### Composting a digestate from the organic fraction of urban solid wastes

## Composteando el digestato de la fracción orgánica de residuos sólidos urbanos

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#### Abstract

A biosolid was produced from the composted digestate of the organic fraction of urban solid wastes (OFUSW). During a first hydrolytic-acidogenic stage, the milled OFUSW mass is transformed into volatile fatty acids and other soluble or suspended substances in the leachate while the residual solids constitute the digestate. To obtain a composting mass (CM) with a C/N between 25 and 35 and a matrix to allow aeration, mixtures of digestate ( $f_D$ ), OFUSW ( $f_F$ ) and sawdust ( $f_S$ ) were prepared using a simplex centroid design. Results were adjusted to a multiple regression model with volatile solids degradation efficiency ( $\eta_{VS}$ ) as a response variable. It was found that the operation zone with the highest  $\eta_{VS}$ , were CM with the following compositions:  $f_D$ : 0.425-0.625,  $f_F$ : 0.275-0.450; and  $f_S$ : 0.1-0.15. Three assays were carried out in a bench scale reactor, the final biosolids had a pH = 7.5, a C/N = 15 and a germination index (GI) = 84%. A linear correlation between  $\eta_{VS}$  and CO<sub>2</sub> production was proposed for process control. A rapid CM stabilization was reached, up to  $\eta_{VS} = 35\%$  in 12 days and the biosolids with high GI, can be used to improve soils.

Keywords: Composting matrix, simple centroid design, biosolids, temperature, C/N ratio.

#### Resumen

Se produjo un biosólido composteando el digestato de la fracción orgánica de sólidos urbanos (FORSU). La masa molida de FORSU es transformada en ácidos grasos volátiles y otras sustancias solubles en el lixiviado, mientras que los sólidos no degradados conforman el digestato. Para obtener una masa de compostaje (CM) con una C/N entre 25 y 35 y una matriz que permita la aireación, se prepararon mezclas de digestato ( $f_D$ ), FORSU ( $f_F$ ) y aserrín ( $f_S$ ) utilizando un diseño experimental simplex centroide. Los resultados se ajustaron a un modelo de regresión multiple para la eficiencia de degradación de sólidos volátiles ( $\eta_{vs}$ ) como variable respuesta. Se encontró que la zona de operación con la  $\eta_{vs}$  mas alta fue con una CM con la siguiente composición:  $f_D$ : 0.425-0625,  $f_F$ : 0.275-0.450;  $f_S$ : 0.1-0.15. Se diseñó un reactor de 95 L donde se realizaron tres ensayos. Se obtuvo un biosólido con un pH =7.5, una C/N=15 y un índice de germinación (IG) =84%. Se propuso para el control del proceso una correlación lineal entre  $\eta_{VS}$  y la producción de CO<sub>2</sub>. Se alcanzó una rápida estabilización de CM hasta  $\eta_{vs}$  =34% en 12 d pudiendo utilizarse el biosolido como mejorador de suelos.

Palabras clave: Digestión anaerobia, compostaje, diseño simplex centroide, biosolidos, relación C/N.

## 1 Introduction

Mexico City generates around 6,700 T/day of the organic fraction of urban solid wastes (OFUSW) mainly composed of food and park residues (Campuzano and González-Martínez, 2016) and is composted in plants or dumped in landfills located far away from the city. This practice cause soil and aquifer pollution (Assmuth and Strandberg, 1993).

A two stages anaerobic digestion (2SAD) process has been proposed to reduce 70% the OFUSW mass (Paixão *et al.*, 2000), reclaim nutrients back to agricultural soils and to obtain methane (Rodríguez-P. *et al.*, 2015, Rivas *et al.* 2020). In the first stage, in an anaerobic hydrolysis leaching bed (AHLB) reactor the milled OFUSW is fermented to produce a leachate and a digestate. The former, a liquid, highly concentrated in soluble organics, mainly volatile fatty acids, is sent to an upflow anaerobic sludge blanket (UASB) reactor to produce methane.

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The latter, a sludge which contains microbial biomass and undigested solids with a carbon to nitrogen ratio (C/N) of  $16 \pm 3$ , a moisture content of 85% and a variable composition, determined by the incoming OFUSW which in turn depends on the season and management practices (Bibby et al., 2010). A composting process, required to stabilize, deodorize and disinfect the digestate and turn it into a biosolid or soil conditioner, is the exothermic (4.1 to 9.46 kcal/g organic matter) partial oxidation of wet organic matter (Livesey et al., 2000) resulting in humus, ammonia, nitrate and carbon dioxide (Kelleher et al., 2002; Kuo et al., 2004). The composting mass (CM) must be well aerated in order to generate enough heat to raise the temperature to 50 °C (Kulcu and Yaldiz, 2004) and to ease the airflow through, materials which increase its porosity are need (Batham et al., 2013). Due to temperature increase there is a selection of the microorganisms taking part in each composting stage (Franke-Whittle et al., 2014). Enough nitrogen for growth is provided with a C/N between 25 and 35. Lower values would mean loss of nitrogen (Jiang et al., 2011) and higher values will mean nitrogen limitation for cell growth and therefore no heat generation (Hao and Benke, 2008). A compost to be used as biosolids must provide environmental conditions for seeds to yield high germination index (GI).

The aim of this work is to define the production conditions of a soil conditioner by composting a digestate complemented with OFUSW to increase the C/N and sawdust to improve aeration. To determine the optimal mixtures composition a simplex centroid design coupled to surface response methodology was applied.



Fig. 1. Compost ternary plot. Restriction area  $\blacksquare$  and selected points  $\bullet$ .

# 2 Materials and methods

## 2.1 Selection of mixtures for digestate compost

Digestate was composted together with sawdust and OFUSW inoculated with compost from the *Bordo Poniente* Compost Plant at Mexico City. Several mixtures with initial C/N (C/N<sub>i</sub>) between 17 and 22.5 were prepared. Glass bottles (1.4 L) containing the composting mass (650 -740 g) were incubated at 35 °C in a water bath (Perkin Elmer Polystat12104-00) and aerated based on the initial volatile solids at 0.5 L/(min·kgi·VS), or 0.273 L/(min·kgi OFUSW (Iñiguez *et al.*, 2019). A rubber stopper with two tubes allowed the gas entry and exit (Monroy *et al.*, 2020, figure S1).

# 2.2 Delimitation of experimental or restriction area

The restriction area is the set of mixtures that uses the digestate as the main component and maintains a C/N<sub>i</sub> close to 20. The component diagram (figure 1) shows all the combination of mixtures and the restriction area generated by the three components, weight fractions are expressed on dry basis. The limits of the restriction area are: Digestate fraction ( $f_D$ ): 0.45 to 0.70, OFUSW fraction ( $f_F$ ): 0.20 to 0.45, Sawdust fraction ( $f_S$ ): 0.10 to 0.35. The inoculum mass is constant at 5% w/w in all mixtures and is not considered in the ternary plot.

### 2.3 Experimental simplex centroid design

The simplex centroid design with 3 components consists of 10 points representing the mixtures: one located at the centroid of the operation area, 3 at the apexes, 3 at the midpoint between the sides and the centroid, 3 more were located at the midpoints between the apexes and the centroid (Aguilera *et al.*, 2001). The results were analyzed with Scheffé statistical model (Scheffé, 1963), by using PASW Statistics 18, which resulted in polynomial equations 1 and 2 for the response variables, final C/N (C/N<sub>f</sub>) and the volatile solids removal efficiency ( $\eta_{vs}$ ):

$$CN_{final} = \beta_1 f_D + \beta_2 f_F + \beta_3 f_s + \beta_4 f_D f_F + \beta_5 f_D f_S + \beta_6 f_F f_S + \beta_6 f_D f_F f_S$$
(1)

$$\nu_s \nu = \gamma_1 f_D + \gamma_2 f_F + \gamma_3 f_s + \gamma_4 f_D f_F + \gamma_5 f_D f_S + \gamma_6 f_F f_S + \gamma_6 f_D f_F f_S$$
(2)

where  $\beta_i$  and  $\gamma_i$  are the model coefficients.

#### 2.4 Composting bench scale reactor

A 95.4 L (45 cm diameter, 60 cm long) galvanized steel reactor (figure S2 Monroy *et al.*, 2020) covered with polyurethane foam (2.85 cm thickness) was constructed. It was placed on a 300 kg capacity weighing scale (Braunker YP200) to monitor the weight loss during the compost process. Temperature was monitored with two type J thermocouples. A manual stirring device mixes the composting mass for five minutes per day. Aeration was kept at the same rate as in the glass bottle experiments (0.5 L/(kg<sub>i</sub>VS·min)).

#### 2.5 Analytical techniques

- 2.5.1 Moisture content and organic matter as volatile solids (VS) were analyzed by standard methods (Wu *et al.*, 2000).
- 2.5.2 Carbon and nitrogen were determined with an elemental Analyzer Series II 2400 CHNS/O (Perkin Elmer, Boston, USA) using acetanilide (C8H9NO) as standard. Fresh samples were dried in aluminum trays at 60 °C for 3 days and ground to particle size lower than 100 mesh. In the CHN operating mode, samples of 1.5 to 2.5 mg were weighed in a high precision microscale (Perkin Elmer) and burned to combustion to convert the sample elements to simple gases (CO2, H2O and N2). Other elements, such as halogens and sulfur are removed by scrubbing reagents in the combustion zone. Temperatures in the combustion, reduction and detector zones were 925 °C, 640 °C and 82.5 °C, respectively. Helium and argon are used as carrier gases and oxygen for the combustion of samples.
- 2.5.3 pH was measured with a calibrated potentiometer (HANNA HI 255) on wet 5 g samples suspended in 10 mL of distilled water, stirred for 30 min and reposed for 15 min (Zhang *et al.*, 2004).
- 2.5.4 Reactor temperature was measured using two type J thermocouples while the environmental

greenhouse temperature was measured using a thermistor coupled to a data logger and NOVUS My PCLab logging data every 20 min.

2.5.5 CO<sub>2</sub> content in the exit air was measured every 20 min by passing the exit gas stream through a silica-gel packed column for dehydration and then to a Fuji ZFP9 infrared analyzer (Figueroa *et al.*, 2011) calibrated with prepared CO<sub>2</sub>/air mixtures. Data was integrated to obtain the specific CO<sub>2</sub> production rate (equation 3).

$$m_{CO_{2i}} = \frac{y_{CO_2} F_i P \, M W_{CO_2}}{M_o R T_i} \tag{3}$$

where

 $m_{CO_2}$ : Specific CO<sub>2</sub> production rate (gCO<sub>2</sub>h<sup>-1</sup>kg<sup>-1</sup><sub>SVo</sub>)  $y_{CO_{2i}}$ : CO<sub>2</sub> volume fraction at the exit gas stream  $F_i$ : Flow rate at the exit gas stream (L h<sup>-1</sup>) P: Atmospheric pressure at México City/(0.7697 atm)  $MW_{CO_2}$ : molecular weight  $M_o$ : Intial dry matter (kg)  $T_i$ : instantantaneous temperature (K)

- R: Universal gas constant at STP
- 2.5.6 Germination Index (GI): Determined according to Huang *et al.* (2004). A sample of 5 g of compost was suspended in 50 mL distilled water, agitated for 1 h in with magnetic stirrer and then filtered. Samples of 5 mL filtrate and a distilled water control were placed in Petri dishes wetting two pieces of filter paper with 20 radish (*Raphanus sativus*) seeds and incubated in darkness at 33±2 °C for 48 h after which the size of the roots was measured and the GI obtained (eq. 4):

$$GI(\%) = \frac{Ns * \bar{L}_R}{(Ns * \bar{L}_R)^c}$$
(4)

Ns = germinated seeds,  $\bar{L}_R$  = average size of root (mm), <sup>c</sup> control.

# **3 Results and discussion**

# 3.1 The elemental analysis of the components of the mixture

From the elemental analysis of the three components of the compost mixture (table S1 Monroy *et al.*, 2020)

their empirical formula was determined: Digestate -  $C_{17}H_{32}NO_{14}$ ; OFUSW -  $C_{25}H_{45}NO_{22}$ ; and sawdust -  $C_{25}H_{40}NO_{19}$  which show that the digestate presents the lowest C/N ratio (17). In order to be composted, digestate needs to be supplemented with a carbon source as OFUSW (C/N= 23±3) and sawdust (77±10) as a structural material to allow for aeration. Ten mixtures of around 124±12 g VS with C/N<sub>i</sub> ratios between 17 and 22.5 were prepared according with the simplex centroid design and composted for 9 days (table 1).

# 3.2 Laboratory scale assays with mixtures of the simplex centroid design

All treatments exhibited a VS and C/N reduction (figure 2 and table S2 Monroy *et al.*, 2020) due to the mineralization of organic matter into CO<sub>2</sub>. C/N<sub>f</sub> and VS reduction efficiency ( $\eta_{VS}$ ) as function of the mixtures composition were adjusted to Scheffé polynomial model to determine if there is a correlation that would lead to an operation zone (eq. 5 and 6).

$$CN_f = -9.6f_F + 7.14f_D + 20.8f_S + 34.2f_F f_D + 57.1f_F f_S - 20.8f_S f_D R^2 = 0.99$$
(5)

$$\eta_{\nu}s = -154.1f_F - 53.6f_D + 72.3f_S + 552.8f_F f_D$$
  
- 3.1f\_F f\_S - 41.5f\_S f\_D R<sup>2</sup> = 0.84 (6)

Using the operation zone model, contour plots were built to characterize the operation zone which

is bounded by the initial composition of the mixture:  $f_D$ : 0.425-0.625,  $f_F$ : 0.275-0.450;  $f_S$ : 0.1-0.15 in order to obtain a biosolid with a C/N<sub>f</sub> around 13. In this operation zone, the  $\eta_{VS}$  increases as the C/N decreases, (red zone in figure S3 Monroy *et al.*, 2020).

Sawdust weight fractions  $(f_S)$  larger than 11% decrease  $\eta_{VS}$ , with most of the original material remaining in the compost. On the other hand, the operation zone corresponds to mixtures where digestate is the main component, suggesting that it contains easy biodegradable compounds due to the fact that the digestate comes from a first stage of hydrolysis-acidogenesis, unlike digestates coming from single stage anaerobic digestion where most of the biodegradable compounds are stabilized (Abdullahi *et al.*, 2008; Bustamante *et al.*, 2013; Tambone *et al.*, 2010).



Fig. 2.  $\eta_{VS}$  and C/N<sub>f</sub> from two composting repetitions of ten mixtures.

Mixture	1	2	3	4	5	6	7	8	9	10		
$f_F/f_D/f_S$ VS; (g)	20/70/10	33/58/10 123.3	25/60/15 115.8	45/45/10 112.3	35/50/15 117.7	28/53/19 114.2	20/58/23 127.7	25/50/25 135.5	33/45/23 135.2	20/45/35 148.5		
C/N <sub>i</sub>	17	18.1	18.3	19	19.1	19.3	19.5	20.4	20.7	22.5		

Table 1. Resulting mixtures with the simplex centroid experimental design.

Run/rt (days)/CM (kg)	H (%)		TS (kg)		VS (%TS)		C/N		рН		$\eta_{SV}$ (%)
	i	f	i	f	i	f	i	f	i	f	
1/17/35	79	71	7.89	6.9	90	85	29	17.5	4.5	7.6	10
2/12/19	82	82	3.5	2.3	93	79	26	13.6	4.6	7.7	34
3/12/23	83	79	3.8	3	85	70	24	15.2	4.9	7.5	21

Table 2. Composting mass (CM) characterization in the bench scale reactor.



Fig. 3. Compost reactor performance. Internal (—) and external temperatures (- - -) (a), CO<sub>2</sub> production rate (b).

#### 3.3 Assays at the bench scale bioreactor

Three runs at different retention times (rt) and CM were evaluated (table 2); the first one was fed with 35.3 kg of CM ( $f_D = 0.53$ ,  $f_F = 0.28$  y  $f_S = 0.19$ ) but the mixture resulted too compact to allow good aeration. The second test was fed with 19.5 kg ( $f_D = 0.62$ ,  $f_F = 0.28$ ,  $f_S = 0.10$ ). The third assay fed with 22.9 kg ( $f_D = 0.53$ ,  $f_F = 0.36$ ,  $f_S = 0.11$ ). The compost obtained from the second and third assays had a dark appearance and not an unpleasant odor (figure S4 Monroy *et al.*,2020).

The first run was located outside the operation zone and a low  $\eta_{VS}$  (=17%) was obtained probably because aeration was not enough and the sawdust fraction was greater than recommended by the operation zone. Runs 2 and 3 had better results (44 and 35% respectively) because aeration and solids mixing improved and the composition of mixtures were within the operation zone; resulting altogether in a high metabolic activity.

The pH increased in all experiments from acid to neutral as a result of the initial presence of organic acids in both digestate and OFUSW (Ziemiński and Frąc, 2012) and at the end of the process they were consumed and probably ammonia was also produced.

Throughout the process daily temperature oscillations are observed (figure 3a). The external temperature ( $T_E$ ) variates from 40-45 °C during the day to 18 °C during the night, this behaviour must have driven the oscillating internal temperatures ( $T_I$ ) and the heat loss through the aeration stream (Kulcu and Yaldiz, 2004) which did not allow for a temperature increase during the process (Franke-Whittle *et al.*,

2014) reached temperatures above 70 °C in a thermally isolated 100 L reactor.

CO<sub>2</sub> production rate ( $m_{CO_2}$ ) was very important in the first days of all runs (figure 3b) as a consecuence of aerobic oxidation of the high concentration of available nutrients (Bertoldi *et al.*, 1985). The first run reached a maximum  $m_{CO_2}$  value of 3.04 g CO<sub>2</sub> kg<sub>SVo<sup>-1</sup></sub> h<sup>-1</sup> resulting in a low mineralization (111 g/kg<sub>Mo</sub>). The second run had the highest  $m_{CO_2}$ (6.75 g CO<sub>2</sub> kg<sub>SVo<sup>-1</sup></sub> h<sup>-1</sup>) showing the highest volatile solids removal yield (401 g/kg<sub>Mo</sub>) and  $\eta_{SV} = 34.28\%$ . During the days, the  $m_{CO_2}$  increased while decreased during the nights. The same behaviour was observed in the temperature oscillations. Run 3 had a maximum  $m_{CO_2}$  of 3.46 g CO<sub>2</sub> kg<sub>SVo<sup>-1</sup></sub> h<sup>-1</sup> resulting in a good mixture for composting, with a total VS removal yield of 266.41 g/kg<sub>Mo</sub>.

Figure 4a shows  $m_{CO_2}$  as a function of time for the three runs. Run 2, which gave the highest temperatures and  $\eta_{VS}$  produced more CO<sub>2</sub> at higher rates than the other two assays. Figure 4b shows the linear correlation (eq. 7) between the  $\eta_{VS}$  and CO<sub>2</sub> production, which indicate the level of microbial activity of the system and can be used to monitor the process.

$$P = Y_{CO_2} * \eta_{VS} \tag{7}$$

where:

*P* is CO<sub>2</sub> production (kg/kg<sub>SV0</sub>)  $Y_{CO_2}$  is the yield (kgCO2/kg<sub>SV0</sub>)  $\eta_{SV}$  organic matter loss efficiency (kg<sub>degradedVS</sub>/kg<sub>SVo</sub>)

Table 3. Seed germination bioassay for toxicity evaluation. Experiments were analyzed by triplicate (Table S4,<br/>Monroy et al., 2020).



Fig. 4. CO<sub>2</sub> produced during composting of mixtures in the bench scale reactor. a) Integrated data from fig. 3b. b) CO<sub>2</sub> yield (eq 7) - -95% confidence limits, Y= 1.21 g CO<sub>2</sub>/gSV<sub>removed</sub> R<sup>2</sup> = 0.96.

## 3.4 Toxicity evaluation of the compost

The resulting compost of each of the three runs were tested for toxicity with the germination index for its use as a biosolid. Table 4 shows that compost from run 1 (with the highest sawdust content) gave the lowest GI=83% which according to NADF-020-AMBT-2011 is recommended to use in ecological agriculture or reforestation. The products from runs 2 and 3 (higher in digestate and lower in sawdust) which yielded better GI (95% and 94% respectively), are recommend as substrate in nurseries and soil substitute for pots.

## Conclusions

With this composting process the full utilization of the organic fraction of urban solid wastes (OFUSW) can be accomplished. By a two stage anaerobic digestion process the solids are converted into biogas and the solid residue called digestate is composted into a biosolid with C/N =15.2, 80% moisture content, with good odor and high germination index (GI>94%) The operating conditions resulting from this study are: initial mixture composition: 62% digestate, 28%

day and night. With the combined anaerobic and composting processes an 88% VS reduction of the OFUSW can be obtained while the remaining solids can be applied as soil improver. Acknowledgements

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fresh OFUSW, and 10% sawdust; an aeration rate of 0.5 L min^{-1} kg\_{VSo^{-1}} and 12 days composting time

to obtain a 32% mass reduction. The maximum

temperature reached was 50 °C oscillating between

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