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ORIGINAL ARTICLE

Statistical model to estimate Physicochemical Parameters of soilcompost Mixtures and their relationship with Blackberry Production

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ABSTRACT

The aims of this research were: 1) to deduce a statistical model to determine the PZC and the Ψ_0 of soil-compost mixtures; 2) to know the relationship of these parameters on blackberry dry matter production and 3) to know the influence of the sugar cane rum compost addition, on the blackberry dry matter production. It was worked with 8 treatments: 100, 50, 25, 12.5, 6.25, 3.125, 1.562 and 0.0% respectively of soil-compost mixtures. The pH at soil, soil-SCRC mixtures and pure SCRC was determined in an aqueous suspension in 1:5 and 1:15 ratios respectively. PZC and Ψ_0 to the soil and the mixtures were determined. The addition of SCRC increased the soil pH from 6.05 to 7.5. The mixtures PZC were increased with the SCRC additions from 4.0 to 7.0. The soil Ψ_0 was increased from 0.4348 to 80.0 mV. The SCRC addition increased the blackberry dry matter production, which showed a close relationship with the PZC and Ψ_0 behavior respectively. The deduced statistical model: $\Psi_0 = D(pH - PZC)$ from data herein, was functional for estimating the physicochemical parameters studied.

Keywords: Blackberry, statistical model, sugar cane rum compost, zero point of charge, surface electric potential.

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INTRODUCTION

Nowadays, environmental pollution by organic solid waste management, and fossil fuels burning for chemical fertilizers production, has worried Man, especially global warming caused by the greenhouse gas generation (CO₂, CH₄ and N₂O). The sugar industry is generating pollution by-products such as sugar cane rum (SCR), which is transformed through a humification process in compost, whose quality depends on its chemical and physicochemical characteristics. When this compost is applied to soil, dew to its physicochemical characteristics, it improves its physical properties, such as soil structure, porosity, aeration, water holding capacity, promoting root growth, and consequently, decreases the use of chemical fertilizers [1].

The physicochemical characteristics of soils, composts, organic fertilizers, and so on, related to agricultural production like pH, cation exchange capacity, anion exchange capacity, point of zero charge (PZC), surface electric potential (Ψ_0), diffuse double layer, and isomorphic substitution, are important parameters, because from they depend the agricultural species to grow, its yield and harvest quality.

Among the soils and compost physicochemical characteristics, which influence on crop production, it is the PZC, which is defined as the suspension pH, in which the net surface charge is zero [2-4].

The PZC of soil colloids, is a useful and meaningful chemical parameter, because the surface charges are positive if pH > PZC and they are negative if pH < PZC [5]. This point is dependent on the surface properties and the solution composition [6] and is the most important characteristic for describing a reversible surface charge [7]. In general, it can say that the PZC reflects the system overall composition [8]. For the soil systems, the PZC reflects the average weight of organic and inorganic constituents [9] and can be determined by a titration curve mass [10].

The presence of Fe and Al oxides tends to increase the PZC towards higher pH values, whereas clay minerals with permanent negative charge, as well as organic matter change it towards lower pH values [11].

Organic matter plays an important role in soils, because increments the Ψ_0 and contributes to the negative charge at field pHs, because of adsorbed PDI [12].

In soil system, there are two types of charges: 1) the constant charge from clay isomorphic substitution and 2) the variable charge on organic matter under the solution pH, from the functional groups dissociation of the humic substances.

The surface charge of soil colloidal system, can be positive or negative charge [13, 6]and according to Morais *et al.* [9], any material that increases the soil negative charge, increases the PZC, while those which increase the positive charge, decrease its value, so they are responsible for the variation of the above parameters in the soil, and consequently, any action performed with the object of changing electrical flow in the soil, will modify its fertility degree and thus, the crops productivity. An example for this Parks [8] in his works, indicates that with the organic matter application, it is adding positive charges; however, others researchers differs from this criterion, such as Stevenson [14] who indicates, that when it is added organic matter in the soil, humic substances are added, and with them are incorporated negative charges, increasing the soil pH and PZC, and therefore, the soil Ψ_0 .

Gillman and Uehara [3] indicate that at the pH of the PZC, the PDI adsorption is the same, and it is the intersection point of potentiometric titration curves, at different electrolyte concentrations.

H⁺ and OH⁻ ions have an important influence on the surface electric properties of soil colloids, which affect significantly their fertility level, therefore, they have been considered as potential determining ions (PDI) [7], so adding an organic material to the soil, can increment positive or negative charges, depending on its humification degree, as generally with lower humification degree the soil solution pH is lower, and can fall into the acid range,and then,itwould add positive charges. On the other hand, if the humification level is high, it would fall in the alkaline range, and it would be added negative charges; that is, pH, PZC and Ψ_0 , among others, depend on added H⁺ and OH⁻ ions, so Van Raij and Peech [2] indicated that the PZC is a characteristic value for a reversible phase. They add that if it is assumed that the surface charge originates only from the PDI adsorption, the Ψ_0 due to charges is determined by the solution pH in equilibrium, and is given by the Nernst equation, that is: $\Psi_0 = kT/e Ln (H^+)/(H^+)PZC = 59(PZC - pH) mV$ at 25°C.

Uehara and Gillman [15] mention that: a) the particles surface charge rises from defects in its interior, or from the adsorption of PDI, b) there are soils which don't have minerals with permanent charge, which can be treated as pure charge variable systems and c) the overall charge density of a mixing system, like soil is: $\sigma t = \sigma p + \sigma v$; where: $\sigma t = total$ surface charge density, $\sigma v =$ surface variable charge density, $\sigma p =$ surface constant charge density. According to these researchers for a 1:1 indifferent electrolyte and at pH in the range of pH₀ ± 1: $\sigma v = 1.67 \times 10^{-6} n1/2$ (PZC – pH), and $\sigma p = -1.67 \times 10^{-6} n1/2$ (PZC – pH); where n = electrolyte concentration in number of ions per cm³.

Yu [16] says that the Van Raij and Peech [2] model, is not consistent with reality and that it is necessary to propose models to describe the relationship between the PDI and the Ψ_0 . It also indicates that several models have been developed. The basic points of these models are almost identical: material balance equation, law of mass action and statistical mechanics, all they are used to describe the chemical balance of these ions, that cause the occurrence of Ψ_0 and the contribution of these ions to the surface charge, so that the interrelationships between Ψ_0 and the PDI can be formulated through thermodynamic considerations.

The aims of this research were: 1) deduce a statistical model to determine the PZC and the Ψ_0 of soilcompost mixtures, 2) to know the relationship of these parameters on blackberry dry matter production and 2) to know the influence of the sugar cane rum compost addition on the blackberry dry matter production.

MATERIALS AND METHODS

The present research was carried out in Ziracuaretiro,Michoacan state, Mexico with blackberry production soils area. The soil is classified as a Chromicluvisol [17],pH in a 1:2 ratio soil water suspension was measured with a glass-calomel electrode potentiometer (Martini instruments, Mi 180). N was determined by the Kjeldahl technique [18]; the P by the Bray P₁technique [19]; the K, Ca and Mg cations using ammonium acetate solution at pH 7.0 [20] (Table 1).

Preparation and chemical analysis of sugar cane rum compost (SCRC)

The sugar cane rum (SCR) was obtained from Taretan, Michoacan, Mexico sugar mill which was subjected to an eight months composting period by passive aeration technique [21].

The chemical analyzes performed on the SCRC were: pH measured in a suspension SCRC:H₂O and SCRC:KCl 1.0 N solution at a 1:5ratio, a potentiometer was used with glass-calomel electrodes(Martini instruments, Mi 180) [22]. The Δ pH was calculated with the formula Δ pH =pH_(KCl) - pH_(H2O). The CEC was measured using the technique developed by Harada and Inoko [23] (the displacement solution pH was adjusted to that of the SCRC. The extraction of humic acids was carried out with a mixture of 0.1 M sodium hydroxide and 0.1 M sodium pyrophosphate according to Kononova *et al.* [24]. The total content of functional groups was determined by the method proposed by Dragunova, quoted by Kononova *et al.* [24]. Total organic carbon was determined by the TOC-5050A analyzer equipment and ASI 5000 autosampler. Total nitrogen content was determined by the Kjeldahl technique [18].The optical density ratio (E₄/E₆) was obtained by dividing the humic acids absorbance at a wavelength to 465 nm by that at 665 nm [26]. The equipment CARY 50 Conc. UV-Visible Spectrophotometer VARIAN it was used (Table 2).

Preparation and conditioning of soil-SCRC mixtures

Soil-SCRC mixtures were prepared by applying abinary method (Table3).

The soil samples and their corresponding SCRC-soil mixtures were washed 12 times by repeated centrifugations with 0.001M HCl to remove interchangeable cations and anions; since the fourth washing presented colloids deflocculation, so it was necessary to ultracentrifuge in successive washings. Then 8 washings more were given with distilled water until free of chloride (applied AgNO₃ test). Then the samples were washed by shaking with a solution of an indifferent electrolyte (1.0M KCl), after which they were centrifuged removing the supernatant solution. Subsequently, they were washed with water, and by repeated centrifugations, the KCl concentration was decreased about 0.002M, (the ultracentrifuge was used starting from the third washing). Finally, samples were dried in an oven at 70°C for 72 hrand sieved through a mesh of 0.8 mm [15].

Determination of PZC and ψ_0

PZC was determined by the technique proposed by Gillman and Uehara [3] which is as follows: 4 g of the soil sample were placed in each of 10 centrifuge tubes of 50 mL with 10 mL of 0.002M KCl. The pH was adjusted of each of the 10 tubes so as to cover a pH range in which it been found the zero point of charge (6.5, 6.0, 5.5, 5.0, 4.5, 4.0, 3.5, 3.0, 2.5, 2.0). Make up to 20 mL with 0.002M KCl. The suspension was balanced for 4 days with stirring each 24 hours. The pH at equilibrium (pH 0.002M) was recorded. Add 0.5 mL of KCl 2.0 M solution and shake gently each 3 hours and record the pH (pH0.05M). The ΔpH (pH0.05M - pH0.002M) for each of the tubes were calculated. The pH 0.002M vs ΔpH were plotted. The PZC corresponds to a pH_{0.002M} wherein the pH = 0. Finally, the $ψ_0$ was determined by the equation proposed by Uehara and Gillman [15]: $ψ_0$ = 59 (PZC - pH) for variable charge systems and by a model derived from data herein: $Ψ_0$ = D(pH - PZC) where D = SCRC dosage.

Greenhouse experiment

An experiment was conducted in a greenhouse in which the blackberry (*Rubus*spp) response to the application of the SCRC at different dosage (0, 1.875, 3.75, 7.5, and 15 g kg⁻¹ of soil) was studied. A randomized block design with four repetitions was employed. For its installation five kg of soil were placed in pots dried in the shade and filtered through a mesh of 2 mm, then added air-dried SCRC in the quantities shown in the top. They were fertilized with 50, 50 and 100 mg of N, P_2O_5 and K_2Oby kg of soil respectively. As source of N was applied urea (46% N) to P, calcium triple superphosphate (46% P_2O_5) and to K, potassium chloride (60% K_2O). The three ingredients (soil, SCRC and fertilizer) were mixed and distilled water was added to field capacity. Then two blackberry seedlings with two month old were transplanted per pot and finally was left the most vigorous. The plants were watered twice a week until harvest. After 45 days transplanting, a second fertilization was carried out with 50 mg of N per pot. At 90 days after transplantation they were harvested by cutting the plants two centimeters above the ground level. Plant dry matter production and plant height data were taken. The plants were washed with tap water to remove soil particles and rinsed quickly with distilled water they were dried in an oven(CRAFT, Model SW-17TA) at 70°C for 72 hr. Later dry matter weight was obtained.

RESULTS AND DISCUSSION

Chemical and physicochemical properties of the soil were affected by the SCRC additions at different rates, the increase in the proportion of it, caused an increase in pH, as well as the PZC, and the Ψ_0 (Table 4).

The PZC elevation found in this study is consistent with the results obtained by Morais *et al* [9], as they found that the addition of organic matter increases PZC; however, does not agree with Sparks (1995), as he found that if the surface charges are positive, pH >PZC, and if they are negative charges, the pH < PZC. In this work, it was found that with the soil organic matter addition, the organic colloids surface charge is negative and pH > PZC. However, in this case, the pH and PZC increases were because with the SCRC

addition, the soil had a supply of negative charges from the functional groups dissociation, constituents of humic and fulvic molecules, and consequently, pH dependent variable charge, so it was generated an statistical model, to estimate the surface electric potential from the experimental data obtained in this work, taking into account the proposal of Yu [16], who mentioned that it is necessary to propose models to describe the relationship between the PDI and the Ψ_0 . In this sense, it has been developed a statistical model by applying the following steps:a)plot the compost dosage data vs pH and PZC data (Fig. 1), b) get statistical models of the corresponding trend lines, c) get the differentiation of statistical models for its standardization, d) get the integrals of each equation, e) add up to integrals algebraically to obtain the surface between the two trend lines (Ψ_0), f) to relate the regression coefficients with the y-axis, ie the pH and the PZC and substitute v g) obtain the general model.

Statistical models:

$$y_1 = -0.0003x^2 + 0.0383x + 6.4551$$

The x^2 coefficient (-0.0003) corresponds to the curvature (c_1) of the trend line and x coefficient corresponds to its slope (m_1) :

Obtain the differential of the model

 $y_1 = c_1 x^2 + m_1 x + 6.4551$ $\partial y_1 = 2c_1 x \partial x + m_1 \partial x$

Obtain the integral of the differential of the model

 $2c_1 \int x \partial x + m_1 \int \partial x$

When the slope tends to zero, c = 0 therefore:

$$2c_1 \int x \partial x + m_1 \int \partial x = 0 + m_1 \int \partial x = m_1 \int \partial x$$

Second model:

$$y_2 = -0.0005x^2 + 0.0662x + 4.5217$$

As in the first model, the x^2 coefficient (- 0.0005) corresponds to the curvature (c_2) of the trend line and x coefficient corresponds to its slope (m_2) .

$$y_2 = c_2 x^2 + m_2 x + 4.5217$$

Obtain the differential of the model

 $\partial y_2 = 2c_2 x \partial x + m_2 \partial x$

Obtain the integral of the differentials of the model $2c_2\int x\partial x + m_2\int \partial x$

When the slope tends to zero, c = 0 therefore

 $2c_2 \int x \partial x + m_2 \int \partial x = 0 + m_2 \int \partial x = m_2 \int \partial x$

The algebraic sum of the integrals corresponds to the Ψ_0

 $\Psi_0 = m_1 \int \partial x \cdot m_2 \int \partial x$

Factoring

Integrating

$\Psi_0 = (m_1 - m_2)x$

 $\Psi_0 = (m_1 - m_2) \int \partial x$

When the slope (m) tends to zero, m₁ becomes the pH and m₂becomes the PZC, consequently:

 $\Psi_0 = (pH - PZC)x$

But x corresponds to the compost dosage, therefore:

$$\Psi_0 = D(pH - PZC)$$

The estimated Ψ_0 with the formula $\Psi_0 = 59$ (PZC - pH) in all cases yielded negative figures, which does not make sense, however, when applying the proposed model herein, data are positive (Table 4).

By linking increases the SCRC dosage with the soil-compost mixtures pH variation, was observed an increase in these physicochemical parameter values, passing from the acid range (pH = 6.43) with the minimum compost dosage (0.195%), to the alkaline range (pH = 7.8) with the maximum compost dosage (100%) (Table 4).

This behavior is explained considering that the studied compost is a source of one of the potential determining ions (OH); that is, of negative electric charges; as a result, the physicochemical parameters studied in this research, are affected in the same sense that the pH, although according to Parks [8], with the soil organic matter application it is adding positive charges, however, the HS from applied SCRC, contain unstable functional groups, whose dissociation rate is dependent on the medium pH [26], and at a pH in the alkaline range, there are release of OH⁻ which is one of the potential determining ions.

On the other hand, the trend in PZC increasing, agrees with the results obtained by Fernandez Caldas et al. [27], who found that soils with higher organic matter content showed higher point of zero charge.

The soil Ψ_0 was affected in the same direction as the PZC and pH, as by increasing the soil variable charge there was a tendency to increase its value to a maximum level, according to the proposed model (Table 4).

There was a high relationship between increases in the dosage of compost applied and the values of physicochemical parameters studied: pH, PZC and Ψ_0 ; however, increases in the production of dry matter were observed until dosage of 6.25% (Fig. 4) and from this dosage, dry matter productions were decreasing to a minimum with the maximum dosage. This behavior is due to the mixture soil-compost pH to this dosage, was in the acid range (pH = 6.9), and from this, its value changed to the alkaline range up to a maximum pH of 7.8, whereupon the nutrient dynamics of some nutrients from the blending was modified [24]. On the other hand, increases in dry matter production are due to the increases in the nutrient contents of the mixture, especially those of N.

All data contained in table 4, indicate that of all physicochemical characteristics studied in the mixture soil-compost, only pH was which influenced significantly on the blackberry dry matter production. The blackberry dry matter production was affected by soil SCRC additions, while the higher the addition of this was, the higher dry matter production was, observing a very close relationship ($R^2 = 0.90$) between the Ψ_0 and the ZPC behavior with the dry matter production (Table 4).

It was found a significant correlation between the Ψ_0 and dry matter production (R² = 0.74) (Fig. 2), in mixtures that had an acid pH; this is because when adding organic matter to the soil, improving their physical conditions, such as structure, and thereby its water retention capacity, radiant energy absorption, increases the beneficial microorganisms proliferation; in general, improving the conditions for the crop roots growth, promoting the better plant growth. However, going to the alkaline range, production was decreased (Table 4), presumably because the dynamics of nutrients, especially micronutrients changed, making them less available [28].

There was a very close lineal relationship ($R^2 = 0.9642$) between the ZPC and the soil Ψ_0 (Figure 3), which is because with the negative charges addition, the ZPC approaching the soil pH, increasing its value and, consequently, increasing the Ψ_0 which is a function of these two parameters.

The behavior of dry matterproduction with respect to the Ψ_0 , the PZC and the pH of the soil-SCRC mixtures, was similar, that is, corresponding to a quadratic model, so that at the point of inflection of the curvature, dry matter production starts to decrease until a minimum, with the maximum values of these parameters.

An important aspect to be considered in this research, is that with the SCRC addition, humic acids were added to the soil (Table 4), which are a source of negative charges, that interfere with all the phenomena of surface charge of the colloids, and consequently, with crop yields. The humic acids content was directly proportional with the SCRC added to the soil (Figure 4).





 $\label{eq:surface} Surface\ Electric\ Potential\ (\Psi_0)$ Figure 2: Relationship between surface electric potential and dry matter production.



Figure 3: Relationship between Point of Zero Charge (PZC) and dry matter production





Table 1: Soil Chemical Analysis										
	т.П	OM	С	Ν	C/N	P (Bray P ₁)	Κ	Са	Mg	
I	рн _(Н20)	%	%	%	(mg/kg) (Cmol _c /J			nol _c /kg o	kg of soil)	
	6.05	5.80	3.37	0.29	11.63	4.00	1.29	6.00	2.25	
OM:	OM: Organic matter									
Table 2: Sugar cane rum compost chemical analysis										
pH _(H20)			7.80		E_4/E_6		3.866			
pH _(KCl)			7.50		С%	52.34		34		
ΔрН			-0.30		N (%)	2.26				
Humic Acids (%)				8.80		C/N	23.16			
Functional groups (me/100g)				500		$CEC (Cmol_c/kg) \qquad 63.28$		28		
-COOH groups (me/100g)					409					

me = miliequivalent

Table 3: Preparation of soil-sugar cane rum compost mixtures

Mixture	Mixture Composition				
number	Soil (%)	Sugar cane rum compost (%)			
1	100.000	0.000			
2	99.805	0.195			
3	99.610	0.390			
4	99.219	0.781			
5	98.438	1.562			
6	96.875	3.125			
7	93.750	6.25			
8	87.500	12.500			
9	75.000	25.000			
10	50.000	50.000			
11	0.000	100.00			

Table 4: Soil chemical and physicochemical parameters variation by effect of adding different SCRC proportions

			prop	010100		
Compost			$\Psi_0 (mV)$	Ψ_0 (mV)		Dry Matter
	pH(H ₂ O)	PZC	$\Psi_0 = D(pH -$	$\Psi_0 = 59(PZC -$	Humic acid	(g/plant)
Dosage (%)			PZC)	pH)	(g)	
0.000	6.05	4.00			0.000	2.732
0.195	6.43	4.2	0.4348	-131.57	0.017	3.300
0.390	6.48	4.3	0.8502	-128.62	0.034	3.860
0.781	6.50	4.3	1.7182	-129.8	0.069	4.220
1.562	6.58	4.35	3.4832	-131.57	0.138	4.537
3.125	6.65	4.4	7.0312	-132.75	0.275	4.641
6.25	6.90	4.46	15.250	-143.96	0.550	4.650
12.500	7.02	5.47	19.375	-91.45	1.101	4.555
25.000	7.35	5.49	46.500	-109.74	2.202	4.442
50.000	7.55	6.5	52.500	-61.95	4.404	3.954
100.00	7.80	7.0	80.000	-47.2	8.809	3.186

 Table 5: Relationships between sugar cane rum compost added to the soil, with dry matter production, point of zero charge and surface electric potential

	Sugar cane rum compost added (%)					
Parameter	0.00	0.195	0.391	0.781	1.562	
Dry matter (g/kg soil)	2.732	3.300	3.860	4.220	4.537	
PZC	4.00	4.20	4.30	4.30	4.35	
$\psi_0(mV)$		0.4348	0.8502	1.7182	3.4832	

PZC: Point of zero charge.

 Ψ_0 : Surface electric potential.

CONCLUSIONS

- The deduced statistical model from data herein, is functional for estimating the physicochemical parameters studied.
- The physicochemical parameters considered (PZC and Ψ_0) in soil-compost mixtures, were related to blackberry dry matter production through a quadratic equation.
- The pH of the soil-compost mixtures had a positive effect on blackberry dry matter production in the acid range, but in the alkaline one, production was reduced.

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