoal (SBC)** when it loses its porosity or when the particle size breaks down by attrition. The resulting waste material is in the form of a fine powder. Being derived from bone, SBC is high in phosphorus and calcium. According to Gupta (1973), the solubility of  $P_2O_5$  in water, relative to the total phosphate content, and its availability to plants increases after the calcination process used in the production of bone char.

The agricultural values of these residues as amendments is currently unknown. The availability of the nutrients they contain, their behaviour with respect to cultivation and the residual effects on land treated with such residues must be evaluated in order to issue appropriate recommendations for their use as soil amendments and/or fertilizers.

Since the three materials have widely differing properties and hence their impacts on soil are expected to differ, an experimental project was carried out to determine the agricultural and environmental impact arising from the use of such residues. This paper reports the initial study of this project in which the nutrient dynamics, enzymatic and microbial activity in soil amended with each of these wastes under laboratory conditions was determined. The specific objective of the experiment was to evaluate the extent to which sugar refinery by-products and a compost containing scum and filter-press mud affected the concentrations of Melnich III extractable macro-elements and modified microbial respiration and phosphatase activity of two contrasting soils. The performance of each material was to be determined in order to establish which parameters relating to nutrient cycling and microbial activity were likely to be affected by the use of the residues as fertilizer in a field experiment.

## **MATERIAL AND METHODS**

The behaviour of four materials in soil was examined. SBC was characterized by a high levels of P and Ca, FPM by low levels of extractable nutrients but high C:N ratio, SCU by high P and OM contents (Table 1). Compost (COM), made up of 33% SCU, 17% FPM, 37% wood chips and 13% septic sludge, was characterised by high P content and

**Table 1. Chemical analysis of sugar refining by-products and their compost (dry matter basis)**

Descriptors	FPM	SCU	SBC	COMPOST
1 soil : 1 water				
pH	6.4	7.3	7.7	7.3(2)
%				
H2O in fresh residues	50	66	5	59 (1)
O.M.	9	43	7	17 (1)
C/N	64	21	8.5	44
ms/cm				
Electrical conductivity	1.3	1.1	1.0	---
mg/kg				
Total N	840	11800	4810	3800 (1)
Total P	1250	81800	153100	29300 (1)
Extractable P	480	6561	1405	6860 (1)
Total K	410	2461	6379	1380 (2)
Extractable K	120	75	42	---
Total Ca	35900	24400	355000	10700 (2)
Extractable Ca	3050	29600	14200	---
Total Mg	470	8763	1550	1900 (2)
Extractable Mg	150	4370	556	---

(1) = Chemical analysis provided by Nutrite inc. Brossard, Qc, Canada. J4Z 3N2

(2) = Chemical analysis provided by CreaLab, Qc, Canada. J1N 2T3

C:N ratio. Two contrasting agricultural soils, common in the region of Montréal, were used; Ste-Rosalie clay (humic gleysol) and Chicot sandy-loam (dystric brunisol) (Table 2), sampled at 0-20 cm. Before incubation, the content in total P, K, Ca and Mg, N total (Keeney and Nelson, 1982), pH (Black, 1965) and OM (Nelson and Sommers, 1982) in the residues were determined (Table 1). Chemical and physical characteristics of these soils are shown in Table 2. One hundred grams of soil was weighed into incubation containers and brought to 80% of capacity (Ste-Rosalie and Chicot soils, 27% and 24% respectively). Containers were preincubated for 5 days before treatment application.

Overall, nine treatments including an unamended control were replicated three times. The four materials (COM, FPM, SCU and SBC) at two rates (2.5 and 5% fresh weight/100 grams of air dried soil) and a control were prepared for two series of analyses for each soil. The refining process produces residues having a moisture content within a narrow range and hence the treatments were based fresh weight to be comparable to direct application of the waste in the field. The  $P_2O_5$  equivalent rate of application ranged between 31 kg ha<sup>-1</sup> for FPM applied at 2.5% rate to 696 kg ha<sup>-1</sup> for COM at 5% rate of application. Extractable P, Ca and organic C provided by the residues and their equivalent  $P_2O_5$  and Ca application are presented in the appendix (Table A-1). Containers were maintained at 80% of field capacity by weekly additions of deionized water, and incubated at room temperature (20-23° C) in the laboratory. Containers were opened for aeration at two-day intervals.

### **Series 1: Destructive soil sampling**

One set of incubation containers was assigned for destructive soil sampling on days 6, 13 and 27. For both soil types, a total of 81, 590 ml plastic incubation containers were kept covered with polyethylene sheets (plastic bags), allowing gas exchange. Destructively sampled soils were then air dried as proposed by Juma and Tabatabai (1978) and Eivazi and Tabatabai (1977) for phosphatase analysis, and analysed for soil P, K, Ca, Mg,  $NH_4$ -N,  $NO_3$ -N, pH, acid and alkaline phosphatase.

**Table 2. Some initial soil characteristics (0-20 cm) for Ste-Rosalie and Chicot**

<b>Descriptors</b>	<b>Ste-Rosalie</b>	<b>Chicot</b>
OM (%)	5.5	3.1
Organic C (g/kg)	32	18
Particle size (g/kg)*		
sand	100	500
silt	190	220
clay	710	280
Moisture content at		
field capacity (g H <sub>2</sub> O/100g soil)	27	24

\*\* = Data obtained from Liang and MacKenzie, 1994.

For the analysis of P, K, Ca and Mg, soil subsamples were shaken in Melhich III solution (2.5 g of soil with 25 ml of Melhich III solution) for 5 minutes and suspension filtered through Whatman # 5 filter paper (Melhich, 1984). Filtrates were analyzed colorimetrically for P and by flame photometry for K using Lachat instruments. Filtrates were analysed for Ca and Mg using atomic absorption spectrophotometry.

Soil subsamples were shaken in 1N KCl (1 g soil: 10 ml KCl) for 1 hour and filtered through #2 Whatman filter paper for the analysis of nitrogen. Filtrates were analysed colorimetrically for  $\text{NH}_4\text{-N}$  and  $\text{NO}_3\text{-N}$  using a Technitron auto-analyzer, in accordance to procedures suggested by Keeney and Nelson (1982). Soil pH was measured in a 1 g soil:2 ml 0.1M  $\text{CaCl}_2$  suspension (Black, 1965).

Acid and alkaline phosphatase activity was measured by the conversion of the P-nitrophenyl phosphate (p-NPP) to p-nitrophenol (Tabatabai, 1982). One gram of air dried soil subsamples with 0.2 ml toluene, 4 ml modified universal buffer (MUB) at pH 6.5 for acid and 11 for alkaline phosphatase and 1 ml p-NPP was substrates incubated at 37° C. One millilitre of 0.5 M  $\text{CaCl}_2$  and 4 ml NaOH were used to stop the phosphatase activity. Solutions were filtered through #2v filter papers and p-NPP was measured from the yellow colour intensity on spectrophotometer at 400 nm wavelength.

### **Series 2: $\text{CO}_2$ evolution**

For each soil, a total of 27 one liter incubation containers were incubated for one month to monitor  $\text{CO}_2$  evolution. Carbon dioxide was monitored by suspended vials above the soil surface, containing 25 ml of 1N NaOH solution on which  $\text{CO}_2$  was trapped. Containers, incubated in a complete randomly randomized arrangement were opened twice a week to provide ambient air exchange and to replace the alkali (NaOH) which was analyzed for  $\text{CO}_2$ . Carbon dioxide concentration was determined on six dates by adding 2N  $\text{BaCl}_2$  to allow precipitation of carbonates as insoluble  $\text{BaCO}_3$ . The unreacted NaOH was titrated with 1M HCl, using phenolphthaleine as indicator (Anderson, 1982).

### **Statistical analysis and calculations**

The experiment was analysed using the SAS general linear model procedure (proc GLM) as a randomized complete block design for both series independently. The effect of rate of application was analysed by polynomial contrasts. The LSD test was performed to compare treatments and also sampling dates in the case of acid phosphatase. All statistical analyses were done using SAS for Windows (SAS, 1990).

The amounts of P fixed in each treatment were calculated as the difference between the initial Mellich III extractable P added in residues and in the soil, and the extractable P in the soil/residue mixture after the incubation. The percentage of fixed P was determined using the amount of fixed P divided the initial extractable P.

## **RESULTS**

### **Soil Chemical Properties**

Significant effects involving residues, soils and dates were observed for the chemical parameters measured (Table 3). The significant RES\*RATE effect were observed for all parameters, except  $\text{NH}_4\text{-N}$ . This is indicative of the generally linear increase in extractable nutrients and pH with increasing application rate for all residues except FPM, and significant decreases or increases in  $\text{NO}_3\text{-N}$  with application rate (Table 4).

A significant RES\*SOIL effect was observed in extractable nutrients, except Mg and  $\text{NH}_4\text{-N}$  indicate that the residues behaved differently on different soils. Concentration of extractable P in the Ste-Rosalie soil was highest in the SBC treatments whereas the SCU treatment had higher extractable P than SBC in the Chicot soil (Table 4). A similar pattern was observed in extractable Ca. The COM treatment increased  $\text{NO}_3\text{-N}$  concentration in the Ste-Rosalie soil but caused a decrease in the Chicot soil, in contrast to the SBC treatment that appeared to stimulate nitrification in the Chicot soil (Table 4). The decrease in  $\text{NO}_3\text{-N}$  with the FPM treatment on the Chicot soil was particularly apparent. FPM had no effect on pH in the Ste-Rosalie soil but increased pH significantly in the Chicot soil (Table 4).

**Table 3. Significance of extractable P, K, Ca, Mg, NH<sub>4</sub>, NO<sub>3</sub> and pH for the destructive sampling analysis.**

SOURCES	df	P	K	Ca	Mg	NH <sub>4</sub> -N	NO <sub>3</sub> -N	pH
RES	4	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
REP	2	0.8067	0.8337	0.5524	0.9749	0.6744	0.4205	0.6524
SOIL	1	0.0715	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
TIME	2	0.0150	0.0193	0.0240	0.0034	0.0001	0.0001	0.0001
RATE	1	0.0001	0.4376	0.0001	0.0149	0.0001	0.0053	0.0001
RESxREP	8	0.9065	0.6017	0.5587	0.6081	0.9848	0.7109	0.8512
RESxTIME	8	0.0264	0.2970	0.0184	0.8637	0.0001	0.0003	0.0001
RESxSOIL	4	0.0001	0.0157	0.0001	0.1329	0.0936	0.0001	0.0001
RESxRATE	3	0.0001	0.0042	0.0001	0.0001	0.7214	0.0012	0.0001
RESxSOILxRATE	4	0.0134	0.8682	0.0282	0.0226	0.1676	0.3021	0.0259

RES= Residues, REP= Replicates or block, SOIL= Soil types, time= sampling time, RATE= Rates of applications

df = Degrees of freedom

Shaded values represent Prob < 5% level.



Table 4. Mellich III-extractable P, K, Ca, Mg, KCl-extractable NH<sub>4</sub>, NO<sub>3</sub> and pH in extractant from two incubated soil types. Values are means of three sampling dates, replicated three times.

SOIL TYPE	RES	RAT	P	K	Ca	Mg	NH <sub>4</sub> -N	NO <sub>3</sub> -N	pH
					ppm				1 soil: 2 water
Ste-Rosalie	CON	0.0	38 (f)	219 (c)	1515 (e)	597 (c,d)	91 (a,b)	42 (c)	4.7 (f)
	COM	2.5	98 (e)	227 (b)	1659 (d)	604 (b,c)	89 (b,c)	46 (a,b)	4.8 (e)
	COM	5.0	156 (d)	234 (a)	1861 (c)	606 (b,c)	87 (c)	46 (a)	4.9 (d)
	Relation		Linear	Linear	Linear	N.S.	(Linear)	Linear	Linear
	FPM	2.5	38 (f)	221 (c)	1442 (e)	603 (b,c)	84 (d,e)	38 (d)	4.7 (f)
	FPM	5.0	41 (f)	218 (c)	1457 (c)	591 (d)	77 (f)	33 (e)	4.7 (f)
	Relation		N.S.	N.S.	N.S.	N.S.	(Linear)	(Linear)	N.S.
	SCU	2.5	183 (d)	218 (c)	1833 (c)	612 (b)	92 (a)	41 (c)	4.9 (d)
	SCU	5.0	310 (b)	218 (c)	2184 (b)	629 (a)	86 (e,d)	36 (d)	5.1 (b)
	Relation		Linear	N.S.	Linear	Linear	(L. and Q.)	(Linear)	Linear
	SBC	2.5	271 (c)	216 (c,d)	2171 (b)	595 (c,d)	87 (c)	43 (b,c)	5.0 (c)
	SBC	5.0	379 (a)	211 (d)	2603 (a)	587 (d)	83 (e)	41 (c)	5.2 (a)
	Relation		L. and Q.	(Linear)	L. and Q.	N.S.	(Linear)	N.S.	L. and Q.
	LSD <0.05		28.5	6.2	95.0	11.3	2.8	2.6	0.04
Chicot	CON	0.0	64 (f)	237 (b,c,d)	2392 (e)	897 (b,c)	18 (a)	19 (b,c)	5.9 (f)
	COM	2.5	103 (e)	239 (a,b)	2514 (d)	888 (c,d)	18 (a)	20 (b,c)	5.9 (e)
	COM	5.0	187 (d)	241 (a)	2708 (c)	903 (b,c)	15 (b)	22 (a,b)	6.1 (d)
	Relation		L. and Q.	Linear	Linear	Linear	(Linear)	N.S.	Linear
	FPM	2.5	44 (f)	238 (a,b,c)	2349 (e)	898 (b,c)	13 (c)	10 (d)	6.0 (d)
	FPM	5.0	45 (f)	234 (c,d)	2376 (e)	914 (b)	10 (d)	2 (e)	5.9 (e)
	Relation		N.S.	N.S.	N.S.	N.S.	(Linear)	(Linear)	Linear
	SCU	2.5	222 (c)	235 (b,c,d)	2750 (c)	902 (b,c)	18 (a,b)	18 (c)	6.1 (c)
	SCU	5.0	408 (a)	236 (b,c,d)	3315 (a)	952 (a)	17 (a,b)	17 (c)	6.3 (a)
	Relation		Linear	N.S.	L. and Q.	L. and Q.	N.S.	N.S.	Linear
	SBC	2.5	198 (d)	233 (d,e)	2761 (c)	888 (c,d)	17 (a)	21 (a,b)	6.1 (c)
	SBC	5.0	302 (b)	229 (e)	3165 (b)	873 (d)	16 (a,b)	23 (a)	6.3 (b)
	Relation		Linear	(Linear)	Linear	(Linear)	N.S.	Linear	L. and Q.
	LSD <0.05		22.4	4.5	86.1	24.5	1.7	2.8	0.04

Variable means with the same letters are not significantly different within the same soil type.

Relation=Relationship as prob &lt; 5% between treatments and rates interaction as a polynomial contrast (parentheses indicate negative relationship).

RES=Residues were CON=Control; COM=Compost; FPM=Filter press mud; SCU=Clarification cum and SBC=Spent calcinated bone char.

A significant RES\*DATE effect was observed for all parameters except K and Mg (Table 3). This effect arose from an increase over time in  $\text{NO}_3\text{-N}$  from COM in Ste-Rosalie and SBC in Chicot soils, while a decrease was observed in the FPM treated soil (data not shown). A general decrease occurred in  $\text{NH}_4\text{-N}$  for all residues though out the experiment. Extractable P and Ca from SCU, SBC and COM amended soils increased with incubation time in the Chicot soil types but did not change after day 6 in the Ste-Rosalie soil. The significance of the RES\*DATE interaction for pH arose from the general increase over time in all residue treatments, but especially in COM treatments in Ste-Rosalie and FPM treatments in the Chicot soil (data not shown). Temporal changes during the incubation were relatively minor and thus were considered to be of lesser importance than other effects and will not be discussed further.

Concentrations on  $\text{NH}_4\text{-N}$  were affected by RES, SOIL and RATE (Table 4). In general,  $\text{NH}_4\text{-N}$  was significantly lower than the control at the high application rate, resulting in a linear RATE effect (Table 4). This effect was particularly apparent in the FPM treated soils.

Calculation of fixation of added P indicated distinct differences in the fixation properties between residues (Table 5). Fixation was high (24 to 50%) in the COM and FPM treatments, with the exception of 5% COM in the Ste-Rosalie soil where 57% more P was recovered at the end of the incubation than was present initially. Fixation was low in the SCU treatments. In contrast to the other residues, SBC increased extractable P levels by 100 and 250% in the Chicot and Ste-Rosalie soils, respectively, over the period of incubation.

### **Evolution of $\text{CO}_2$**

Microbial activity, as measured by  $\text{CO}_2$  evolution, was stimulated by the addition of all residues to both soils (Table 6). The SCU and the SBC treatments in the Ste-Rosalie soil tended to have the highest  $\text{CO}_2$  evolution, whereas evolution was lowest in the COM treated soils. Differences in the quality of the organic matter in the residues are apparent in the percentage of added C evolved (Table 6). The high proportion of C added in FPM

**Table 5. Fixation of Melhich III extractable phosphorus after 27 days of soil incubation**

Soil	Residues	Amount of P fixed (mg/kg)		% of initial P fixation	
		2.5%	5%	2.5%	5%
Ste-Rosalie	COM	44	-139	31%	-57%
	FPM	12	31	24%	50%
	SCU	19	56	9%	15%
	SBC	-198	-271	-271%	-250%
Chicot	COM	65	85	39%	31%
	FPM	32	43	42%	49%
	SCU	6	-16	3%	-4%
	SBC	-99	-167	-100%	-124%

**Table 6. Carbon added with residues and percentage of added C consumed for CO<sub>2</sub> production during the 27 days incubation in 100g of soil.**

Soil	RESIDUES	Rates (%)	C input (mg C/ 100g soil)	Total C evolved as CO <sub>2</sub> (mg C/ 100g soil)	Added C consumed as CO <sub>2</sub> (%)
Ste-Rosalie	COM	2.5	363	46(e)	13
	COM	5.0	725	45(e)	6
	FPM	2.5	136	63(d)	46
	FPM	5.0	273	98(b)	36
	SCU	2.5	631	81(c)	13
	SCU	5.0	1232	110(a)	9
	SBC	2.5	103	49(e)	48
	SBC	5.0	205	102(a,b)	50
Chicot	COM	2.5	363	22(e)	6
	COM	5.0	725	23(e)	3
	FPM	2.5	136	46(c,d)	33
	FPM	5.0	273	84(b)	31
	SCU	2.5	631	63(c)	10
	SCU	5.0	1232	104(a)	8
	SBC	2.5	103	39(d,e)	38
	SBC	5.0	205	52(c,d)	25

Variable means with the same letters are not significantly different within the same soil type.

and SBC consumed during the incubation indicates that the C contained in these residues is easily degradable by microorganisms relative to the C in the SCU and COM. The potential for degradation apparently depends on soil characteristics as indicated by the lower overall consumption of added C in the Chicot soil (Table 6). An interaction between SOIL and RATE is indicated by the relatively low percentage of degradation in the 5% application rate SBC on the Chicot soil.

### **Phosphatase Activity**

Activities of acid phosphomonoesterase induced by the residues after 6, 13 and 27 days and alkaline phosphatase after 6 days are presented in table 7. On the Ste-Rosalie soil, FPM and SBC at 2.5% were the only treatments to stimulate acid phosphatase activity whereas FPM, SCU and COM at 5% stimulated alkaline phosphatase. The pattern of results was more complex in the Chicot soil with FPM stimulating acid phosphatase at 6 and 27 days but inhibiting activity at 13 days. Activity was also inhibited in SBC treatments at 13 days. FPM at 2.5% stimulated alkaline phosphatase activity as did compost at 2.5% and SCU at 5%. At 5%, FPM reduced alkaline phosphatase activity relative to 2.5% FPM application.

## **DISCUSSION**

### **Residues as phosphorus sources**

On the basis of the P contents of the residues themselves, SCU and the compost were expected to be the best sources of extractable P in the incubated soils. The results, however, indicate that the highest extractable P levels were obtained by amendment of soil with SBC, despite the lower extractable P content of this residue. The observation that FPM had no effect on extractable P levels was expected because of the low P content of this residue. Net fixation of extractable P occurred with the compost, FPM and SCU whereas significantly more P was extractable from the SBC - soil mixtures than expected. The physical form (i.e. powder) of SBC was expected to increase fixation but this did not appear to be the case.

**Table 7. Soil acid and alkaline phosphatase activity after 6, 13 and 27 days of incubation**

SOIL TYPE	RES RATE		ACID PHOSPHATASE			LSD <0.05 between dates	ALKALINE PHOSPHATASE
			after 6 days	after 13 days	after 27 days		after 6 days
			(ug/g soil/hr) (1)				(ug/g soil/hr) (1)
Ste-Rosalie	CON	0.0	328	298	338	57.0	35
	COM	2.5	315	312	354	46.1	45
	COM	5.0	326	317	388	36.2	53
	FPM	2.5	519	422	396	68.0	65
	FPM	5.0	483	425	432	73.1	62
	SCU	2.5	327	291	355	55.7	55
	SCU	5.0	350	303	356	58.4	64
	SBC	2.5	336	367	392	44.0	46
	SBC	5.0	309	319	346	76.1	44
	LSD <0.05 between residues		60.3	33.0	53.1		13.5
Chicot	CON	0.0	97	130	106	31.9	35
	COM	2.5	119	123	125	65.8	58
	COM	5.0	112	128	142	33.9	45
	FPM	2.5	146	80	190	36.6	65
	FPM	5.0	172	114	202	47.3	40
	SCU	2.5	105	130	129	24.5	49
	SCU	5.0	90	100	152	28.9	52
	SBC	2.5	107	76	146	29.7	35
	SBC	5.0	109	67	142	22.1	45
	LSD <0.05 between residues		24.7	37.8	33.5		15.3

(1) ug p-PN/g soil/hr = all data are expressed as micrograms of p-nitrophenol per gram of soil per hour.

The mechanisms causing P fixation and reducing the extractability of P in soils amended with compost, FPM and SCU are unknown. The results are not explained by precipitation of P with Ca in the residues because extractable Ca/P was twice as high in SCU as in SBC. The reaction of P and Ca is modified by organic matter, as observed by Perrot (1992) with other organic materials and hence variation in fixation between residues may relate to variation in their organic matter contents. Evidence from other incubation studies (Mnkeni and MacKenzie, 1988) suggests, however, that added organic matter should reduce fixation, which appears to be contrary to our results, although net desorption/dissolution of P occurred in the high COM treatment on one soil.

The fixation is also not clearly related to microbial demand for P because the fixation was high in compost, the residue with the lowest CO<sub>2</sub> evolution. The increased extractability of P in the SBC treatments during the incubation may represent a change in the chemistry of SBC-P caused by incubation at a pH much lower than that of the residue alone. This mechanism is consistent with the differences in pH between the soils, because net dissolution/desorption with SBC was higher at pH 5.0 to 5.2 in the Ste-Rosalie soil than at pH 6.1 to 6.3 in the Chicot soil. Extractable soil P increased with amendment more significantly in Chicot than in Ste-Rosalie, with the exception of SBC. This may have been related to soil pH, which is a determinant factor of phosphorus availability (Kolar et al, 1992 and Holford and Doyle, 1993) that reaches maximum availability at pH between 5.5 and 6.5. Overall, the results suggest that Mehlich III extractable P in residues prior to application is not a good predictor of the extractability of the residue P added to soil, except for SCU.

The behaviour of the residues with respect to soil chemical parameters differed between the two soils in many respects, in particular, the extractability and fixation of P added in the SCU and SBC residues. These results suggest, that soil-specific testing will be required to allow the prediction of the effects of field applications of these residues as P sources as has been proposed by William (1971) for the use of superphosphate in different types.

### **Residue effects on microbial activity**

The addition of residues high in organic matter to soil has the potential to stimulate microbial activity and reduce the availability of soil N to plants by microbial immobilization of ammonium and nitrate. This effect was observed to some degree with all residues on both soils, with the exception of SBC and COM in the Chicot and Ste-Rosalie soils, respectively, in which nitrification appeared to be stimulated. Nitrogen immobilization was greatest in the FPM treatments where ammonium and nitrate concentrations were reduced by up to 41 % and 88 % respectively. This result is consistent with the high C:N ratio of this residue and the high level of CO<sub>2</sub> evolution it supported. Wood (1981) also found immobilization of N in filter cake material when C:N ratio was above 20:1. The compost also had a high C:N ratio but supported less microbial activity and immobilized less N, presumably because the easily degraded organic components of the residue had been consumed during the composting process. SBC had a low C:N ratio and thus, this residue would not have been expected to cause N immobilization (Wood 1981). The immobilization observed may have been a result of the highly labile nature of the organic material in the residue. With the exception of the FPM, the degree of immobilization was low, despite the high application rates, suggesting that a reduction in N supply is unlikely to be an agronomic problem with residue applications at field rates. Overall, loss of N from plant available pools due to microbial immobilization caused by FPM and SCU is expected to be of short duration because of the rapid depletion of labile C sources.

Application of organic residues to agricultural soils has the potential to increase soil organic matter contents (N'Dayegamiye and Dubé, 1986) with beneficial effects on tilth and nutrient cycling, over the long-term (Angers et al., 1993). The large proportion of the added C evolved as CO<sub>2</sub> during the experiment in FPM and SBC treatments suggests that application of these materials is not likely to cause a buildup of C in the soil.

The results for consumption of added C show that FPM stimulated microbial activity and increased enzymatic activity as well.



Microbial production of phosphatase is stimulated under conditions of high microbial demand for P such as under high carbon availability with low concentration of orthophosphate (Juma and Tabatabai, 1978, McGill and Cole, 1981). Our data showing increased levels of phosphatase in the FPM treatments and, to a lesser extent, in the COM treatments are consistent with this mechanism as both of these treatments had low extractable P. In contrast, SCU and SBC treatments had high levels of P which presumably were sufficient to meet microbial demand. Perruci (1990) suggested that added available C in municipal solid waste compost stimulates the proliferation of phosphatase producing organisms. In addition, the irregular and porous nature of diatomaceous earth in FPM may provide better soil aeration than the of the other residues, thereby insuring a better distribution of oxygen in the soil.

Differences between the two soils were observed in the response to residue addition of several of the parameters relating to microbial activity. In particular, the proportion of added C consumed as  $\text{CO}_2$  was higher in the Ste-Rosalie than in the Chicot soil,  $\text{NH}_4\text{-N}$  concentrations tended to be reduced more by addition of residues in the Chicot than in the Ste-Rosalie soil. The acid phosphatase had a more dynamic response to residues in the Chicot than in the Ste-Rosalie. The results support the conclusion that the effect of the residues on soil microbial activity varies with soil conditions and the soil - specific data are required in order to predict the effects of residue applications.

#### **Residue effects on soil cations and pH**

Although of lesser practical significance than the results for P and N, addition of residues other than FPM increased extractable Ca substantially, particularly in the Ste-Rosalie soil. Hence, application of the wastes could be considered an agronomically important source of Ca on acid soils, such as podzols in southern Quebec. Such soils would also benefit from the increase in pH observed in SCU and SBC treatments. The SCU treatments augmented extractable Mg levels slightly, which may have agronomic importance on low Mg soils.

Prassad (1976) recommended the use of filter cake (a combination of FPM and SCU) as a source of K for sugarcane. Our data showing minor effects of residues on soil K are inconsistent with this previous work, with the discrepancy due to differences in the processes used during sugar refining. In the case of the Lantic refinery, no chemical sources of K are used in the refining process, there are, therefore no statistical differences of K to be noted between treated and untreated soils. SCU, when applied at a high rates can be considered as a source of Mg, although quantities supplied are low.

### CONCLUSION

In summary, clay content and pH of soils had a direct effect on available P and Ca content at the end of the period of incubation of each residue. SCU appeared to be the treatment supplying the greatest concentration of P and Ca to the soil, and supporting the highest level CO<sub>2</sub> evolution. However, a large proportion of added extractable P became fixed during incubation of treated SCU soils. FPM did not appear to be a good source of macro-elements, but the high concentration of immediately available C and the coarse physical nature of FPM caused a stimulation of microbial and enzymatic activity under incubation conditions. SBC supplied a high concentration of P in amended soils, whereas acid phosphatase was inhibited by increasing the rate of application of SBC. Mellich III procedure applied on the residue appeared to be not an adequate indicator of the P extractable after SBC is applied to the soil. Compost made with a 50% mixture of SCU and FPM showed a poor performance with respect to biological parameters (enzymatic activity and CO<sub>2</sub> evolution), due to its high C:N ratio and the rate of fixation of P was greater than that of SCU and SBC.

In general, the results of this study showed that the residues, with the exception of FPM, increased extractable P levels in soils and hence could be used as phosphate fertilizers in agriculture. The residues stimulated microbial activity which resulted in increased phosphatase activity and N immobilization but these two effects are relatively small and unlikely to be agronomically significant at field application rates.

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## CONNECTING SECTION

The incubation study demonstrated that sugar refinery by-products have potential as soil enriching or fertilizing agents especially as a source of phosphorus and/or calcium. They also promote enzyme and microbial activities at agronomically significant levels. A second study was carried out under field conditions to determine the comparative agronomic impacts of different forms of Lantic sugar refinery by-products and their influence on phosphorus availability and uptake in wheat and corn crops.

**CHAPTER 3. AGRONOMIC VALUE OF SUGAR REFINING BY-PRODUCTS  
AND THEIR COMPOST ON ACID CLAY SOIL**

## ABSTRACT

Sugar refinery by-products, namely spent bone char (SBC), containing high levels of total P and Ca, a mixture (MIX) made up of filter press mud (FPM) and clarification scum (SCU) residues and compost COM produced from the previously mentioned residues were evaluated under field conditions. These residues were compared to a commercial fertilizer, triple superphosphate (TSP) and to controls (CON) which did not receive phosphorus treatments. This was done using a randomized complete block design in two crops, wheat and corn. The experiment was conducted over a two-year period (May 1993 to November 1994) in the Montréal area, Québec, Canada. Soil extractable  $\text{NH}_4\text{-N}$ ,  $\text{NO}_3\text{-N}$ , P, K, Ca, and Mg and OM, pH and wet aggregate stability (WAS) were determined as well as crop yield and N, P, and K concentrations in plant tissues.

Temperate, heavy clay, low pH soil conditions resulted in fixation of P in the applied residues. Several problems were encountered such as water accumulation over some corn blocks and a weed invasion in 1994. They resulted in reduced yields in 1994. Contrast analysis showed that TSP treatments did not differ from the control in soil P or in concentrations of all nutrients in tissues. Treatments did not significantly increase the Ca levels in corn plots nor did they increase the WAS in soils under either crop. On the other hand, soil OM under both crops was raised by the use of MIX and COM, while SBC did not have a clear effect on soil OM by the end of a second year of application. COM increased the OM content of the soil relative to TSP. A second COM application caused net N immobilization in soil under corn and significantly increased N content over controls in both corn tissues (grain and stover). In corn, MIX applied at three times the recommended  $\text{P}_2\text{O}_5$  rate significantly increased soil Mehlich III P relative to control soils, where P concentration in corn stover decreased versus TSP1. The opposite was observed in wheat grain. SBC applied at the recommended  $\text{P}_2\text{O}_5$  rate raised P concentration in corn grain over TSP at the end of 1993. A residual effect of P added in SBC was apparent in soil from CON, TSP1 and TSP2 plots where the cumulative fertilizer effect was superior to the control. The results of this study support the laboratory observations indicating that



SCU and SBC application would increase soil extractable P but showed that only SBC had an effect on plant P. content.

Key words: Sugar refinery by-products, P availability, Compost, Spent bone char, Heavy clay soil, Low soil pH.

## RÉSUMÉ

Les sous-produits de raffinage de sucre, soit les poussières de noir animale (SBC), riche en P et Ca total, un mélange (MIX) composé de boues de filtre-presse (FPM) et d'écumes de clarification (SCU), ainsi qu'un compost (COM) produit à partir des deux derniers résidus ont été évalués sous condition de champ. Ces résidus ont été comparés à un fertilisant commercial, le TSP et à des parcelles témoins (CON) lesquelles n'ont reçues aucune source de P. Cette expérience a été réalisée selon un design par blocs complets randomisés sur deux cultures, du blé et du maïs. L'expérience s'est déroulée au cours de deux saisons de croissance, soit entre mai 1993 et novembre 1994 dans le région de Montréal, Québec, Canada. Les paramètres chimiques analysés au sol ont été:  $\text{NH}_4\text{-N}$ ,  $\text{NO}_3\text{-N}$ , P, K, Ca, Mg extractible, la matière organique (MO), le pH ainsi que la stabilité des agrégats humides (WAS), de même que les rendements et la concentration de N, P, et K dans les tissus des plantes.

Sous des conditions de climat tempéré, d'argile lourd et un sol à bas pH, on a observé le niveau de fixation du P suite aux apports des résidus appliqués au sol. Quelques problèmes sont survenus au cours de l'expérience, notamment l'accumulation d'eau sur certaines parcelles de maïs et l'envahissement de mauvaises herbes en 1994. Ces conditions ont été perçus par la réduction notable des rendements de 1994. L'analyse par contraste a démontré que les parcelles traitées au TSP ne se sont pas différenciées en P obtenu par l'extractant Mellich III par rapport aux parcelles témoins CON, de même que la concentration tous les éléments nutritifs dans les tissus. Aucun traitement n'a rehaussé significativement la teneur en Ca dans les parcelles de maïs, ni n'a augmenté WAS dans les sols des deux cultures. D'autre part, la MO des sols a augmenté dans les sols des deux cultures suite à l'apport de MIX et COM, alors que SBC n'a pas démontré de tendance sur le niveau de MO au terme de la seconde année d'application. COM a augmenté le contenu en MO du sol relativement à TSP. Une seconde application de COM a causée une nette immobilisation de N dans les sols des parcelles et a significativement augmenté le contenu en N par rapport aux témoins dans les deux tissus

de maïs (grains et tiges). Dans le maïs, MIX appliqué à trois fois les taux recommandés de  $P_2O_5$  a significativement augmenté l'extractant Mellich III en P des sols relativement aux parcelles témoins, alors que la concentration en P dans les tiges de maïs a diminué versus TSP1. L'effet inverse a été observé dans les grains de maïs. SBC appliqué à une fois le taux recommandé en  $P_2O_5$  a augmenté la concentration en phosphore dans les grains de maïs par rapport au TSP au terme de la saison de croissance 1993. L'effet résiduel de la teneur de P au sol a été observé suite à l'application de SBC par rapport aux parcelles de CON, TSP1 et TSP2, alors qu'un effet cumulatif du fertilisant a été largement supérieur aux contrôles. Les résultats de cette étude supportent les observations des analyses en laboratoire (chapitre 2), lesquels indiquaient que des applications au sol de SCU et SBC devraient augmenter le P extractible du sol, alors que l'on a pu observer un effet de SBC seulement sur la concentration en P dans les tissus des plantes.

Mots clés: Sous-produits du raffinage du sucre, P disponible, Compost, Poussière de noir animale, Sol argileux, Sol à bas pH.

## INTRODUCTION

Lantic Sugar Ltd, located in Montréal, Québec, Canada generates three residues in large quantities. The clarification by phosphatation process used to refine sugar produces by-products having the potential for high agronomic value because of the different chemical and organic components present in it. Incubation test results on each residue (chapter 2) demonstrate their value as soil enriching and fertilizing agents.

Clarification either by adding phosphoric acid ( $P_2O_5$ ), hydrated lime ( $Ca(OH)_2$ ) with a polymer, Taloflote, or by induction heating causes the precipitation of solubles and colloidal impurities (Chen, 1985). Addition of phosphoric acid and lime neutralises the organic acids found in raw sugar, causes the formation of calcium phosphate (Paturau, 1989) and generates end products reformed to as clarification scum (SCU).

Under pressure, liquors pass into a filter-press where calcinated diatomaceous earth is used in the presence of alkali (Celatom). The mixture of earth, retained particles and an inert filtering aid used in small quantities to enhance filtration, are all components of filter-press mud (FPM) (Chen, 1985).

Thus, the clarification by phosphatation generates two residues (FPM and SCU) which combined are referred to as filter cake, filter-press cake, filter-mud, scums, etc according to the author or sugar plant country of origin (Alexander, 1972). These residues are mixed together for disposal.

Black bone char is the most commonly used adsorbant to bleach and purify liquors in the refining of sugar (Chou and Hanson, 1971) and must be replaced on a monthly basis at the Montréal plant. Repeated handling reduces its drying properties and transforms it by attrition into dust, subsequently called spent bone char (SBC). At the Lantic refinery, the SBC is composed of carbonate and tricalcium phosphate with organic impurities.

Results from the incubation experiment (chapter 2) show that each residue has characteristics that may be beneficial in agricultural applications. In the previous experiment (chapter 2), SCU and FPM were incubated separately. In order of importance, the use of SCU, SBC and COM has increased the soil concentration of P and Ca. The use of FPM did not increase the content of soil macro-elements, but clearly contributed instead to the development of the microbial and enzyme activity in the soil. High P fixation appeared in soil treated with SCU and SBC through a distinct mechanism. Fixation also occurred moderately in COM and FPM treated soils.

The large majority of work undertaken regarding the agricultural use of sugar refinery residues has been carried out on sugar cane production. Rao (1983) presented a list of nutrient content of FPM from different countries compiled from many publications reporting that the  $P_2O_5$ , Ca and N percentages varied from 0.45 to 2.81, 1.05 to 3.76 and from 0.005 to 2.80 respectively. Variations were attributed to cane variety, native soil type, the amount and type of fertilizers used, the climate as well as factory performance.

From past experiments on the subject, many authors (Paul, 1974; Kuong et al., 1988; Prasad, 1976; Cooper and Abus Idris, 1980) concluded that FPM is a good fertilizer and a soil enriching agent. On the other hand, each author had reservations because of the lack of equilibrium between the different macro-elements, some of which, such as the N, show up in non-available forms and because of a C:N ratio superior to 20 in some cases.

Prasad (1976) demonstrated that FPM was a more effective source of P than triple superphosphate (TSP) at field rates, and that FPM could be used as a substitute for K fertilizer where a maintenance application is required. According to Eastwood (1978, reported from Rao, 1983), ratoon yields, juice quality and soil pH effects, were higher with FPM application than on TSP treated plots. The similar results were obtained in Mauritania by Kuong et al. (1988).

Phosphorus solubility and availability of bone powder in water increases following its calcination and temperature elevation (Gupta, 1973), as is the case in the composting process (Mathur et al., 1987). This suggests an important phosphorus content of SBC available for crops. In fact, SBC P availability is superior to what it would be expected, based on Mellich III extraction values (chapter 2).

Application of P index to an individual field site requires input regarding anticipated P addition from fertilizers. Calculating average crop P removal could serve as a P predictor for recommendation programs, as proposed, with restrictions by Fixen and Grove (1990). This is based in the assumption that P fertilizer applications will balance P removal (Pierzynski and Logan, 1993). This assumption is, however, a function of the type of cultivar and soil texture.

Plant macro-nutrient concentrations in grain and their uptake are influenced by corn heat unit and water input, especially grain N concentration (Liang et al, 1993). It has long been established (Glover and Mertz, 1987) that N (or crude protein) is used as a measure of grain quality. As opposed to grain-N content, balanced P and K (Fageria et al, 1991) are necessary nutrients, but do not directly account for corn and wheat grain quality. Generally, wheat and corn grain contains more N than any other soil-derived nutrient. More K is found in the corn stover than in the grain (Shaw, 1988), where K and N needs are often superior than that of P. From Liang et al (1993) results, about 85 % of total P uptake is found in the corn grain where K uptake is mainly found in stover.

Different responses of grain P uptake are dependent on climatic conditions. Heat units, water availability and soil type affect P availability, especially when P soil concentration is low (Tisdale et al, 1993). By increasing the water content in the root-zone, the diffusion rate of P increased accordingly. On the other hand, massive P applications by fertilization enhances P diffusion, which is limited, however by the rapid root growth (Barber, 1984). It follows that the moisture level in the soil does not influence the P uptake.

In accordance with results from Kuo and Jellum (1987), seasonal variations in pH are correlated with water-soluble P, which suggests that solution P can control soil pH under field conditions (Xie and Mackenzie, 1990). According to Khasaweh et al. (1980), a phosphated fertilization source applied in a matrix with a high level of organic matter enhances the availability of P by creating an orthophosphate compound easier to assimilate by the roots. Chen et al. (1987) demonstrated in experiments conducted in greenhouses that P combined with a nitrogen source (urea) raises soil pH and  $\text{NH}_3\text{-N}$  concentrations around fertilizer particles. This results in a dissolved organic matter fraction and thus raises P availability for the plants.

The behaviour of the Lantic sugar refinery residues as soil enriching agents in agriculture applications is somewhat uncertain. Their P and Ca values and their impact on the microbial activities have been established in Chapter 2. Their effects on yield of annual crops under temperate field conditions, as well as the residual effects on the treated soil remain to be validated.

The supply of organic materials as compost could lead to positive impacts on soil chemical and biological parameters and to improved soil properties (Avnimelech and Cohen, 1988). According to Angers and Mehuys (1993), aggregate soil stability is a measurement that predicts the resistance of the bonds within soil aggregates to external forces of impacts, or disruption arising from the escape of entrapped compressed air. Aggregate stability is highly dependent upon soil humus content and somewhat dependent on soil texture. From the available methods existing, wet aggregate stability (WAS) was chosen to quantify this parameter.

Rasaih et al. (1992) suggested that clay, OM contents and pH of soils explained 80 % of the variability in the WAS sensitivity. According to these authors, the sensitivity of WAS increased with increasing clay and OM contents. Decreasing pH and its associated soil properties together with water content explained 63 % of the variability in WAS.

Drury et al. (1991) observed that wet aggregate stability was negatively correlated with soil water content, independently of cropping management indicating that improvements in structure were not solely the result of the cropping treatments and associated rhizosphere populations. Perfect et al. (1990) observed that structural stability decreased with increasing soil water content ( $r = 0.74$ ), where soil moisture and microbial biomass (in that order) were significant predictors of structural stability within the growing season.

As previously mentioned (chapter 2), sugar refinery processes use cationic and anionic polymers for coagulating and precipitate organic substances. Gu and Doner (1993) investigated the influences of three organic polyanions (a soil humic acid, a soil polysaccharide, and a commercial anionic polysaccharide) on soil clay dispersion and soil aggregation. This study indicates that soil colloidal stability is strongly influenced by soil organic components in addition to factors such as pH, ionic strength and composition. In the absence of polyvalent cations, negatively charged humic substances, especially humic acid, may not contribute to stable soil aggregation, whereas the presence of both polyvalent cations and polyanions may be cumulative in preventing soil clay dispersion.

The effect of aliphatic compounds, as used during the refining process, on the water aggregate stability of a chemically altered lacustine heavy clay was studied in incubation experiments by Diné et al. (1992). Addition of insoluble aliphatic compounds produced  $\text{CO}_2$  and increased water-stable aggregates significantly. Such information indicates that the increase in soil aggregate stability appears closely related to bound aliphatic compounds incorporated into the aggregates as a result of microbial activity, and that the effect of aliphatic compounds on soil aggregate stability is associated with both their quantity and chemical nature. Since our incubation tests (chapter 2) for each Lantic residue showed an increased content of microbial activity and raised C evolved as  $\text{CO}_2$  from control, it follows that the addition of these residues, especially the ones high in OM (COM and MIX), may significantly increase WAS.



The sugar refining activities are carried out year long thus generating a steady stream of the three residues. Their use on crops is thus impossible without the intervention of a stabilizing process. To simulate real conditions, we composted SCU and FPM in accurate relative proportions. The resulting compost will be used as an enriching soil agent for the following year crops.

Decomposability of organic residues is dependant on their chemical constituents and physical configuration which in turn determines their efficiency when added to the soil (N'Dayegamiye et Angers, 1993). In general, wood residues such as tree clippings have high lignin content and C:N ratio ( $>90$ ) (Beauchemin et al, 1990). In the case of our compost used in the 1993 crop season, the C:N ratio was 44:1 and for 1994, C:N ratio was 17/1 (Table 1). As observed by N'dayegamiye and Dubé (1986), a net immobilization of N appends the year following it's application. They also observed a net decrease in yield for wheat, corn and potatoes on the first application year with substantial increases in the subsequent 5 years following that unique application. The present experiment being limited to two years of observation, it is conceivable that benefits from the use of compost will not be observed.

Beauchemin et al. (1992) found that one year is sufficient to obtain a stabilized and mature composted material from yard trimming compost produced outside by the returned windrow technique. The authors indeed found that a large quantity of mineralizable C always remained available. Thus, the addition of tree clippings monopolises nitrogen and immobilises it for the decomposition of C by micro-organisms.

It is to answer some of these questions that the following experiment was conducted. The general objectives of the research were to evaluate the direct field application of clarification scum mixed with filter-press mud (MIX) and once composted, as well as SBC, and to compare the behaviour of theses residues on crop growth with a known P source (triple superphosphate) and with a control plot with no P added.

**Table 1. Chemical analysis of sugar refining by-products and their compost (dry matter basis)**

Descriptors	FPM	SCU	SBC	COMPOST-93	COMPOST-94
1 soil : 1 water					
pH	6.4	7.3	7.7	7.3 (2)	7.7
%					
H <sub>2</sub> O in fresh residues	50	66	5	59 (1)	36
O.M.	9	43	7	17 (1)	25
C/N	64	21	9	44	17
ms/cm					
Electrical conductivity	1.29	1.14	0.98	---	1.08
mg/kg					
Total N	840	11800	4810	3800 (1)	8550
Total P	1250	81800	153100	29300 (1)	17800
Extractable P (Mellich III)	480	6561	1405	6860 (1)	4160
Total K	410	2461	6379	1380 (2)	1570
Extractable K (Mellich III)	120	75	42	---	730
Total Ca	35900	24400	355000	10700 (2)	150000
Extractable Ca (Mellich III)	3050	29600	14200	---	20800
Total Mg	470	8763	1550	1900 (2)	12540
Extractable Mg (Mellich III)	150	4370	556	---	2510

(1) = Chemical analysis provided by Nutrite inc. Brossard, Qc, Canada. J4Z 3N2

(2) = Chemical analysis provided by CreaLab, Qc, Canada. J1N 2T3

The specific objectives were to determine (1) the effect of the rate of application of the different treatments on several parameters, especially those related with the P availability in the soil; (2) the impact of the residues on N, P and K concentration in wheat (*Triticum aestivum*, L.) and corn (*Zea mays*, L.) tissues harvested in 1993 and 1994, their yield and the P uptake; (3) the cumulative or residual effects of residue application on different soil (P, Ca, OM, pH and WAS) and tissue (N, P and P uptake) parameters and (4) if the treatments containing phosphated agents significantly raises the P levels compared to the control (CON) which did not receive any external phosphated sources and TSP treated plots. This evaluation was conducted for the two years of the experiment (1993 and 1994) and results were compared between plots that received a second year application with plots that did not receive an application in 1994.

## MATERIAL AND METHODS

The designated experiment sector is the area south of Montréal in which the clay soil with low pH is characteristic of the St-Lawrence river valley. This site, adjacent to the residue production facility has been chosen because it offers a viable waste management alternative for Lantic. Two crops have been identified and were consistently used and compared throughout this experiment. They are corn grain and wheat which account for two thirds of this region's production with the exception of forage crops and prairies (BSQ, 1990).

Experimental plots were established in May 1993 at Saint-Basile-le-Grand, Québec, Canada on an orthic humic gleysol of clay texture, from the Providence series (Canadian soil classification system, Agriculture Canada, 1990). The actual field lay out respected the experimental plan of a randomized complete bloc design and the blocs were aligned with the underground drainage network to eliminate any variations induced by that network. The entire experiment (wheat and maize plots) did not require more than 2 hectares in total.

Ten (10) treatments including a control, designated CON, where no phosphate was applied, were replicated four times for both crops. Phosphorus sources were spent bone char (SBC), the residue mixture SCU-FPM, designated MIX, combined in a volumetric ratio of 2 to 1 and the composted mixture designated COM.

Physico-chemical characteristics of Lantic sugar by-products and the two compost used in this project (1993 and 1994) are presented in Table 1.

The following differences in the compost (COM) characteristics for the two years of the experiment was the consequence of variations in the input material, combined with the different techniques used; aerated static piles in 1993 and turned windrows in 1994. In 1993, MIX was blended equally with a mixture consisting of 3 wood shavings parts to 1 septic sludge part, screened at 2 cm to form compost (COM; C:N = 44:1). In 1994, MIX was blended equally with leaves and yard trimmings to form compost (COM; C:N = 17:1). In that instance, the leaf nerves were not degraded. This compost was not screened prior to application.

The triple superphosphate (TSP) commercial fertilizer served as a reference P source through the experiment. The quantity of each P source applied to the soil was always a function of the theoretical available P concentration, except for TSP.

Thus, the ten (10) parcels from each bloc received phosphated sources from the different treatments, at application rates of 60 kg ha<sup>-1</sup> and 120 kg ha<sup>-1</sup> of P<sub>2</sub>O<sub>5</sub> for wheat and 80 kg ha<sup>-1</sup> and 160 kg ha<sup>-1</sup> of P<sub>2</sub>O<sub>5</sub> for corn. These application rates correspond to 100 % and 200 % of recommended levels (AFEQ, 1990) and were chosen in function of soil type and indigenous P levels in the soil for each crop. One plot per bloc for each crop received 240 kg ha<sup>-1</sup>, or 300 % of recommended P<sub>2</sub>O<sub>5</sub> levels of MIX. The Messier wheat variety was used both years while corn hybrid DeKalb 2600 and 2300 corn heat units (CHU) were used in 1993 and 1994 respectively. The equivalent quantity of residues applied as P source is presented in appendix, Table B-1.

Sowing was done on June 7, 1993 and June 1, 1994 for both crops. Prior to the experiment, the site was growing grain corn. Two passes of chisel plowing to a depth of 10 centimetres, preceded the addition of residues and was followed by a finishing passage of common finishing harrow equipment in conformity with the land owner's practices. The size of each plot was 5 x 8 meters for wheat and 5 x 6 meters for the corn crop.

In 1994, the experiment was carried out under the same conditions as in 1993, with one exception. Each parcel was divided in two and one half, randomly selected, did not receive phosphorated material from any source while the other half received a treatment identical to the previous year. This was necessary to evaluate the residual versus the cumulative effect of the P treatment on the soil as well as some crop tissues (wheat and corn grains and corn stover). Nitrogen and K ( $K_2O$ ) were also applied at 130 and 40 kg  $ha^{-1}$  in wheat and 240 and 100 kg  $ha^{-1}$  in corn respectively from ammonium nitrate (33,5-0-0) and muriate of potash (0-0-60). Corn plant population was 89000 plants  $ha^{-1}$  and sowed with a two row corn planter. Wheat was seeded at recommended plant population using the Case-International grain seeder owned by the land owner.

The wheat parcels were treated with Buctril herbicide (MCPA, 1L  $ha^{-1}$ ) on June 30 1993 while the corn parcels were treated using a blend of Laddock (Benzatone and Atrazine at a rate of 200g  $L^{-1}$  and 4L  $ha^{-1}$ ) and Assist (2 L  $ha^{-1}$ ) on July 2, 1993. In 1994, a late application of the Laddock and Assist blend was performed by the land owner. No herbicides were applied to the wheat parcels in 1994.

The wheat crop was harvested on September 1, 1993 and August 15, 1994. The corn crop was harvested on November 7, 1993 and October 25, 1994.

#### **Soil sampling and analysis.**

A total of 40 composite soil samples for both crops were taken to a depth of 15 centimetres before any treatment was applied in 1993 (sample set 93a), at the first harvesting in 1993 (sample set 93b) and the second harvest in 1994. Samples for the half

plot that received no additional P source are referred to as 94c and 94d half plots that received a second year treatment for a total of 80 composite samples in 1994 are designated 94d. The samples were then air dried for three days before being ground to pass a 2 mm seive and analysed.

Soil samples used for determining the wet aggregate stability of corn and wheat plots were taken at the wheat harvesting of August 16, 1994. They were analysed using the method proposed by Angers et Mehuys (1993).

Soil extraction of P, K, Ca and Mg was conducted according to Mehlich III (Mehlich, 1984). Ammonia and nitrate were extracted using the technique developed by Keeney and Nelson (1982). The analyses of available P and K, and of the nitrate and ammonia were done with Quick Chem Methods of Lachat Instruments. Ca and Mg levels were measured using an atomic absorption spectro photometer (Perkin-Elmer AS-1). The organic matter content was measured using the procedure developed by Nelson and Sommers (1982). Soil pH was determined using the method of Black (1965).

WAS (wet aggregate stability) was determined in a 10 g soil sample aggregate air-dried, spread on a 250 mm mesh sieve and on a wet sieving apparatus, which was lowered to the water surface, washed, dried, weighed and mixed with 50 ml of 0.5 % Na-hexametaphosphate and washed again on 250 mm sieve. The primary particles were then collected from the sieve and dried. A subsample of 1 to 2 mm aggregates was used to measure gravimetric water content (Angers and Mehuys, 1993).

#### **Tissue sampling and analysis**

Wheat and corn grain as well as corn stover was harvested in a representative manner from each parcel to determine the N, P and K levels. Wheat grain was harvested right across the combine (2.5 meters width) along the whole length of the sub-plots (5m). Ten corn stalks with cobs plus ten other corn cobs within each sub-plot were harvested by hand.

Grain was weighted before and after drying in order to determine the surface unit yield (hectare). Samples were then grounded and sieved to 1 mm for further analyses. Tissue samples were digested in 4 ml of  $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$  solution following a modified technique proposed by Thomas et al. (1967). Total N, P and K were analysed using an auto analyzer Quick Chem Method of Lachat Instruments.

### Statistical analysis

Results were submitted to analysis of variance. All comparisons were carried out using Statistical Analysis System for Windows (SAS Institute, 1990). The experimental arrangement and statistical analysis were analysed as a randomized complete bloc design.

The total pool of samples for both crops consisted of 4 series of results for soil; spring 1993 before treatment (93a), fall 1993 following the first application of amending agents (93b), fall 1994 taking into account the parcel halves which did not receive a second application (94c) and those that did (94d). Same series determination was used for the tissue samples of grain and stover, although the "e series" represents plot halves that did or would not receive additional residues in 1994 while the "f series" represents halves (sub-plots) that did or would receive residues in 1994.

ANOVA's were carried out according to PROC GLM for all series. Previously, covariance and repeated measures techniques were used and yielded similar results. Because of some missing values, especially for 1994, some tests could not be carried out using these statistical techniques.

Contrasts were done to compare differences between residue treatments at all rates of application versus control plots (CON) and plots treated with TSP at 1 and 2 rates (TSP1 and TSP2 respectively).

Testing cumulative and residual effects of treatments was done by simultaneous contrasts in a two-way classification. In order to obtain variation among particular residues, rates and time, residues and rates were linked to give treatments (TREAT) and interacted with

series (93a, 93b, 94c and 94d for soil data and 93e, 93f, 94e, 94f tissue data). A test that took into account two series at a time for the comparison of each residue at different rates of application with control and TSP1 and TSP2 was constructed (SAS, 1991).

Contrast analysis was performed on soil and tissue data, where results highlight the differences between series (two sampling periods) for each treatment versus CON and TSP1 and TSP2. Probabilities test the tendency of a treatment to affect a given parameter following its application.

## **RESULTS AND DISCUSSION**

### **Constraints on interpretation**

Before discussing results, several events that occurred during the two cropping seasons that may have introduced variability into the experiment and prevented us from detecting treatment effects in the statistical analysis. Firstly, rain delayed seeding in 1993 and may have substantially limited the growth of both crops. The rain made the site access impossible until May 21 1993. A 2600 CHU hybrid was used but didn't reach full physiological maturity in 1993. Secondly, the fine texture of the soil created an impervious layer making it impossible to work the field after a single rainfall.

Even with the great care taken to level the parcels, the furrows left behind by equipment became small ponds for days following rainfall. The existing underground drainage network did not allow surface water to be removed because of the very low permeability of the indigenous clay soil found at the surface. Yield variations between the two years for both crops were readily noticeable. The furrows and the underground drains being aligned with the blocks, (replicates) we can link the variability between replicates to this drainage problem. Despite the low soil pH before the 1993 treatments, no lime was applied on the soil prior to the experiment.

In 1994, ineffective herbicide application in corn and wheat resulted in crop losses due to weed competition although mowing between corn rows in August did limit the weed



invasion. Problems caused by weeds were observed all around the experiment sector in 1994 even with the use of herbicides. The weeds (*Setaria sp*, *Ambrosia sp* and *Chenopodium album*) competed with the wheat and especially the corn for the fertilizing elements.

Heavy machinery used to work the ground may, considering the relatively small size of the parcels, have transported material between parcels as well as enhanced soil compaction due to frequent passage in specific places.

Finally, at the end of the second year, there was observable non- degraded MIX at the time the soils were sampled and at the corn harvesting of 1994, even where material had been applied at the beginning of the 1993 season. On the other hand, all the other P sources (COM, SBC and TSP) were well decomposed and were to apparent in the soil within the cropping season.

Despite theses difficulties, however, significant experimental effects were observed in many of the parameters measured.

### **Soil physical and chemical characteristics**

In soil under wheat, the ANOVA results (Table 2) indicated that no soil chemical parameters showed significant differences between residues, rates and their interaction at the 5 % level of significance, with the exception of OM, while clear differences between series were observed. From the soil ANOVA results for both wheat and corn (Tables 2 and 3), the organic matter and soil pH varied significantly between replicates, series and their interaction.

Contrast analysis carried on soil descriptors in wheat plots (Table 4) showed that none of the treatments present marked differences in terms of soil pH and soil Ca versus TSP and CON. For all parameters, TSP did not differ from any by-products.

Table 2. Probability of treatment effects on soil moisture, P, K, Ca, Mg, NH<sub>4</sub>-N, NO<sub>3</sub>-N, OM, pH and wet aggregate stability of soil wheat plots (type III).

SOURCES	df	HUM	P	K	Ca	Mg	OM	pH	NH <sub>4</sub> -N	NO <sub>3</sub> -N	WAS
RES	4	0.4610	0.4460	0.0947	0.6649	0.6051	0.0716	0.3585	0.2837	0.6389	0.0460
PATE	2	0.7240	0.3470	0.7120	0.4757	0.6790	0.2054	0.4704	0.5800	0.5462	0.0229
REP	3	0.7070	0.6130	0.1310	0.4530	0.0281	0.0009	0.0001	0.0001	0.0583	0.0059
TIME (1)	2	0.0993	0.0043	0.0001	0.8129	0.0002	0.0001	0.0001	0.0001	0.0001	0.4116
RES*TIME	7	0.0255	0.8190	0.9660	0.1644	0.1444	0.7208	0.2880	0.9062	0.8755	0.7911
RATE*TIME	4	0.1960	0.4260	0.6600	0.7292	0.9225	0.7554	0.8732	0.7677	0.7455	0.3299
REP*TIME	6	0.3590	0.8730	0.1140	0.0118	0.0507	0.0032	0.0001	0.0001	0.0002	0.7999
RES*RATE	3	0.8260	0.7040	0.5100	0.9524	0.5464	0.0018	0.2554	0.6737	0.6983	0.0876
REP*RATE	6	0.9090	0.5440	0.7160	0.8360	0.3590	0.9996	0.1194	0.4335	0.8417	0.1596
C.V.		7.99	60.88	9.65	24.55	24.29	11.34	2.52	16.22	80.71	8.53
R-2		33.0%	40.0%	90.3%	36.5%	49.0%	61.7%	92.0%	97.0%	61.8%	51.0%

Shaded values represent significance at Prob &lt; 5% level.

RES = Residues; REP = Replicates; TIME = sampling time or series; RATE = rate of application for each residues.

df = Degrees of freedom

(1) = Time refers to series

Table 3. Probability on treatment effects on corn soil moisture, P, K, Ca, Mg, NH<sub>4</sub>-N, NO<sub>3</sub>-N, OM, pH and wet aggregate stability (type III).

SOURCES	df	HUM	P	K	Ca	Mg	OM	pH	NH <sub>4</sub> -N	NO <sub>3</sub> -N	WAS
RES	4	0.0143	0.0188	0.0138	0.0282	0.0137	0.0001	0.0001	0.0282	0.0004	0.2981
RATE	2	0.7550	0.0009	0.5390	0.3588	0.5665	0.0148	0.5253	0.2798	0.0825	0.3542
REP	3	0.2519	0.0010	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001
TIME (1)	2	0.1169	0.0957	0.0001	0.0001	0.0069	0.0001	0.0001	0.5041	0.0001	0.9870
RES*TIME	7	0.0001	0.3738	0.0025	0.0450	0.0099	0.6073	0.0298	0.0952	0.0001	0.6828
RATE*TIME	4	0.1452	0.0287	0.2506	0.1921	0.8725	0.0363	0.0434	0.0003	0.0400	0.6360
REP*TIME	6	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0001	0.0002	0.0001	0.9319
RES*RATE	3	0.0002	0.0060	0.0001	0.0096	0.2391	0.0010	0.0050	0.0952	0.6425	0.0023
REP*RATE	6	0.0894	0.0093	0.0001	0.1176	0.1163	0.0001	0.0001	0.0615	0.0691	0.3112
C.V.		9.40	37.84	6.83	31.27	25.75	11.16	2.57	27.85	63.21	13.01
R-2		42.5%	47.5%	89.9%	53.4%	63.7%	66.5%	80.7%	45.1%	63.9%	42.9%

Shaded values represent significance at Prob &lt; 5% level.

RES = residues; REP = Replicates; TIME = sampling time or series; RATE = rate of application for each residues.

df = Degrees of freedom

(1) = Time refers to series

Table 4. Probability of treatment effects on wheat grain N, P, K, Yield and P uptake (type III).

SOURCES	df	N	P	K	Yield	P uptake
RES	4	0.4243	0.4243	0.1070	0.2585	0.0323
RATE	2	0.1722	0.1722	0.8138	0.6182	0.0568
REP	3	0.0350	0.0350	0.9137	0.0102	0.0597
TIME (1)	2	0.0495	0.0495	0.0089	0.0001	0.0001
RES*TIME	7	0.2418	0.2418	0.6885	0.9877	0.7702
RATE*TIME	4	0.9713	0.9713	0.9399	0.6429	0.6362
REP*TIME	6	0.1187	0.1187	0.8778	0.8968	0.5168
RES*RATE	3	0.0292	0.0292	0.3961	0.0161	0.3339
REP*RATE	6	0.1867	0.1867	0.5138	0.1018	0.0318
C.V.		5.75	11.21	14.86	22.36	28.72
R-2		50.8%	40.8%	37.1%	92.8%	92.2%

Shaded values represent significance at Prob < 5% level.

RES= Residues; REP= Replicates; TIME= sampling time or series; RATE= rate of application for each residues.

df = Degrees of freedom

(1)= Time refers to series

## **Phosphorus**

The P levels for all treatments in all the plots were higher at the end of the 1993 growing season compared to 1994 (Figure 1). Referring to the soil ANOVA results (Table 2), only the series (time) as a source of variation influenced the model in the wheat section, whereas in corn (Table 3), residue, rate of application and time of sampling and their interactions affected extractable P concentration. Only the treatment\*series interaction did not show significant differences. In both crops, however, the  $r^2$  for extractable soil P indicated that the sources of variation included in the model account for a small proportion of the variability found in the data.

TSP did not differ from CON throughout the experiment in terms of soil P and all other soil variables. Thus, the differences between 80 and 160 kg of P ha<sup>-1</sup> applied and the 20 (1993) and 10 kg ha<sup>-1</sup> (1994) of P assimilated by corn grain and stover remain fixed in the soil, even after 2 years of application.

In corn, MIX3 significantly raised soil P compared to check plots over years (Table 6). This was observed at the end of the second experimental year when residual and cumulative effects were observed, but not at the end of the first year (93b). We can conclude that P from MIX requires more than one year to result in statistically higher availability than TSP, since no significant differences between MIX and CON, and between MIX and TSP appeared the first year of the experiment.

In wheat (Table 5), SBC remained similar to CON and TSP throughout the experiment, while in corn, SBC significantly raised the Mellich III P levels compared to CON and TSP2 (Table 6).

## **Calcium and Magnesium**

Generally, the treatments did not raise the level of Ca in either crop compared to the control (CON) (Table 5 and 6). The low Ca levels observed in 93b corn soils resulted in significant SBC1 contrasts in comparison with CON, TSP1 and TSP2.

Table 5. Probability of treatment effects on contrasts comparing soil wheat over time of sampling for each sugar refinery by-products.

Residue contrast	P			Ca			OM			pH		
	93a and 93b	93b and 94c	93b and 94d	93a and 93b	93b and 94c	93b and 94d	93a and 93b	93b and 94c	93b and 94d	93a and 93b	93b and 94c	93b and 94d
COM1 vs CON	0.9592	0.8411	0.9003	0.4253	0.0842	0.1928	0.0104	0.4796	0.3717	0.1070	0.7892	0.4891
COM2 vs CON	0.6682	0.9356	0.9900	0.5202	0.3289	0.0899	0.0992	0.6777	0.6518	0.2386	0.7870	0.5059
MIX1 vs CON	0.8967	0.7359	0.8859	0.3697	0.2835	0.0179	0.7395	0.7187	0.5555	0.8262	0.5891	0.6485
MIX2 vs CON	0.0258	0.0114	0.0042	0.8640	0.4722	0.6757	0.0006	0.1985	0.1334	0.1305	0.8394	0.8131
MIX3 vs CON	0.5477	0.1246	0.1638	0.3106	0.4951	0.1075	0.7246	0.9054	0.9621	0.0702	0.8262	0.9059
SBC1 vs CON	0.6350	0.9007	0.6642	0.3504	0.7704	0.4160	0.0360	0.8804	0.9243	0.0966	0.4579	0.5775
SBC2 vs CON	0.8524	0.5920	0.3310	0.5559	0.3559	0.6397	0.6547	0.9558	0.6518	0.1679	0.4579	0.5661
TSP1 vs CON	0.6043	0.6388	0.5851	0.1272	0.0506	0.3190	0.0026	0.4154	0.1024	0.2592	0.4891	0.4579
TSP2 vs CON	0.4084	0.7608	0.8142	0.7627	0.3889	0.5411	0.3272	0.9432	0.9684	0.1144	0.8394	0.9327
COM1 vs TSP1	0.6404	0.5029	0.5023	0.4428	0.8073	0.3684	0.6320	0.9400	0.4548	0.6244	0.3991	0.1530
COM2 vs TSP1	0.9285	0.5821	0.5766	0.3370	0.3001	0.6295	0.1571	0.6893	0.2348	0.9596	0.3367	0.3898
MIX1 vs TSP1	0.6976	0.4201	0.6872	0.5074	0.3495	0.8097	0.0069	0.2411	0.0272	0.3626	0.8792	0.7740
MIX2 vs TSP1	0.0064	0.0029	0.0008	0.1564	0.1922	0.0940	0.6547	0.6348	0.8929	0.6977	0.3715	0.6125
MIX3 vs TSP1	0.2640	0.0460	0.0538	0.5889	0.1796	0.5657	0.0073	0.3510	0.1126	0.4891	0.6364	0.5323
SBC1 vs TSP1	0.3216	0.5522	0.3279	0.5323	0.0812	0.1587	0.1826	0.3350	0.0843	0.8910	0.1530	0.8526
SBC2 vs TSP1	0.4816	0.3153	0.1304	0.3247	0.2751	0.0776	0.0095	0.4477	0.0381	0.8000	0.1530	0.8659
COM1 vs TSP2	0.3801	0.9171	0.9125	0.2516	0.3608	0.4677	0.1074	0.4176	0.3510	0.9730	0.7229	0.5435
COM2 vs TSP2	0.2105	0.8228	0.8239	0.3399	0.9040	0.2535	0.4989	0.6264	0.6236	0.6853	0.6364	0.9730
MIX1 vs TSP2	0.3391	0.9738	0.7051	0.2111	0.8243	0.0637	0.5165	0.7726	0.5823	0.1731	0.7356	0.5891
MIX2 vs TSP2	0.1553	0.0253	0.0084	0.6210	0.8809	0.8400	0.0121	0.1750	0.1236	0.9461	0.6853	0.7483
MIX3 vs TSP2	0.8210	0.2168	0.2461	0.1701	0.8506	0.2934	0.5293	0.9621	0.9306	0.8131	0.9865	0.8394
SBC1 vs TSP2	0.7241	0.8574	0.8422	0.1975	0.5508	0.8320	0.4477	0.9369	0.9558	0.9327	0.3452	0.5213
SBC2 vs TSP2	0.5213	0.8167	0.4604	0.3525	0.9482	0.8809	0.5932	0.8992	0.6806	0.8394	0.2	0.5155

(1) = Each residue applied at 1, 2 and 3 the application rate compared to control (CON), triple superphosphate (TSP) at 1 and 2 times the recommended P205 rate.

Shaded values represent significance at Prob &lt; 5% level.

93a = Soil sampling before treatments, 23 May 1993.

93b = Soil sampling at harvest time, September first 1993.

94c = Soil sampling at harvest time, August 15th 1994, on half plots that have not received additional by-products.

94d = Soil sampling at harvest time, August 15th 1994, on half plots that have received additional by-products.

Table 6. Probability of treatment effects on contrasts, comparing soil corn over time of sampling for each sugar refinery by-products.

Residue contrast	P			Ca			OM			pH		
	93a and 93b	93b and 94c	93b and 94d	93a and 93b	93b and 94c	93b and 94d	93a and 93b	93b and 94c	93b and 94d	93a and 93b	93b and 94c	93b and 94d
COM1 vs CON	0.6472	0.4974	0.6573	0.5783	0.6491	0.6009	0.0466	0.0062	0.0019	0.5977	0.0007	0.0014
COM2 vs CON	0.0161	0.2708	0.0017	0.8105	0.2132	0.3678	0.0052	0.0213	0.0857	0.8104	0.0704	0.3372
MIX1 vs CON	0.3597	0.4598	0.1269	0.0118	0.9242	0.7550	0.2164	0.2614	0.5985	0.0659	0.1348	0.1164
MIX2 vs CON	0.4359	0.4318	0.3853	0.5284	0.1625	0.2199	0.0005	0.0006	0.0001	0.4572	0.3622	0.1499
MIX3 vs CON	0.4718	0.0001	0.0343	0.5136	0.7564	0.8191	0.0274	0.2536	0.5768	0.3257	0.1999	0.6358
SBC1 vs CON	0.0284	0.4165	0.0282	0.0348	0.0083	0.0095	0.0001	0.0001	0.0001	0.8988	0.0561	0.0101
SBC2 vs CON	0.1981	0.0667	0.0007	0.6793	0.8458	0.8066	0.0042	0.0529	0.1099	0.8478	0.1292	0.1474
TSP1 vs CON	0.2290	0.4130	0.1761	0.1469	0.0891	0.0640	0.3616	0.4180	0.6215	0.0219	0.6890	0.9700
TSP2 vs CON	0.2533	0.5055	0.1986	0.0117	0.0902	0.0755	0.0582	0.1126	0.2254	0.4428	0.6914	0.5284
COM1 vs TSP1	0.0973	0.1590	0.0624	0.0453	0.0426	0.0139	0.2789	0.0677	0.0064	0.0049	0.0004	0.0100
COM2 vs TSP1	0.2248	0.8083	0.0580	0.0912	0.6329	0.3301	0.0582	0.1663	0.2009	0.0398	0.0373	0.3383
MIX1 vs TSP1	0.7854	0.9259	0.8382	0.0001	0.1222	0.0228	0.7360	0.7820	0.9660	0.6498	0.3073	0.9560
MIX2 vs TSP1	0.7094	0.9574	0.6233	0.4480	0.7377	0.5260	0.0085	0.0147	0.0001	0.1197	0.2149	0.1453
MIX3 vs TSP1	0.6280	0.0007	0.4245	0.4239	0.1912	0.0915	0.1933	0.7547	0.9467	0.1877	0.1147	0.6507
SBC1 vs TSP1	0.3189	0.9771	0.3707	0.5055	0.3987	0.4246	0.0031	0.0011	0.0001	0.0387	0.0297	0.0085
SBC2 vs TSP1	0.7208	0.3681	0.0307	0.2988	0.1555	0.0296	0.0490	0.2866	0.2500	0.0131	0.0712	0.1425
COM1 vs TSP2	0.1101	0.2141	0.0732	0.0022	0.0460	0.0169	0.9234	0.3188	0.0435	0.1958	0.0006	0.0001
COM2 vs TSP2	0.2026	0.6263	0.0463	0.0058	0.6042	0.3743	0.3616	0.5834	0.5783	0.5977	0.0440	0.0990
MIX1 vs TSP2	0.8319	0.9787	0.7743	0.0001	0.1232	0.0275	0.5241	0.6068	0.4920	0.2893	0.3320	0.3163
MIX2 vs TSP2	0.7531	0.9479	0.7788	0.0734	0.7032	0.5853	0.0894	0.1164	0.0012	0.9809	0.2284	0.0316
MIX3 vs TSP2	0.7717	0.0007	0.3744	0.0604	0.1869	0.1076	0.7528	0.6425	0.4890	0.8290	0.1247	0.2500
SBC1 vs TSP2	0.2904	0.9290	0.3251	0.6761	0.4517	0.3686	0.0466	0.0178	0.0010	0.5440	0.0354	0.0009
SBC2 vs TSP2	0.6758	0.3271	0.0238	0.0344	0.1543	0.0357	0.3237	0.7945	0.6762	0.3376	0.0804	0.3020

(1) = Each residue applied at 1, 2 and 3 the application rate compare to control (CON), triple superphosphate (TSP) at 1 and 2 times the recommended P2O5 rate.

Shaded values represent significance at Prob &lt; 5% level.

93a = Soil sampling before treatments, 23 May 1993.

93b = Soil sampling at harvest time, November 3, 1993.

94c = Soil sampling at harvest time, October 22, 1994, on half plots that have not received additional by-products.

94d = Soil sampling at harvest time, October 22, 1994, on half plots that have received additional by-products.

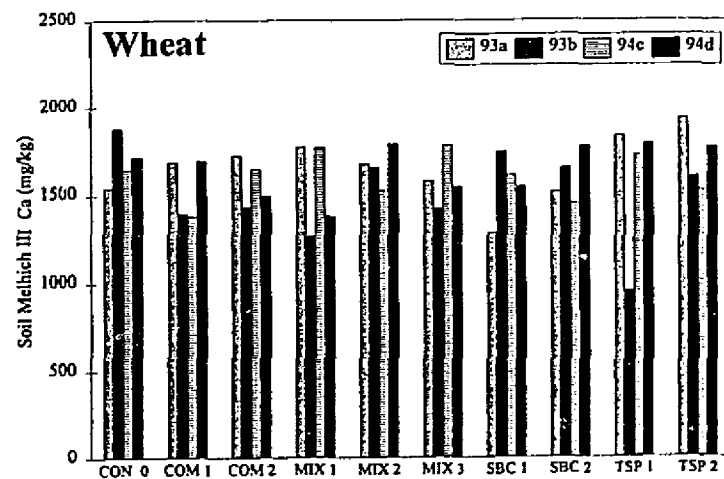
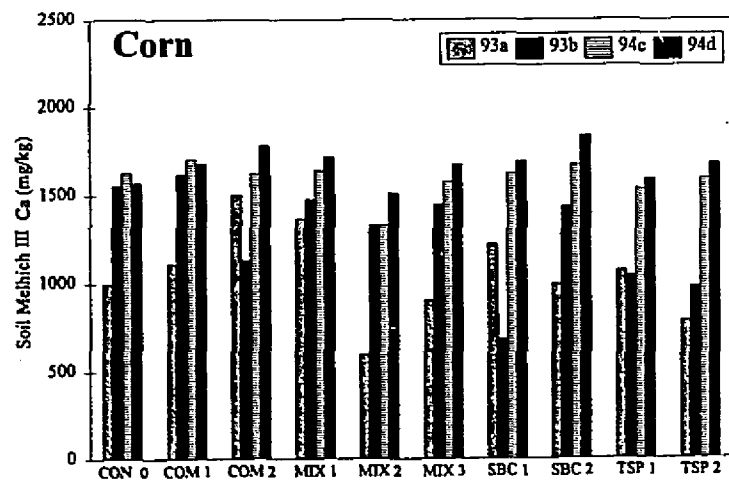
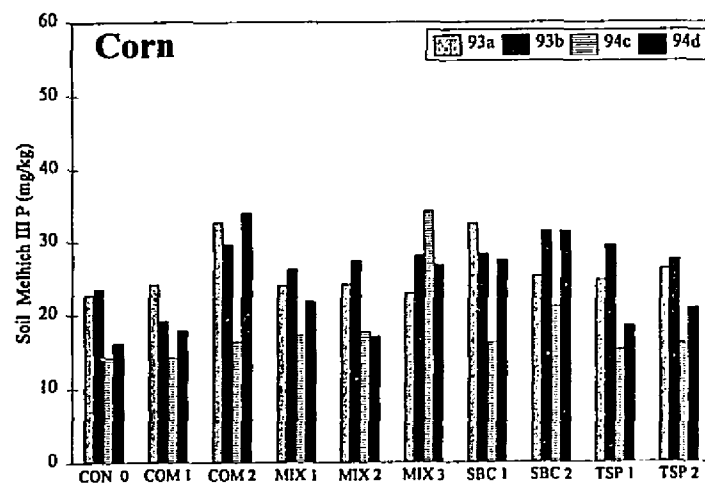
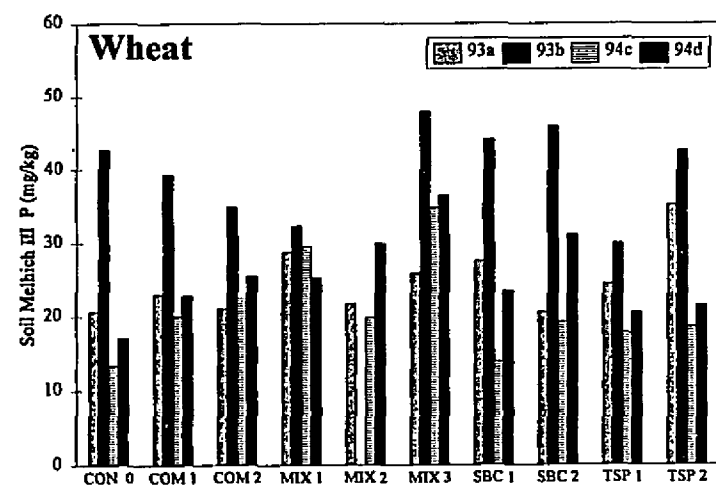


Figure 1. Level of soil extractable P and Ca (Melnich III) influenced by sugar refinery by-products and TSP in wheat and corn soils in 1993 and 1994.



Ca and Mg averages followed the same tendency in the wheat plots (Figure 2 and Table B-1 in appendix), while the corn parcels showed different results for all the treatments. The experiment does not demonstrate any links or similar trends between P and Ca availability (Figure 1), since Ca was not influenced by the rate effect in both soil types (Table 2 and 3).

### **Potassium**

As for P, but in a more pronounced fashion, average soil extractable K in all treatments diminished more significantly for wheat than corn in 1994 (Tables B-1 and B-2 in appendix and Tables 3 and 4).

### **Organic Matter**

Soil OM % was the only parameter, with the exception of soil P, to significantly differ with treatments. Changes arose especially from MIX and SBC treatments in both crops (Table 2 and 3). In soils of both wheat and corn plots, MIX and SBC, at all application rates cumulatively increased soil OM at the end of the second year application.

MIX2 significantly raised soil OM under both crops, where residual (93b vs 94c) and cumulative (93b vs 94d) effects of MIX2, in both crops, and effect of MIX3 in wheat only, were noted when compared to CON and TSP (Tables 7 and 8). Under wheat (Table 4), SBC2 raised the OM level in 93b, but lowered it in 94d when compared to TSP2. SBC2 differed from TSP1 and TSP2 by reducing the OM level in the corn plots (Table 6). Under corn (Table 6), contrast analysis demonstrated that OM content increased with the COM treatment by the end of the first cropping season, relative to control plot soils. Thus, residual and cumulative effects on OM levels were observed following COM addition. This is confirmed by the contrasts between COM and TSP2, where probability varied from 3 to 15 %.

Table 7. Probability of treatment effects on contrasts, comparing wheat grain over time of sampling for each sugar refinery by-products.

Residue contrast	N			P			P uptake		
	93e and 94e	93e and 94f	93e and 94f	93e and 94e	93e and 94f	93e and 94f	93e and 94e	93e and 94f	93e and 94f
COM1 vs CON	0.5470	0.8144	0.1178	0.6654	0.7730	0.2276	0.7063	0.8475	0.8125
COM2 vs CON	0.2378	0.8503	0.9124	0.6965	0.7913	0.4119	0.3491	0.2244	0.9487
MIX1 vs CON	0.7698	0.9844	0.5713	0.4124	0.7777	0.9211	0.3814	0.4424	0.3180
MIX2 vs CON	0.7679	0.5110	0.4229	0.7505	0.9295	0.4241	0.9415	0.6822	0.6716
MIX3 vs CON	0.3733	0.9412	0.9533	0.1345	0.1993	0.0763	0.2686	0.2477	0.7939
SBC1 vs CON	0.3335	0.0736	0.1164	0.4436	0.1283	0.0641	0.8188	0.9877	0.9840
SBC2 vs CON	0.8208	0.4250	0.1384	0.6671	0.1607	0.7422	0.5099	0.5399	0.1454
TSP1 vs CON	0.4641	0.2346	0.0857	0.7715	0.6378	0.1663	0.9310	0.9525	0.5336
TSP2 vs CON	0.6531	0.4520	0.1656	0.3923	0.6727	0.2334	0.8520	0.7481	0.9040
COM1 vs TSP1	0.9151	0.3718	0.9599	0.8971	0.8751	0.9270	0.7615	0.7923	0.3904
COM2 vs TSP1	0.7358	0.2023	0.1220	0.9518	0.4908	0.5621	0.2853	0.2271	0.4923
MIX1 vs TSP1	0.7718	0.2656	0.4306	0.3304	0.4801	0.3386	0.3151	0.4588	0.1045
MIX2 vs TSP1	0.6752	0.5836	0.3630	0.9789	0.6961	0.5628	0.7646	0.6240	0.8423
MIX3 vs TSP1	0.9459	0.1855	0.0597	0.1401	0.0245	0.2053	0.2862	0.2042	0.7176
SBC1 vs TSP1	0.8340	0.0032	0.0014	0.6552	0.2723	0.6915	0.8786	0.9548	0.5205
SBC2 vs TSP1	0.3376	0.6466	0.6396	0.4881	0.0593	0.2399	0.5495	0.4922	0.4016
COM1 vs TSP2	0.3235	0.6205	0.8463	0.7015	0.8980	0.9713	0.8423	0.8929	0.7208
COM2 vs TSP2	0.3300	0.1232	0.3843	0.6022	0.5255	0.6703	0.2412	0.1095	0.8533
MIX1 vs TSP2	0.5412	0.4747	0.5723	0.1616	0.5150	0.4074	0.2677	0.2559	0.2634
MIX2 vs TSP2	0.4766	0.8958	0.5458	0.6062	0.7352	0.6780	0.6863	0.9263	0.7615
MIX3 vs TSP2	0.2018	0.4003	0.1342	0.0413	0.0395	0.2945	0.3357	0.3917	0.8881
SBC1 vs TSP2	0.0332	0.1772	0.6162	0.9181	0.2971	0.6099	0.9579	0.7459	0.8881
SBC2 vs TSP2	0.8000	0.9702	0.8715	0.2040	0.0852	0.3360	0.6212	0.7600	0.1212

(1)= Each residue applied at 1, 2 and 3 the application rate compare to control (CON), triple superphosphate (TSP) at 1 and 2 times the recommended P2O5 rate.

Shaded values represent significance at Prob &lt; 5% level.

93e= tissues sampling on half plots that will not receive additional treatments application 1994.

93f= tissues sampling on half plots that will receive additional treatments application 1994.

94e= tissues sampling on half plots that have not received additional treatments application 1994.

94f= tissues sampling on half plots that have received additional treatments application 1994.

Table 8. Probability of treatment effects on contrasts, comparing corn grain over time of sampling for each sugar refinery by-products (1)

Residue contrast	N			P			P uptake		
	93e and 94e	93e and 94f	94e and 94f	93e and 94e	93e and 94f	94e and 94f	93e and 94e	93e and 94f	94e and 94f
COM1 vs CON	0.9011	0.0538	0.4880	0.7845	0.2616	0.7583	0.3206	0.7756	0.5454
COM2 vs CON	0.0340	0.7356	0.5137	0.1065	0.0673	0.8173	0.0416	0.0024	0.0865
MIX1 vs CON	0.7559	0.3285	0.5119	0.4378	0.3738	0.5333	0.2328	0.8725	0.1564
MIX2 vs CON	0.2392	0.2120	0.2569	0.3734	0.3141	0.6914	0.7476	0.7859	0.2462
MIX3 vs CON	0.7455	0.0151	0.1174	0.8473	0.2743	0.9883	0.6484	0.0281	0.1269
SBC1 vs CON	0.0900	0.5737	0.4266	0.0080	0.8503	0.0290	0.6920	0.0094	0.4354
SBC2 vs CON	0.5223	0.1831	0.4282	0.4124	0.2498	0.2883	0.1002	0.0537	0.6258
TSP1 vs CON	0.7915	0.9164	0.6650	0.8717	0.3426	0.2384	0.8890	0.8644	0.6818
TSP2 vs CON	0.9578	0.3213	0.4037	0.7791	0.8820	0.5986	0.8934	0.5332	0.7584
COM1 vs TSP1	0.7018	0.0666	0.2844	0.9305	0.0574	0.4217	0.2773	0.6578	0.8592
COM2 vs TSP1	0.0769	0.6605	0.2950	0.1669	0.3425	0.3547	0.0702	0.0014	0.2173
MIX1 vs TSP1	0.5894	0.3827	0.3020	0.3684	0.0831	0.0945	0.2995	0.7499	0.3091
MIX2 vs TSP1	0.1703	0.2534	0.1327	0.3165	0.0618	0.1266	0.8620	0.6655	0.4928
MIX3 vs TSP1	0.5747	0.0198	0.0564	0.9763	0.0533	0.2639	0.5704	0.0190	0.2933
SBC1 vs TSP1	0.1704	0.6447	0.7185	0.0023	0.5215	0.3083	0.8067	0.0059	0.7324
SBC2 vs TSP1	0.7152	0.2111	0.3165	0.5226	0.6266	0.9003	0.1435	0.0396	0.8885
COM1 vs TSP2	0.3050	0.8766	0.2901	0.9591	0.3239	0.8295	0.4581	0.7702	0.7943
COM2 vs TSP2	0.0545	0.1951	0.8202	0.2514	0.0566	0.7578	0.0538	0.0133	0.2108
MIX1 vs TSP2	0.8109	0.9883	0.8664	0.3279	0.4582	0.2838	0.2188	0.6762	0.2862
MIX2 vs TSP2	0.3140	0.8135	0.8413	0.2842	0.3921	0.3781	0.6750	0.7140	0.4542
MIX3 vs TSP2	0.8103	0.1421	0.5372	0.9201	0.3441	0.6167	0.7800	0.1147	0.2739
SBC1 vs TSP2	0.1224	0.6885	0.1359	0.0067	0.7395	0.1373	0.6337	0.0433	0.6793
SBC2 vs TSP2	0.5312	0.5743	0.9751	0.6426	0.2058	0.5618	0.1082	0.1459	0.8314

(1)= Each residue applied at 1, 2 and 3 the application rate compare to control (CON), triple superphosphate (TSP) at 1 and 2 times the recommended P2O5 rate.

Shaded values represent significance at Prob &lt; 5% level.

93e= tissues sampling on half plots that will not receive additional treatments application 1994.

93f= tissues sampling on half plots that will receive additional treatments application 1994.

94e= tissues sampling on half plots that have not received additional treatments application 1994.

94f= tissues sampling on half plots that have received additional treatments application 1994.

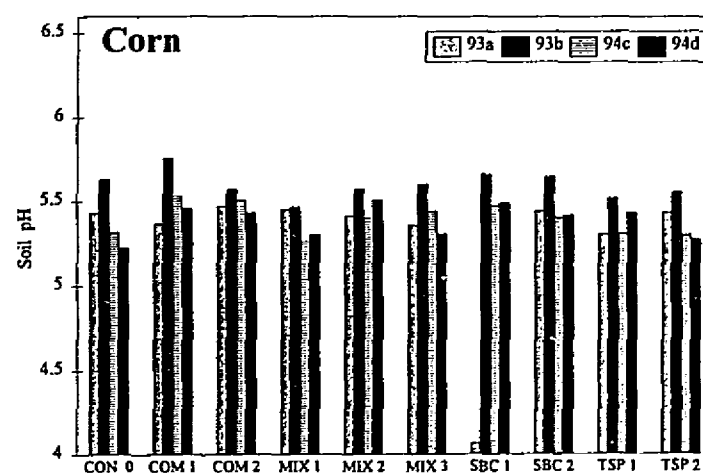
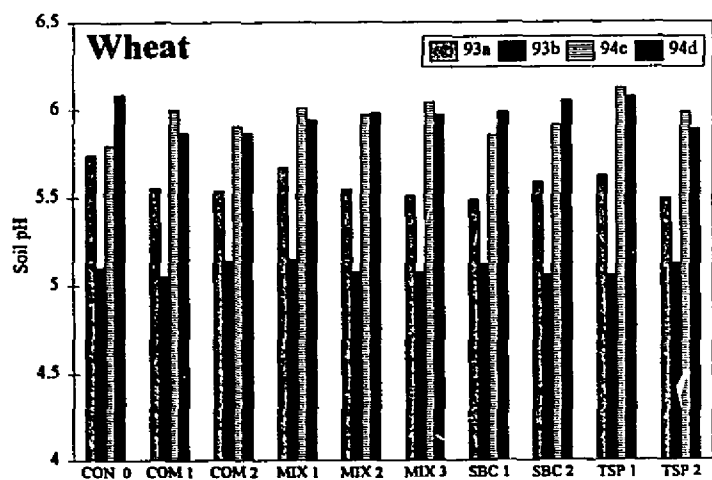
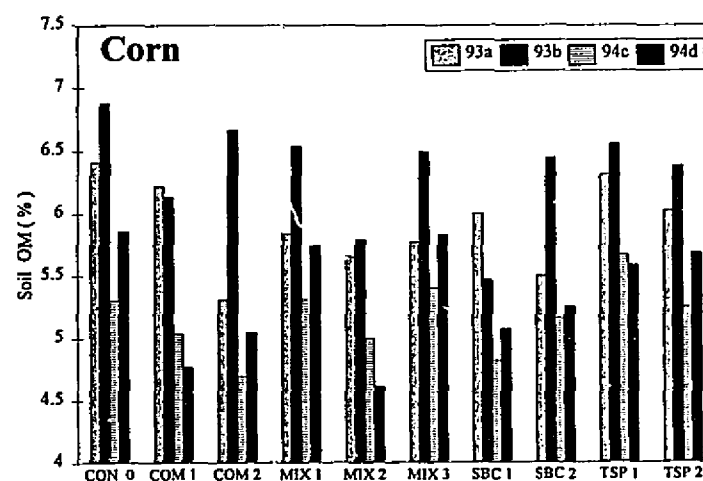
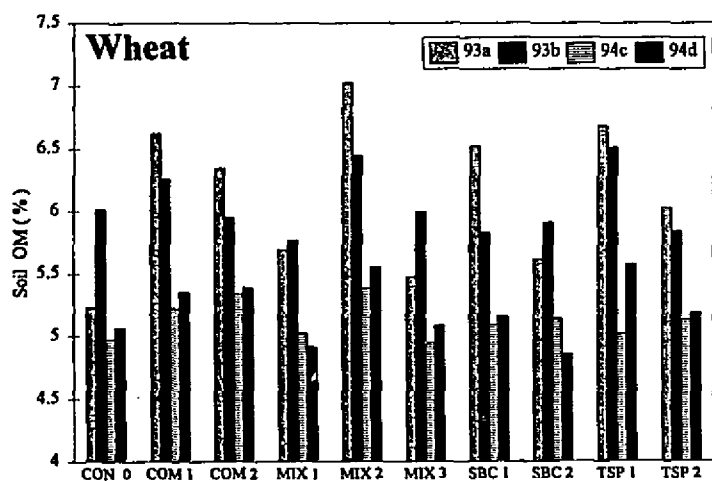


Figure 2. Level of soil organic matter and soil pH influenced by sugar refinery by-products and TSP in wheat and corn soils in 1993 and 1994.

## Soil pH

In corn plots (Table 6), significance of orthogonal contrasts comparing soil pH and soil OM for COM, SBC and MIX showed similar trends. The soil pH lay within 5 to 5.5 at the beginning of the experiment (Figure 2). Soil pH is a determinant factor of P availability. Tisdale et al. (1990) reported that P availability is optimal when soil pH lies between 5.5 and 6.5. At lower soil pH, as in the present experiment, P fixation is related to the establishment of links between phosphate anions ( $\text{PO}_4^{3-}$ ) and oxidized Fe and Al.

The 93b series had a much lower pH than series 94c and 94d in the wheat parcels which is attributable to the heavy rainfall of August 1993 (Brady, 1990) while the reverse is true in the corn crop (Figure 2). Contrasts revealed no significance differences between all residues treatments and CON or TSP.

Contrary to the results of the incubation study (chapter 2), the addition of SCU and FPM together did not significantly raise the soil pH, despite the low soil pH at the beginning of the experiment. In fact, MIX didn't differ from CON and TSP in both crops (Table 4 and 6).

In corn, COM1 raised soil pH over CON and TSP after 1 year of application and cumulative effects were observed at the end year two of application (Table 6). Under incubation conditions (Chapter 2), compost raised soil pH, depending on the application rate. In field, the same situation occurred after one and two cropping season only in corn plots (Table 6), although the effect of the application rate was much lower in field than in incubation study (chapter 2).

SBC1 lowered corn soil pH in the 93a vs 93b contrasts compared to CON and TSP1 (after one cropping season), but was significantly higher than TSP1 and TSP2 in both cumulative and residual effects in 1994.

## Nitrogen

NO<sub>3</sub>-N behaved similarly in both crops, with a slight decrease observed between 93b and both 1994 series (Figure 3). On the other hand, the levels were 4 times higher in the wheat parcels compared to the corn while there was very little difference observed between 93b, 94c and 94d. Time of sampling accounts for a large portion of this observed variability in these results.

In the previous experiment (Chapter 2), a one month incubation allowed the conversion of NH<sub>4</sub>-N to NO<sub>3</sub>-N in amended Ste-Rosalie soil, a soil similar to the one where this experiment was carried out, and was considered as net nitrification. The compost used for the laboratory study was the one used in the present experiment in 1993. The high C:N ratio (44:1) in the previous experiment obscured the availability of some macro-elements in the soil. In the present experiment, at the end of the first corn cropping season, only compost treatment caused inhibitory effects on nitrification (Figure 3). Since the same compost was used in 1993 and in the incubation test (chapter 2), that suggests that at sampling time, the presence of phenolic materials (Azhar et al., 1986) or the high C:N ratio (44:1) (Dick and McCoy, 1993) could explain, in part this situation.

In both 94c and 94d corn plots, there were no significant differences in NH<sub>4</sub>-N and NO<sub>3</sub>-N between amended and control plots (data not showed), suggesting that the process of nitrification is ongoing following the application of the treatment to the soil, as observed by N'Dayegamiye and Dubé (1986). In fact, in 1994, the residual effect of N on COM amended soil caused an increase in N concentration in both corn grain and stover (Table 8 and 9). The previous authors found that net immobilization of N happened the year after lignified residues were applied to cultivated soil. Here, however, even with a second COM application in corn plots, net mineralization occurred. Following COM addition to the soil, micro-organisms may have utilized the N from fertilizers to detriment of plant uptake at the very beginning season (Beauchemin et al., 1992) but this apparently did not affect overall uptake.

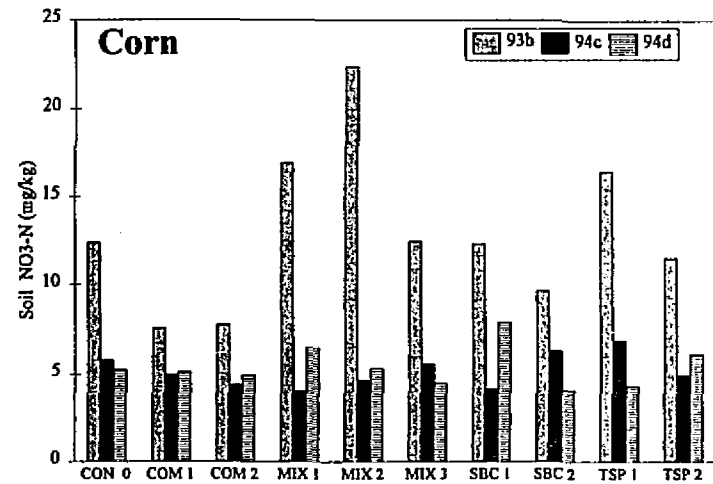
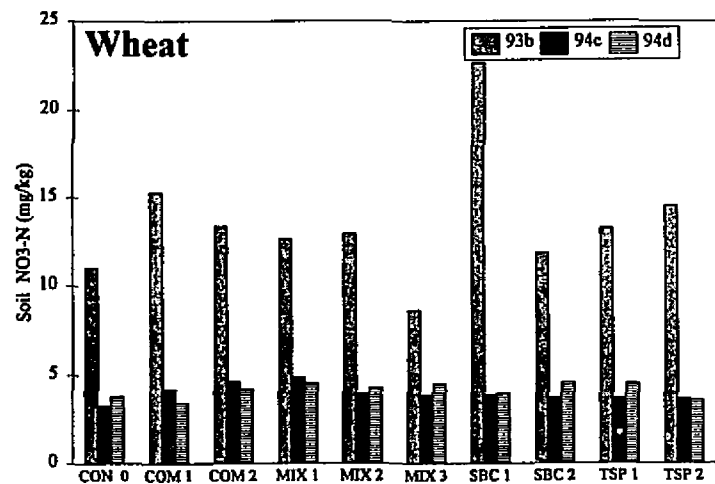
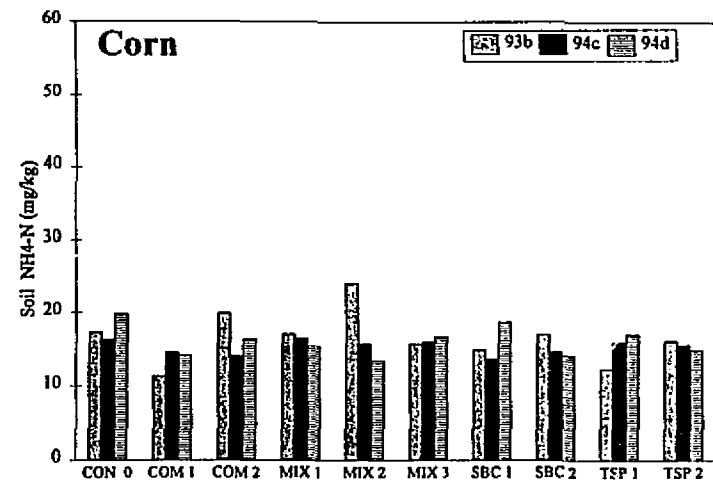
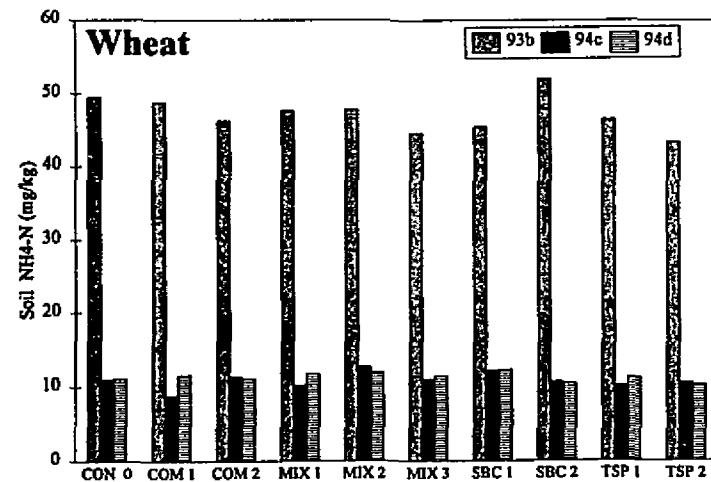


Figure 3. Level of soil extractable NH<sub>4</sub>-N and NO<sub>3</sub>-N influenced by sugar refinery by-products and TSP in wheat and corn soils in 1993 and 1994.

### **Wet Aggregate Stability**

Wet aggregate stability (WAS) varied substantially for the different treatments and crops (Figure 4). It should be noted that soils were not sampled for WAS analysis in 1993 and that results shown in Figure 4 originate from plots that were treated once (94c) and twice (94d) in 1994. No general tendency in the results observed enhanced WAS in 94d over 94c series. Also, the non-treated plots (CON) often showed higher WAS compared to the amended plots in both crops. These two observations together confirm the high variability of the studied soil.

Except for differences between replicates and treatment\*rate interaction, WAS did not exhibit differences between the chosen variation sources (Table 3).

WAS in treated soils did not differ from CON (Table 10). These results were not expected, especially in view of the high OM contents of some applied residues, COM and MIX in particular. Incubation test results (Chapter 2) confirmed that SCU and FPM (MIX in this experiment) as well as SBC when applied to the soil increased microbial activity and soil respiration, which should increase WAS (Perfect, 1990).

This suggests that soil colloidal dispersion already present in the soil prior to organic amendment was sufficiently high to not be influenced, structurally, by the residues. Great care was taken to avoid sampling soil for WAS purposes at locations where traffic on the plots occurred (soil preparation, residue handling, herbicide applications and crop harvesting) which could have resulted in compaction.

Many explanations could explain these results. As reported by Rasaih et al. (1992), clay, OM contents and pH of the soils explained most of the variability in the sensitivity of WAS. But in our case, the sensitivity of WAS generally was not influenced by application of residues rich in OM (COM and MIX). Except for temporal variations of soil pH, water content could explain the variability in WAS, as observed by Drury et al. (1991), where WAS negatively correlated with soil water content, independently of cropping management.



Table 9. Probability of treatment effects on contrasts, comparing corn stover over time of sampling for each sugar refinery by-products (1)

Residue contrast	N			P			P uptake		
	93e and 94e	93e and 94f	94e and 94f	93e and 94e	93e and 94f	94e and 94f	93e and 94e	93e and 94f	94e and 94f
COM1 vs CON	0.0173	0.6710	0.0180	0.5348	0.1757	0.1187	0.7530	0.4314	0.7927
COM2 vs CON	0.3107	0.9156	0.8242	0.3275	0.0664	0.1324	0.0599	0.9121	0.0970
MIX1 vs CON	0.5661	0.8901	0.9676	0.7959	0.1896	0.2616	0.5702	0.3887	0.4922
MIX2 vs CON	0.9399	0.7778	0.6681	0.2459	0.1896	0.6797	0.1588	0.4644	0.4529
MIX3 vs CON	0.4949	0.8391	0.6161	0.7166	0.8661	0.7277	0.0562	0.4709	0.0332
SBC1 vs CON	0.7635	0.6881	0.7764	0.6747	0.6474	0.2227	0.9021	0.6198	0.8735
SBC2 vs CON	0.9089	0.8231	0.8301	0.9126	0.4885	0.2551	0.0399	0.5294	0.0412
TSP1 vs CON	0.8936	0.2287	0.4660	0.7340	0.0035	0.0367	0.2994	0.4454	0.2949
TSP2 vs CON	0.9476	0.7019	0.9922	0.9915	0.3382	0.5922	0.5006	0.3145	0.4247
COM1 vs TSP1	0.0143	0.4445	0.0978	0.3471	0.1153	0.5887	0.4793	0.9915	0.4319
COM2 vs TSP1	0.3898	0.2835	0.3421	0.5316	0.2704	0.5505	0.4036	0.3808	0.5355
MIX1 vs TSP1	0.6883	0.1907	0.4416	0.9379	0.1001	0.3261	0.6451	0.5427	0.7291
MIX2 vs TSP1	0.8383	0.3679	0.2480	0.4218	0.1061	0.0918	0.7141	0.9753	0.7654
MIX3 vs TSP1	0.5918	0.1703	0.2197	0.9808	0.0026	0.0154	0.3882	0.1489	0.2715
SBC1 vs TSP1	0.8703	0.4320	0.3118	0.4589	0.0130	0.3762	0.3715	0.7939	0.2281
SBC2 vs TSP1	0.8086	0.1643	0.6066	0.8224	0.0272	0.3337	0.3137	0.8954	0.3131
COM1 vs TSP2	0.0171	0.9371	0.0176	0.5514	0.6961	0.3028	0.7156	0.8289	0.5950
COM2 vs TSP2	0.3545	0.7870	0.8318	0.3339	0.3855	0.3279	0.2341	0.2656	0.3823
MIX1 vs TSP2	0.6401	0.6113	0.9754	0.7917	0.7490	0.5555	0.9175	0.3975	0.9373
MIX2 vs TSP2	0.8905	0.9228	0.6753	0.4734	0.2530	0.7274	0.4698	0.7879	0.9655
MIX3 vs TSP2	0.5469	0.5676	0.6230	0.7153	0.2721	0.3788	0.2234	0.0934	0.1764
SBC1 vs TSP2	0.8184	0.9854	0.7839	0.6896	0.5867	0.4921	0.5907	0.6176	0.3410
SBC2 vs TSP2	0.8604	0.5552	0.8225	0.9064	0.7950	0.5452	0.1735	0.7118	0.2070

(1) = Each residue applied at 1, 2 and 3 the application rate compared to control (CON), triple superphosphate (TSP) at 1 and 2 times the recommended P2O5 rate.

Shaded values represent significance at Prob &lt; 5% level.

93e = tissues sampling on half plots that will not receive additional treatments application 1994.

93f = tissues sampling on half plots that will receive additional treatments application 1994.

94e = tissues sampling on half plots that have not received additional treatments application 1994.

94f = tissues sampling on half plots that have received additional treatments application 1994.

Table 10. Probability of treatment effects on contrasts, comparing wet aggregate stability in 1994, series c and d combined.

Residue contrast	CORN	WHEAT
	94c and 94d	94c and 94d
COM1 vs CON	0.0010	0.6592
COM2 vs CON	0.3995	0.8928
MIX1 vs CON	0.8384	0.0142
MIX2 vs CON	0.0055	0.4810
MIX3 vs CON	0.9092	0.3741
SBC1 vs CON	0.7025	0.8163
SBC2 vs CON	0.0506	0.0643
TSP1 vs CON	0.0828	0.5312
TSP2 vs CON	0.1954	0.8818
COM1 vs TSP1	0.1783	0.8526
COM2 vs TSP1	0.3656	0.4473
MIX1 vs TSP1	0.0571	0.0025
MIX2 vs TSP1	0.3213	0.4897
MIX3 vs TSP1	0.1186	0.7912
SBC1 vs TSP1	0.1783	0.6931
SBC2 vs TSP1	0.8283	0.8708
COM1 vs TSP2	0.0631	0.7698
COM2 vs TSP2	0.6209	0.7769
MIX1 vs TSP2	0.1851	0.8308
MIX2 vs TSP2	0.2525	0.4584
MIX3 vs TSP2	0.7107	0.6326
SBC1 vs TSP2	0.3566	0.9333
SBC2 vs TSP2	0.5622	0.7522

(1) = Each residue applied at 1, 2 and 3 the application rate compared to control (CON) and TSP at 1 and 2 times the recommended P205 rate.

Shaded values represent significance at Prob < 5% level.

**Table 11. Probability of treatment effects on corn grain N, P, K, Yield and P uptake (type II**

<b>SOURCES</b>	<b>d</b>	<b>N</b>	<b>P</b>	<b>K</b>	<b>Yield</b>	<b>P uptake</b>
<b>RES</b>	4	0.9201	0.0681	0.1281	0.2626	0.4218
<b>RATE</b>	2	0.4023	0.9193	0.9618	0.5547	0.6687
<b>REP</b>	3	0.4126	0.0005	0.1110	0.0001	0.1040
<b>TIME (1)</b>	2	0.0001	0.0001	0.0001	0.0001	0.0001
<b>RES*TIME</b>	7	0.2645	0.1414	0.6567	0.0042	0.0108
<b>RATE*TIME</b>	4	0.2084	0.2671	0.5424	0.0007	0.0291
<b>REP*TIME</b>	6	0.0136	0.0554	0.0002	0.0001	0.0014
<b>RES*RATE</b>	3	0.2199	0.1958	0.2561	0.4853	0.0836
<b>REP*RATE</b>	6	0.8479	0.8039	0.1870	0.3619	0.8996
<b>C.V.</b>		10.67	13.08	12.67	22.05	23.77
<b>R-2</b>		57.0%	68.7%	68.4%	91.2%	91.3%

Shaded values represent significance at Prob < 5% level.

RES= Residues; REP= Replicates; TIME= sampling time or series; RATE= rate of application for each residues.

df = Degrees of freedom

(1)=Time refers to series

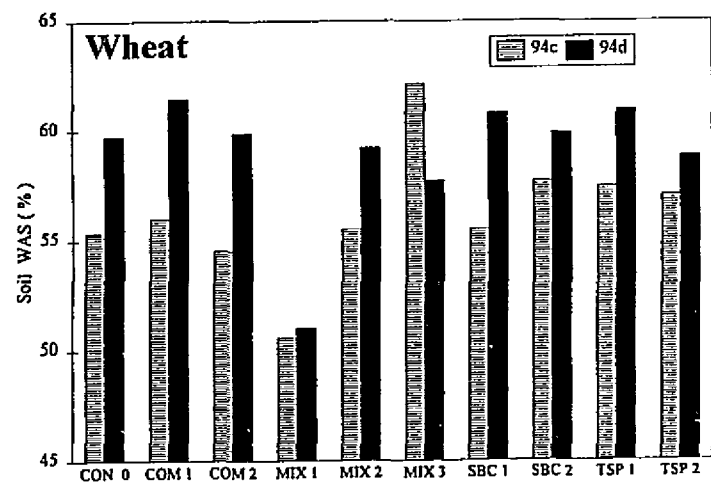
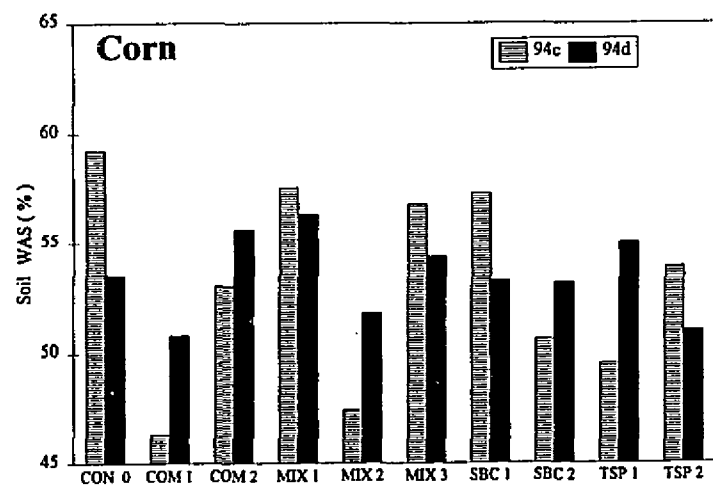


Figure 4. Wet aggregate stability (%) influenced by sugar refinery by-products and TSP in wheat and corn soils in 1994.

Perfect et al. (1990) observed temporal fluctuations in soil structural stability and realized that WAS decreased with increasing soil water content ( $r = 0.74$ ). Because soil sampling was done after heavy rainfall, it is conceivable that climatic condition prior and during soil sampling could explain this situation.

On the other hand, influences of organic polyanions (provided by MIX residue) do not prevent soil clay dispersion and soil aggregation. The differences of soil pH between plots could be part of the variability of WAS (Gu and Doner, 1993).

### **Crop tissue results**

Wheat grain P uptake was generally half of the corn grain results while the 1994 yields were 10 times inferior to 1993 results. The 1993 values of N and P in the wheat grain were doubled that of corn grain (Table B-3 and B-4 in Appendix) while the K concentration was similar in both crops. As proposed by Frageria (1991), corn, in stress conditions may have concentrated the N, P and K in the corn stover, resulting in poor grain quality.

As for soil, series (time) source of variation influenced the three tissue macro-nutrients. Few differences were observed between residues compared to controls for tissue chemical parameters with none of the treatments showing nutrient increases over controls values in wheat grain (Table 7), while few differences appeared in corn grain (Table 8) and stover (Table 9). TSP treatments did not differ from CON for all the nutrients in crop tissues.

### **Nitrogen**

Results of wheat and corn N content are presented in Appendix, Tables B-3 and B-4. From the contrast table (Table 8), COM2 significantly increased corn grain N content over control in 93e vs 94e, suggesting that residual N content in corn grain on COM treated plots, became available for the plant after the first year of experiment. Similar

results were observed (7,7 and 5,5 % probability level) when comparing COM1 with TSP1 and TSP2 on the two other sets of contrasts. In corn stover (Table 9), the same trend was observed, but concentrations were higher in COM1 over COM2. The cumulative effect of MIX3 on corn grain resulted in a marked decline the N concentration compared to CON and TSP1 (93e vs 94f). SBC-N concentration was minimal and, in fact, no increase on N in the crop tissues was observed in contrasts comparing SBC and CON or TSP.

### **Phosphorus and Potassium**

Phosphorus concentration in wheat grain did not show differences between variation sources except the residue\*rate interaction (Table 4) which arose specifically from the MIX treatment. MIX at 3 times the recommended rate provided, as suggested by the contrasts to determine the cumulative effect (93e vs 94f), a superior P contribution in the wheat tissues than TSP, but not compared to CON.

Phosphorus and potassium concentration in wheat grain were highly variable even in untreated controls where, as it was expected (Shaw, 1988), the concentrations in the corn grains were N, K and P in that order. Between 1993 and 1994, P concentrations in the corn stover increased. In fact, it doubled and even tripled according to the treatment.

Residue source of variation showed significant differences in P concentration in corn grain (Table 8). The P and K concentrations in the corn grain decreased significantly from 1993 to 1994 (Table B-4). Contrary to wheat, however, P concentration in corn grain in plots amended by MIX didn't differ significantly from CON and TSP (Tables 9 and 10). The P contribution of SBC1 in the corn grain was higher when there was only one application (1993). The residual effect differed between CON and TSP treatments, while the cumulative effect of SBC1 was significantly higher compared to CON (Table 8). From the 93e vs 94f series comparison, which serves to determine cumulative effect, P concentration in corn stover that received MIX3 decreased relative to TSP1 (Table 8).

### **P uptake**

P uptake is the result of P tissue concentration and crop yield. Poor corn and wheat grain yields explain the low P uptake values in 1994 (Figures 5 and 6). Average yield is expected to be around 3700 kg ha<sup>-1</sup> for wheat and 7600 kg ha<sup>-1</sup> for corn grain (based on 85 % dry basis) for Montréal southshore area (CRÉAQ, 1994), whereas yield in this study was in the range of 150 and 1200 kg ha<sup>-1</sup> for wheat and corn respectively in 1994.

Corn P uptake was generally half the value obtained by Liang et al. (1993) (Figure 6). This is a consequence of late seeding and the use of a high corn heat unit at the moment of planting. In 1994, the P concentration was as much as one eighth of Liang's results, the result this time of the weed invasion. Thus, the 1993 values are more representative and could be used in the interpretation.

Replicate source of variation did not show yield differences in corn stover, as opposed to wheat and corn grain, but series (time) influenced the ANOVA model for wheat and corn as well. Residue source of variation showed significant differences of P uptake in wheat grain (Table 4) and where the MIX effect accounted for the largest proportion of the residue effect relative other residues.

Series (time) is the main source of variation of every descriptor in both crops. P uptake was mostly influenced by replicate and series (time). Nevertheless, the high variability of wheat and corn yields largely contributed to this result. This fact could explain that the P uptake was influenced by the model, especially corn stover ( $r^2 = 0.93$ ) (Table 12).

Figures 5 and 6 present wheat grain, corn grain and stover P uptake and illustrate differences among yields between 1993 and 1994. Because of the low yield problems, contrasts showed in Tables 9 to 11 it should, however, be interpreted with caution.

The low and unpredictable yields of 1994 would suggest that these results are not appropriate for general use. However, macro-element (N and P) concentrations in tissues seem reasonable and can be discussed because the results obtained in the CON and TSP

**Table 12. Probability of treatment effects on corn stover N, P, K, Yield and P uptake (type**

<b>SOURCES</b>	<b>df</b>	<b>N</b>	<b>P</b>	<b>K</b>	<b>Yield</b>	<b>P uptake</b>
<b>RES</b>	4	0.7134	0.7340	0.3961	0.8402	0.9891
<b>RATE</b>	2	0.3757	0.3367	0.6991	0.9830	0.1703
<b>REP</b>	3	0.8525	0.0001	0.1403	0.0001	0.0178
<b>TIME</b>	2	0.0001	0.0001	0.0001	0.3545	0.0001
<b>RES*TIME</b>	7	0.6983	0.5158	0.9379	0.3849	0.9957
<b>RATE*TIME</b>	4	0.9692	0.4133	0.1539	0.2074	0.3049
<b>REP*TIME</b>	6	0.1800	0.0001	0.2950	0.1889	0.0001
<b>RES*RATE</b>	3	0.2447	0.9019	0.0347	0.8690	0.5292
<b>REP*RATE</b>	6	0.3528	0.2742	0.1581	0.9167	0.6966
<b>C.V.</b>		29.07	31.54	18.54	19.38	28.79
<b>R-2</b>		50.5%	81.2%	47.8%	59.6%	72.7%

Shaded values represent significance at Prob < 5% level.

RES=Residues; REP= Replicates; TIME= sampling time or series; RATE= rate of application for each residues.

df = Degrees of freedom

(1)=Time refers to series



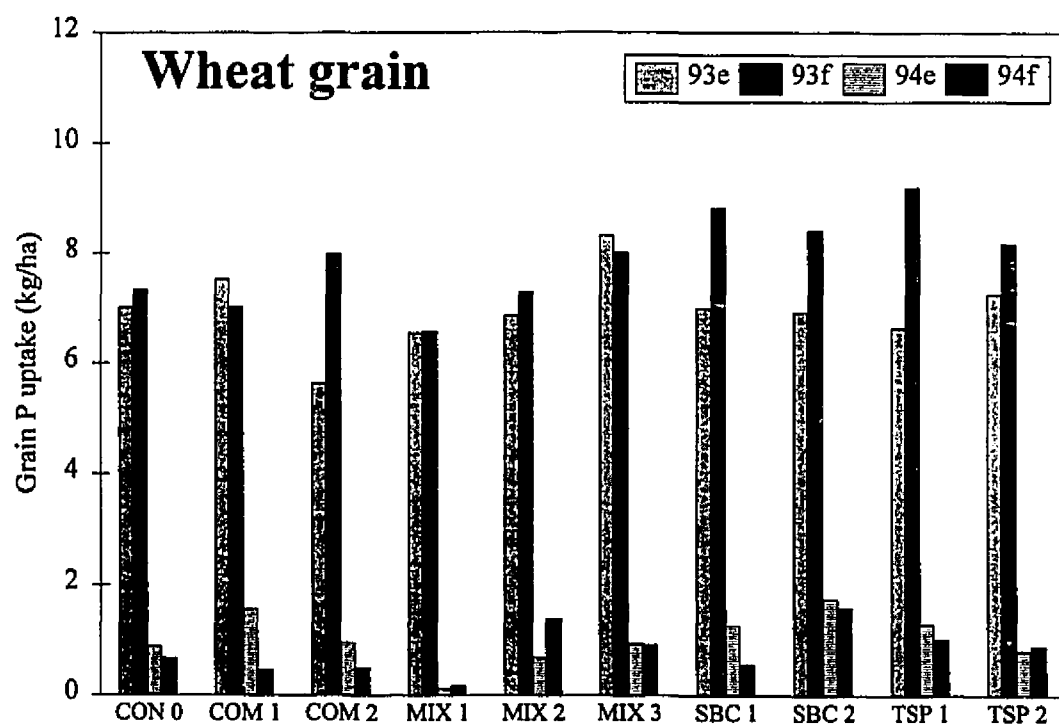


Figure 5. Wheat grain P uptake in 1993 and 1994.

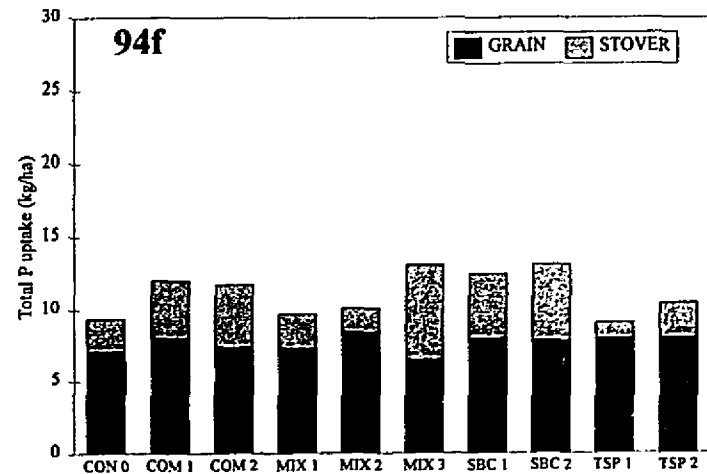
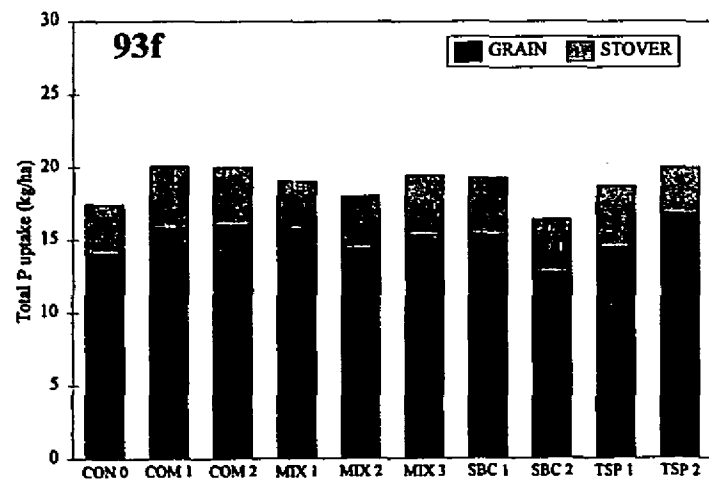
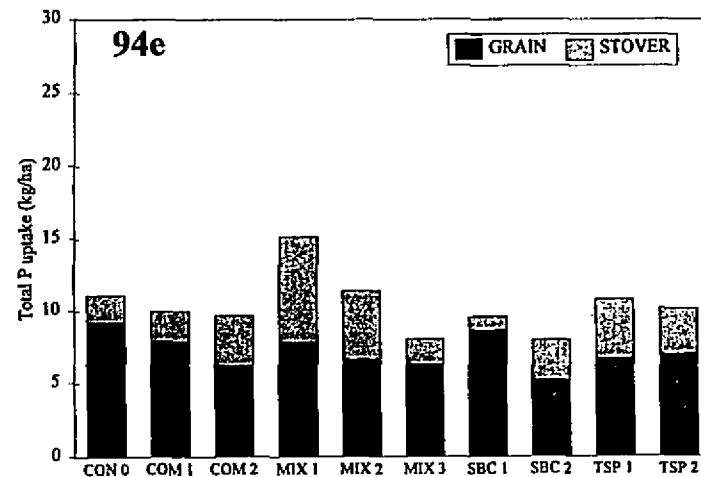
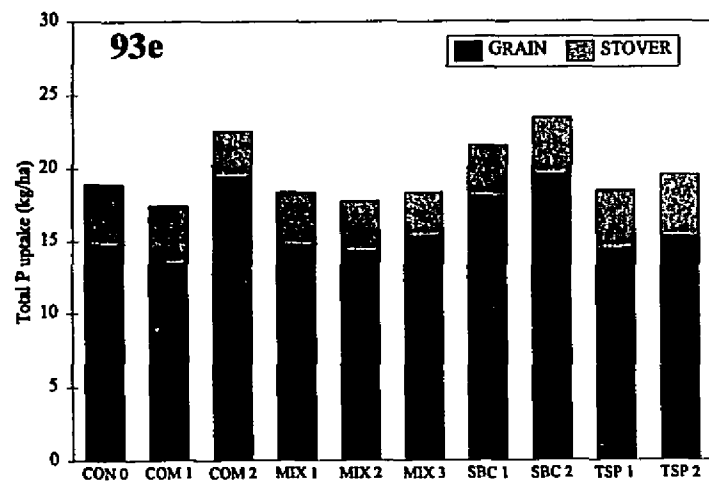


Figure 6. Corn grain and stover P uptake in 1993 and 1994.

treated plots correspond to results obtained in other experiments (Shaw, 1988, Liang et al., 1993).

From Tables 9 to 11, two way classification contrasts between 93c and 94c with two treatments at a time served to determine residual effect after 2 cropping years by a single application of residues, where 94c and 94f contrast allowed the evaluation of a specific treatment compared to controls.

Superior crop yields from amended SBC1 (Figure 5) plots in 1993 contributed to enhance the difference for the P uptake in corn grains between 93e and 94f (residual effect) compared to reference plots (CON, TSP1 and TSP2) (Table 8). This is why P uptake results obtained on the 93e vs 94f contrasts decreased significantly.

No cumulative and residual P uptake of all residues were observed in wheat grain and corn stover (Table 7 and 9). From corn grain results, COM2 amended plots presented both cumulative and residual effects, where only residual effects of COM1 application were noted.

## CONCLUSION

Results obtained during this experiment did not allow us to clearly understand the soil enriching mechanism of each studied residue. Large variations in crop growth related to nutrients and soil parameters before the start and during the experiment resulted from external events with a sphere of influence outside the parameters used in the statistical analysis.

In fact, the prevailing experiment conditions (heavy clay soil, lack of drainage, low soil pH, weed invasion (1994) and late seeding resulting in the immature corn crop of 1993) limited the differences among treatments as well as control parcels. Even TSP applied to soil at or twice the recommended rate did not significantly increase the levels of P in the soil, nor did it increase the P concentration of both crops. The fluctuating and

generally very low yields of 1994 in both crops disrupted the P uptake value and so limited their use as for establishing recommendations for the utilisation the Lantic by-products.

None of the treatments showed significant differences in WAS compared to control parcels. Results showed that, generally, application rates did not influence the model. For corn, treatments clearly modified most parameters without being able to determine the relative importance of the application rates with the exception of phosphorus. None of the treatments affected the Ca levels of the soil. Each by-product and compost raised the soil OM level, either as a residual and/or cumulative manner in both crop studied. However, none of the treatments differentiated itself from TSP or CON in wheat soil plots.

Specifically, contrasts analysis highlighted tendencies related to the use of by-products. COM raised the OM levels in both crops compared to CON and TSP, but a decline in  $\text{NO}_3\text{-N}$  was observed, probably in relation to the break down, by micro-organisms, of the lignified matter contained in COM (Beauchemin et al., 1992). Furthermore, the use of COM significantly increased N concentrations in corn grains and corn stover as a residual effect. Cumulative and residual effect of P uptake increased following COM application but such results should be validated under different circumstances to insure consistency.

As for COM, MIX raised the soil OM where cumulative (in corn) and residual (in both crops) effect were observed when MIX was applied at twice the recommended rate. MIX2 (wheat) and MIX3 (corn) significantly raised the soil P levels. After two years cropping, extractable P was higher from MIX than TSP treatments indicating the residue from P is less subject to fixation than soluble P fertilizer. These results suggest a review of the recommendations for P application rates under the actual conditions is warranted.

P availability from MIX3 appeared to take more than 1 year in corn plots but became readily available in wheat soil. Non degraded matter, even after two growing seasons was

observed in MIX treated plots. Nevertheless, MIX3 significantly reduced N in the corn grain. The contribution from MIX3 significantly reduced P in the corn stover but raised it in the wheat grain at the end of the second year's application.

SBC raised soil P in corn but at the recommended rate, where pH decline was observed after the second year of experiment. The important yield differences between 1993 and 1994 raised P uptake in corn stover amended SBC1 plots to the detriment of its uptake by the corn grain.

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## **CHAPTER 4. GENERAL CONCLUSION AND RECOMMENDATIONS**

Based on the results obtained from the laboratory assessment (chapter 2), field trials (chapter 3), and the observations pertaining to the manipulations of the different residues used in the course of this project, certain conclusions may be drawn for further use of each of the Lantic Sugar by-products.

Under incubation conditions, spent bone char (SBC) and clarification mud (SCU) significantly increased the Melhich III extractable P and Ca. The fixation rate of P in SBC observed in sandy soil was higher than in clay soil, whereas the type of soil does not appear to change the availability of P contained in the SCU. The Melhich III extraction performed on the SCU proved to be an excellent tool for predicting its P availability.

Extractable P was increased by 8 and 10 times in clay and 6 and 5 times in sandy-loam soil by the addition of 5 g of SCU and SBC respectively in 100 g of soil. However, these constitute very high application rates, equivalent to 576 and 342 kg  $P_2O_5$  ha<sup>-1</sup> with a fixation level ranging between 92.5 and 95.1 % of the total P initially contained in the SCU and SBC respectively. The application rates used greatly exceeded the recommended rates that were established at 60 (wheat) and 80 (corn) kg  $P_2O_5$  ha<sup>-1</sup> for the soil conditions at the field trial site (Association des Fabricants d'Engrais du Québec, 1990).

FPM distinguished itself from the other residues by its influence on the enzymatic activity (phosphatase) and its contribution to the enhancement of soil respiration proved to be in equivalent to that of SCU.

During the incubation tests, high C:N ratio of the compost used did not stimulate the enzymatic activity (phosphatase) of the soil in a striking way. The conversion from  $NH_4$ -N to  $NO_3$ -N, in both types of soil being tested supposes that a portion of the nitrogen in the soil was mineralized.

However, one could anticipate that a C:N ratio lower than 20:1 will improve COM degradation and provide an appreciable N uptake for assimilation by crops. COM raised the concentration of extractable phosphate in both incubated soils, but reached a plateau when applied at a rate between 350 and 700 kg  $P_2O_5$  equivalent per hectare.

The difficult conditions under which the field trials were performed, especially the weed problems in 1994, have limited the interpretation of comparisons between residual and cumulative effects of the use of each by-product and compost. The wheat and corn production having been considerably reduced in 1994, the P uptake results must therefore be used with the caution. Thus, the P uptake obtained during the course of this experience cannot, due to the low field trial performances, be used as a measurement for establishing recommendations with respect to the application rates of residues.

Contrast analysis has on the other hand demonstrated significant differences between sampling periods (years), per treatment pair, i.e. each treatment versus plots unamended and amended with TSP. The extensive variability in the contents of the different soil nutrients reduced the differences between treatments. The variance analysis showed that the application rate had little effect of soil and tissue properties, except in the case of phosphorus and the organic matter in corn plots.

This is why it is possible to state that the TSP applied to the soil at once or twice the recommended rates for  $P_2O_5$  never significantly surpassed the soil extractable P of the control plots under which this field experiment was conducted. No treatment whatsoever, despite its strong organic matter (OM) content, ever increased the wet aggregate stability. On the other hand, the OM content was significantly enhanced by MIX and by SBC in corn soil.

In the case of the corn crops, MIX applied at 160 or 240 kg  $P_2O_5$  ha<sup>-1</sup> resulted in the highest concentration of P over control and/or TSP and this occurred in a cumulative and/or residual manner according to application rates. In the case of wheat, MIX applied at 120 kg  $P_2O_5$  ha<sup>-1</sup> proved superior to CON and TSP. Extractable soil P was higher in

MIX than TSP treatment indicating that P from the mixture of clarification scum and filter-press mud is less subject to fixation than soluble P fertilizer under heavy clay and low soil pH. Once again, we must bear in mind that at the end of the 2<sup>nd</sup> year of experimentation, it was possible to detect on the soil, the scum-mud mixture that was applied to the soil during the previous year, a situation that did not occur in the laboratory. Extractable P could therefore be superior to the results obtained during the field trials.

SBC has to a lesser extent than in the case of MIX, resulted in an extractable P content which is significantly higher than the plots treated with TSP in corn plots but not in wheat. SBC at twice the prescribed application rate significantly enhanced the percentage of organic matter more than TSP. During its application in the field, despite all the precautions that were taken, small quantities of dust were dispersed outside of the plots. This problem should be dealt with during the large scale use of this by-product.

The compost produced for the laboratory tests and used in the field during the course of the first year of experimentation generated an enhanced soil pH as early as the first year of experimentation, for both types of soil. The soil losses of NO<sub>3</sub>-N related to COM application after the first year of experimentation may be attributable to the high C:N ratio or to the compost's phenolic substances. A compost with a C:N ratio of 10 to 15:1 would be expected to provide for an reasonable N uptake for the plants. Moreover, during the second year of experimentation, a stronger N concentration in corn grain and corn stover was observed in the plots treated with COM versus the control plots.

It would be useful to review certain of the producer's farming practices (enhancement of soil pH and improvement of adequate drainage in particular), in order that a portion of the fixed phosphorus may be converted into available P.

Due to the various problems encountered during the field trials, specifically because of weed invasion, the cumulative and residual availability of P uptake as well as N, P, K crop concentration remain inconclusive. On the other hand, the results did demonstrate

that the use of sugar refinery by-products in laboratory as well as field situations, improves certain soil characteristics, particularly with respect to extractable P and OM. A third year of study would be advisable due to the poor results of the second cropping season in both crops.

## **APPENDIX A**



**Table A-1. Extractable P, Ca and organic C provided by sugar refining residues for the incubation of soil analysis (1)**

Residue	Available P (1) (mg/kg)	Available P2O5 (mg/kg)	Available Ca (1) (mg/kg)	Organic C (%)	Equivalent P2O5 (2) (kg/ha)		Equivalent Ca (3) (kg/ha)	
rate					2.5%	5.0%	2.5%	5.0%
COM	4158	9522	20710	21	348	696	757	1513
FPM	482	1104	3050	29	31	62	85	171
SCU	6561	15025	29600	38	288	576	568	1136
SBC	1404	3215	14222	3.0	171	343	758	1517

(1) Extractable P and Ca are expressed as Mellich III extraction procedures.

(2) Equivalent of P2O5 at rates of 2.5 and 5g/100g of soil (on dry matter basis).

(3) Equivalent of Ca at rates of 2.5 and 5g/100g of soil (on dry matter basis).

## **APPENDIX B**

**Table B-1. Quantity of sugar refinery by-products applied as P source.**

RESIDUE	Rate	Equivalent of quantity applied ( kg/ha) (1)	
		Wheat (2)	Corn (3)
COM	1	7211	9615
	2	14422	19231
MIX	1	4159	5544
	2	8318	11087
	3	12477	16631
SBC	1	706	941
	2	1412	1882
TSP	1	130	174
	2	261	348

(1) On a wet basis

(2) At 1 fold recommended rate = 60 kg P/ha.

(3) At 1 fold recommended rate = 80 kg P/ha.

Table B-2. Chemical and physical soil analysis of wheat plots

Sampling time	Residue	Rate	P	K	Ca	Mg	NH <sub>4</sub> -N	NO <sub>3</sub> -N	OM	pH	WAS
			(mg/ kg)				(%)				(%)
93a	CON	0	21	247	1540	806	--	--	5.2	5.7	--
	COM	1	23	240	1690	875	--	--	6.6	5.6	--
	COM	2	21	250	1726	887	--	--	6.3	5.5	--
	MIX	1	29	247	1775	961	--	--	5.7	5.7	--
	MIX	2	22	244	1679	890	--	--	7.0	5.5	--
	MIX	3	26	248	1577	845	--	--	5.5	5.5	--
	SBC	1	28	248	1283	676	--	--	6.5	5.5	--
	SBC	2	21	213	1516	798	--	--	5.6	5.6	--
	TSP	1	24	255	1835	941	--	--	6.7	5.6	--
	TSP	2	35	248	1929	976	--	--	6.0	5.5	--
93b	CON	0	43	257	1877	958	49.5	11.0	6.0	5.1	--
	COM	1	39	253	1393	642	48.8	15.3	6.3	5.1	--
	COM	2	35	266	1432	738	46.2	13.3	5.9	5.1	--
	MIX	1	32	257	1267	808	47.7	12.6	5.8	5.1	--
	MIX	2	0	266	1658	736	47.8	13.0	6.4	5.1	--
	MIX	3	48	258	1418	850	44.6	8.7	6.0	5.1	--
	SBC	1	44	258	1745	825	45.4	22.7	5.8	5.1	--
	SBC	2	46	256	1651	838	52.1	11.9	5.9	5.1	--
	TSP	1	30	260	950	552	46.4	13.2	6.5	5.1	--
	TSP	2	43	262	1600	731	43.2	14.5	5.8	5.1	--
94c	CON	0	13	175	1645	694	11.0	3.3	5.0	5.8	55
	COM	1	20	138	1378	586	8.9	4.2	5.2	6.0	56
	COM	2	23	158	1646	593	11.3	4.6	5.3	5.9	55
	MIX	1	29	159	1772	685	10.2	4.9	5.0	6.0	51
	MIX	2	20	156	1525	597	12.7	3.9	5.4	6.0	56
	MIX	3	35	157	1780	631	10.9	3.8	4.9	6.0	62
	SBC	1	14	145	1612	656	12.1	3.9	5.1	5.9	56
	SBC	2	19	148	1449	600	10.6	3.7	5.1	5.9	58
	TSP	1	18	150	1726	651	10.1	3.7	5.0	6.1	57
	TSP	2	19	164	1525	712	10.5	3.7	5.1	6.0	57
94d	CON	0	17	148	1716	615	11.0	3.8	5.1	6.1	60
	COM	1	23	163	1695	623	11.6	3.4	5.4	5.9	62
	COM	2	26	146	1493	600	11.0	4.2	5.4	5.9	60
	MIX	1	25	141	1377	585	11.7	4.6	4.9	5.9	51
	MIX	2	30	170	1790	660	11.9	4.3	5.6	6.0	59
	MIX	3	36	148	1543	604	11.4	4.5	5.1	6.0	58
	SBC	1	24	140	1543	594	12.2	4.0	5.2	6.0	61
	SBC	2	31	154	1776	658	10.4	4.6	4.9	6.0	60
	TSP	1	21	167	1788	635	11.2	4.5	5.6	6.1	61
	TSP	2	22	160	1770	715	10.2	3.6	5.2	5.9	59

93a= Soil sampling before treatments, 23 may 1993.

93b= Soil sampling at harvest time, september first 1993.

94c= Soil sampling at harvest time, august 15th 1994, on half plots that have not received additional by-products.

94d= Soil sampling at harvest time, august 15th 1994, on half plots that have received additional by-products.

Table B-3. Chemical and physical soil analysis of corn plots

Sampling time	Residues	Rate	P	K	Ca	Mg	NH <sub>4</sub> -N	NO <sub>3</sub> -N	OM	pH	WAS
			(mg/ kg)						(%)		(%)
93a	CON	0	23	250	998	841	--	--	6.41	5.4	--
	COM	1	24	248	1110	758	--	--	6.22	5.4	--
	COM	2	33	276	1501	801	--	--	5.31	5.5	--
	MIX	1	24	255	1365	959	--	--	5.84	5.5	--
	MIX	2	24	266	598	908	--	--	5.65	5.4	--
	MIX	3	23	251	904	917	--	--	5.76	5.4	--
	SBC	1	32	255	1216	947	--	--	6.00	4.1	--
	SBC	2	25	255	994	857	--	--	5.50	5.4	--
	TSP	1	25	252	1068	566	--	--	6.31	5.3	--
	TSP	2	26	252	789	770	--	--	6.02	5.4	--
93b	CON	0	23	256	1555	800	17.4	12.4	6.9	5.6	--
	COM	1	19	265	1616	866	11.4	7.6	6.1	5.8	--
	COM	2	30	273	1127	551	20.0	7.8	6.7	5.6	--
	MIX	1	26	267	1474	537	17.1	16.9	6.5	5.5	--
	MIX	2	27	262	1329	637	24.1	22.4	5.8	5.6	--
	MIX	3	28	262	1447	713	15.7	12.5	6.5	5.6	--
	SBC	1	28	258	681	368	15.0	12.3	5.5	5.7	--
	SBC	2	31	293	1432	700	17.1	9.7	6.4	5.6	--
	TSP	1	29	285	1036	566	12.4	16.4	6.6	5.5	--
	TSP	2	28	264	979	529	16.2	11.5	6.4	5.6	--
94c	CON	0	14	188	1630	797	16.3	5.8	5.3	5.3	59
	COM	1	14	194	1706	796	14.6	5.0	5.0	5.5	46
	COM	2	16	194	1621	795	14.1	4.4	4.7	5.5	53
	MIX	1	17	189	1640	736	16.5	4.0	5.3	5.3	58
	MIX	2	18	171	1329	644	15.7	4.7	5.0	5.4	47
	MIX	3	34	181	1575	754	16.0	5.6	5.4	5.4	57
	SBC	1	16	191	1617	806	13.7	4.1	4.8	5.5	57
	SBC	2	21	203	1671	780	14.7	6.3	5.2	5.4	51
	TSP	1	15	217	1531	723	16.0	6.9	5.7	5.3	50
	TSP	2	16	203	1587	748	15.6	4.9	5.2	5.3	54
94d	CON	0	16	205	1568	759	19.9	5.2	5.9	5.2	54
	COM	1	18	191	1679	794	14.2	5.1	4.8	5.5	51
	COM	2	34	202	1782	794	16.4	4.9	5.0	5.4	56
	MIX	1	22	205	1717	763	15.5	6.4	5.7	5.3	56
	MIX	2	17	184	1510	737	13.5	5.3	4.6	5.5	52
	MIX	3	27	201	1674	748	16.7	4.5	5.8	5.3	54
	SBC	1	27	192	1692	798	18.7	7.9	5.1	5.5	53
	SBC	2	31	198	1834	816	14.1	4.1	5.2	5.4	53
	TSP	1	18	193	1587	781	17.1	4.3	5.6	5.4	55
	TSP	2	21	203	1676	770	15.0	6.1	5.7	5.3	51

93a= Soil sampling before treatments, 23 may 1993.

93b= Soil sampling at harvest time, november 3, 1993.

94c= Soil sampling at harvest time, october 22, 1994, on half plots that have not received additional by-products.

94d= Soil sampling at harvest time, october 22, 1994, on half plots that have received additional by-products.

Table B-4. Wheat N, P and K content, Yield and P uptake (1)

Sampling time	Residue	Rate	WHEAT GRAIN				
			N	P	K	Yield	P uptake
			(mg/kg)			(kg/ha)	(kg/ha)
93e	CON	0	27246	4540	4908	1547	7.0
	COM	1	27828	4613	4477	1633	7.5
	COM	2	27114	4645	4652	1206	5.6
	MIX	1	26900	4851	4563	1338	6.5
	MIX	2	26407	4549	4447	1517	6.9
	MIX	3	26640	5356	4723	1564	8.3
	SBC	1	27030	4356	4328	1617	7.0
	SBC	2	26354	5030	4967	1373	6.9
	TSP	1	27212	4647	4814	1423	6.6
	TSP	2	26261	4423	4426	1640	7.3
93f	CON	0	25803	4515	4354	1607	7.3
	COM	1	25754	4275	4450	1639	7.0
	COM	2	26675	4979	4970	1598	8.0
	MIX	1	28707	5135	5224	1295	6.6
	MIX	2	24772	4209	4604	1722	7.3
	MIX	3	26043	5064	4848	1578	8.0
	SBC	1	26803	5525	5318	1618	8.8
	SBC	2	27219	4896	4601	1723	8.4
	TSP	1	26497	5171	3762	1754	9.2
	TSP	2	27595	4875	4690	1674	8.2
94e	CON	0	25031	5123	4360	177	0.9
	COM	1	24451	4714	3911	334	1.6
	COM	2	25935	4822	3810	200	1.0
	MIX	1	25074	5794	4965	21	0.1
	MIX	2	25240	4802	4257	152	0.7
	MIX	3	25958	5079	4134	190	0.9
	SBC	1	25925	4809	4191	270	1.3
	SBC	2	24523	4910	4305	378	1.8
	TSP	1	25188	4721	3845	270	1.3
	TSP	2	24107	4581	3965	176	0.8
94f	CON	0	26626	5233	4728	129	0.7
	COM	1	23987	4618	4040	153	0.5
	COM	2	25414	4948	3948	149	0.5
	MIX	1	25692	4844	3982	36	0.2
	MIX	2	25634	4757	3948	317	1.4
	MIX	3	26442	5075	4085	183	0.9
	SBC	1	29265	4101	4400	141	0.6
	SBC	2	25385	5184	4722	316	1.6
	TSP	1	23961	4427	3732	238	1.0
	TSP	2	24665	4722	3894	199	0.9

(1) = Average of 4 replicates.

93e = tissues sampling on half plots that will not receive additional treatments application 1994.

93f = tissues sampling on half plots that will receive additional treatments application 1994.

94e = tissues sampling on half plots that have not received additional treatments application 1994.

94f = tissues sampling on half plots that have received additional treatments application 1994.

Table B-5. Corn N, P, K, Yield and P uptake (1)

Tissue sampling	Residue	Rate	CORN GRAIN CONTENT AND P UPTAKE					CORN STOVER CONTENT AND P UPTAKE				
			N	P	K	YIELD	P UPTAKE	N	P	K	YIELD	P UPTAKE
			(mg/kg)	(mg/kg)	(mg/kg)	(kg/ha)	(kg/ha)	(mg/kg)	(mg/kg)	(mg/kg)	(kg/ha)	(kg/ha)
93e	CON	0	14084	3173	4569	4641	14.7	7557	950	9289	4338	4.2
	COM	1	13016	2987	4550	4487	13.5	8299	964	9079	4141	4.0
	COM	2	16318	3823	4502	5058	19.4	7338	735	10488	4283	3.1
	MIX	1	13678	3043	4536	4858	14.8	7375	856	10778	4229	3.6
	MIX	2	13207	2960	4495	4820	14.3	8676	824	11019	4153	3.4
	MIX	3	13256	3059	4734	4970	15.3	7618	845	9158	3629	3.0
	SBC	1	14264	3456	4805	5219	18.0	8308	790	11186	4481	3.5
	SBC	2	13951	3401	4707	5756	19.6	7657	756	9535	5155	3.9
	TSP	1	13879	3193	4781	4565	14.5	8249	987	9061	4123	4.0
	TSP	2	14015	3151	4882	4872	15.3	8250	974	12978	4292	4.3
93f	CON	0	13583	3144	4905	4469	14.0	8424	874	10302	3832	3.3
	COM	1	13732	3145	4603	5001	15.8	7342	1032	10495	4343	4.3
	COM	2	13344	3140	4470	5061	16.0	7058	865	11016	4576	4.0
	MIX	1	14012	3209	5111	4905	15.8	7397	841	9307	3880	3.3
	MIX	2	13770	2828	4321	5100	14.4	6799	809	12179	4193	3.6
	MIX	3	13027	2984	4209	5139	15.3	7506	898	12119	4629	4.1
	SBC	1	12874	2944	4384	5219	15.4	7443	828	11371	4750	3.9
	SBC	2	12826	2809	5556	4572	12.8	8189	841	10359	4184	3.6
	TSP	1	13625	3001	4598	4808	14.5	7496	839	10505	5001	4.2
	TSP	2	13727	3253	4442	5116	16.8	6613	827	11232	4004	3.3
94e	CON	0	12211	2515	3928	805	1.9	5229	1939	11141	4782	9.2
	COM	1	12032	2647	3733	878	2.1	9300	2089	7652	4169	7.9
	COM	2	11829	2290	3527	1767	3.4	4068	1622	8825	4665	6.3
	MIX	1	11304	2016	2694	3582	7.3	4883	1815	9682	4702	7.9
	MIX	2	10611	2260	3679	2363	4.7	4703	1454	9241	4625	6.7
	MIX	3	11787	2560	3960	721	1.8	4389	1781	9150	3845	6.3
	SBC	1	13442	3365	4237	358	1.0	4390	2176	8664	4160	8.7
	SBC	2	12057	2322	3876	1307	2.8	5792	1979	8011	3813	5.2
	TSP	1	11590	2287	3814	1984	4.1	4751	1648	7972	4934	6.7
	TSP	2	11367	2330	3228	1352	3.1	4873	1813	8966	4171	7.0
94f	CON	0	11976	2234	3460	1065	2.2	4801	1524	8254	5369	7.1
	COM	1	10914	2079	3117	1484	4.0	5166	2073	9168	3985	8.0
	COM	2	11066	2631	3599	2124	4.3	5551	2514	8011	3832	7.4
	MIX	1	12412	2364	3321	719	2.4	5072	2150	9621	4123	7.3
	MIX	2	12275	2529	3869	774	1.7	4533	2194	9211	4140	8.4
	MIX	3	10635	2270	3545	2969	6.5	4712	1529	9523	4730	6.6
	SBC	1	11752	2376	3361	1940	4.4	5114	1832	9500	4567	8.0
	SBC	2	11095	2454	3043	1616	5.3	4636	1991	7608	4149	7.8
	TSP	1	13500	2876	4440	456	1.2	6630	2753	8970	3217	7.9
	TSP	2	12053	2537	3991	1188	2.4	5139	1889	8466	4639	8.0

(1) - Average of 4 replicates.

93e - tissues sampling on half plots that will not receive additional treatments application 1994.

93f - tissues sampling on half plots that will receive additional treatments application 1994.

94e - tissues sampling on half plots that have not received additional treatments application 1994.

94f - tissues sampling on half plots that have received additional treatments application 1994.