

Modelling vinasse/cotton waste ratio incubation for optimum composting

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Abstract

A central composite experimental design was used to investigate the influence of incubation conditions (vinasse added and operation time) for vinasse/cotton waste mixtures on the properties of products obtained (pH, electrical conductivity, organic matter, Kjeldahl-N, C/N ratio, biodegradability, Kjeldahl-N losses and germination index) in order to determine the best in-vessel composting conditions. The range of the independent variable measured was 0–80% for vinasse added and 1–50 days for operation time. A second-order polynomial model consisting of two independent process variables was found to accurately describe the vinasse/cotton waste incubation. The differences between the experimental values and those estimated by using the equations never exceeded 10% of the former. Obtaining products with acceptable chemical properties, high biodegradability and minimum N losses entails operating at medium operation time (20–35 days) and 20–30% of vinasse added.

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1. Introduction

Beet vinasses are beet molasses which are almost completely biochemically desugarized, distilled and sometimes concentrated. Vinasses are disposed of in ponds and lagoons where they become concentrated during the hot summer periods (June–September) [1]. Concentrated vinasses constitute an environmental problem due to two main factors: a high organic charge (BOD: 61,000–70,000 mg l⁻¹) and high salinity (EC: 250–300 dS m⁻¹) [2]. However, it constitutes a valuable resource as a fertiliser due to its organic matter content (350 g kg⁻¹ dry weight) and nutrient contents (N: 30 g kg⁻¹; K: 30 g kg⁻¹ dry weight). Furthermore, the direct application of vinasse has several shortcomings because of its high salinity, phosphorus content (P: 0.06 g kg⁻¹) and its dense liquid character (1.3 g cm⁻¹) [2]. An alternative to overcome these disadvantages and to recycle the vinasse is the co-composting with other solid waste.

Cotton waste (CW), a waste from the cotton production, could also be recycled as fertiliser due to its organic matter and nutrient contents. The incorporation of cotton waste directly or under composting into agricultural land

is a common practice [3–5]. Cotton waste can be used for co-composting with vinasse.

Composting is the biological decomposition and stabilisation of the biodegradable component under controlled conditions. It is an aerobic process (requires oxygen) and is carried out by micro-organisms which metabolise organic waste as an energy source. The effectiveness of the process is dependent upon the environmental conditions present within the composting system [6].

The success of the composting process depends on several basic conditions including: the moisture content (50–60%) [7,8], effectiveness of the aeration [6,9], the C/N ratio of the initial mixture (20–30) [7,10], to reach a temperature of 50–60 °C [11–15]. The composting process has to produce an environmentally stable product free of disease causing organisms, offensive odors, insects, and weed seeds.

The method of composting can impose some differences with respect to pile design, operations and costs. Although, composting can be achieved using a number of different processes, including windrow and aerated static pile techniques, in-vessel composting offers a number of potential advantages:

- Composting can be completed rapidly.
- Great space efficiency.

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- Can maintain a rapid decomposition process year-round regardless of external ambient conditions.
- Precise control of moisture, temperature and aeration.
- Produces a uniform and high quality organic material.

The main disadvantages of in-vessel methods are their high capital costs and the requirement for intensive and skilful management [6].

When co-composting systems are carried out, another parameter is also important: the correct proportion of each material. This work aims to elucidate the optimum vinasse/cotton waste ratio in order to determine the best in-vessel composting conditions (operation time and vinasse added) to help a correct composting design.

2. Materials and methods

Relevant characteristics of the raw materials and initial mixtures are reported in Table 1. The experiments were carried out in plastic bins (35 cm × 20 cm × 30 cm). The walls and bottom of each bin was perforated with 100 (1 cm²) holes to promote passive aeration. The liquid residue vinasse was mixed homogeneously with the solid residue cotton waste at different ratios 0%, 11%, 40%, 69% and 80% (V/CW, (w/w) wet weight), mixtures V0, V11, V40, V69 and V80, respectively. The mixtures were performed according to the experimental design until maximum vinasse absorption. The mixtures (ca. 5 kg) were placed in the bins and incubated in thermostatic chambers at 55 °C provided by a blower (701 h⁻¹) that delivered air continuously in the thermostatic chamber during the entire period of composting. A randomised complete block design with four replicates per mixture was used. Moisture content was established at 55%,

during the thermophilic phase. By mass balance, after moisture characterisation, the necessary water to maintain 55% of moisture was calculated. Normally, the agroindustrial residues have great variability in form and size distribution. Particle size distribution (weight percentages) was: >5 cm, 3%; 5–4 cm, 18%; 4–3 cm, 21%; 3–2 cm, 10% and <2 cm, 18%. This distribution was the same for all experiments in order to ensure aerobic conditions and the same conditions for mixtures. Vinasse absorbed in the cotton waste did not change the initial particle size.

Samples collected were dried (60 °C) and ground (0–25 mm). Moisture was determined by drying at 105 °C to constant weight. The mixtures were analysed for the following parameters: pH (1:5 (w/v)) using a pH electrode, total organic matter (OM) by loss on ignition (550 °C for 5 h) [16], total P (acid digest) using the ascorbic acid method [17], total K, Ca and Mg (acid digest) by atomic absorption spectrophotometry [17], Kjeldahl-N [17], NH₄⁺-N and NO₃⁻-N using the KCl extraction method [18] and germination index (Gi) was determined using seeds of *Lepidium sativum* L. [19]. Biodegradability, a parameter that relates initial and final content of compost organic matter during composting [6,20], was also calculated for each mixture. Biodegradability, K_b , was calculated using the following equation.

$$K_b = \frac{(OM_i - OM_f)100}{OM_i(100 - OM_f)} \quad (1)$$

where OM_f is the organic matter content (% , dry weight) at the end of the process and OM_i is the organic matter content (% , dry weight) at the beginning of the process.

The Kjeldahl-N losses (%) during the process have been calculated from the initial content of Kjeldahl-N, and the evolution of the organic matter, assuming the mineral

Table 1
Chemical characterisation^a of the raw materials and the initial mixtures

	V ^b		CW ^c -V0 ^d		V11		V40		V69		V80	
	AV ^e	S.D. ^f	AV	S.D.	AV	S.D.	AV	S.D.	AV	S.D.	AV	S.D.
pH	4.7	0.40	7.56	0.03	6.82	0.03	6.25	0.10	5.60	0.39	5.05	0.19
OM ^g (%)	27.0	1.41	68.60	7.85	63.50	4.01	52.30	7.07	39.60	0.76	36.10	0.47
N ^h (g kg ⁻¹)	25.0	1.30	1.32	0.10	1.56	0.04	1.85	0.02	2.00	0.39	2.39	0.47
NO ₃ -N (mg kg ⁻¹)	350	177.3	36	4.16	52.30	68.6	72.30	12.69	95.70	14.7	115.4	21.6
NH ₄ -N (mg kg ⁻¹)	90.0	9.35	13.90	2.10	43.00	2.07	66.00	9.13	76.00	14.39	79.00	8.46
C/N	6.0	0.60	28.90	0.08	22.60	2.67	15.71	2.20	11.00	1.09	8.40	0.63
P (g kg ⁻¹)	0.05	0.60	0.18	0.02	0.15	0.01	0.11	0.01	0.08	0.00	0.06	0.00
K (g kg ⁻¹)	27.0	1.40	1.18	0.16	1.42	0.20	1.77	0.18	2.08	0.19	2.23	0.25
Ca (g kg ⁻¹)	3.4	0.50	2.02	0.12	2.23	0.33	2.01	0.02	2.19	0.09	2.00	0.12
Mg (g kg ⁻¹)	5.0	0.60	0.28	0.01	0.35	0.03	0.52	0.01	0.58	0.03	0.61	0.07

^a Each value is the average of four samples, dry weight basis.

^b V: vinasse.

^c CW: cotton waste.

^d V0, V11, V40, V69, V80 are mixtures with 0, 11, 40, 69 and 80% of vinasse, respectively.

^e AV: average.

^f S.D.: standard deviation.

^g OM: organic matter.

^h N: Kjeldahl-N.

amount in each mixture is constant. N losses increased as the proportion of vinasse in the mixtures was increased.

3. Experimental design for the incubation process

In order to relate the dependent (pH, organic matter, Kjeldahl-N, C/N ratio, biodegradability, Kjeldahl-N losses and germination index) and independent (time and vinasse added) variables with the minimum possible number of experiments, a central composite factor design for two factors was used. This experimental design enabled the construction of second-order polynomials in the independent variables and the identification of statistical significance in the variables [21,22]. The total number of observations required for two independent variables (time and vinasse added) was calculated using the following equation:

$$N = 2^K + 2K + 1 \quad (2)$$

K in the equation is the number of independent variables was 2 and then, N was 9. The ranges used in these two variables were: 1–45 days for operation time (t) and 0–80% of vinasse added (v). The results were subjected to multiple linear regression as implemented in SAS system package. The values of the independent variables (t , v) were normalised from -1.414 to $+1.414$ using Eq. (3) in order to facilitate the comparison of the coefficients and visualisation of the individual independent variables on the response variable. This normalisation also results in more accurate estimates of the regression coefficients as it reduces interrelationships between linear and quadratic terms (Montgomery, 1991) [22].

$$X_n = \frac{X - \bar{X}}{(X_{\max} - X_{\min})/2} \quad (3)$$

where X is the absolute value of the independent variable concerned, \bar{X} the average value of the variable and X_{\max} and X_{\min} are its maximum and minimum values, respectively.

The independent variables used in the equations relating to both types of variable were those having a statistically significant coefficient (viz. those not exceeding a significance level of 0.05 in Student's t -test and having a 95% confidence interval excluding zero). The vinasse added and operation time used in the different experiments of the central composite experimental design were 0, 11, 40, 69 and 80% and 1, 7, 23, 38 and 45 days, respectively. In that form 0, 11, 40, 69 and 80% corresponding to -1.414 , -1 , 0 , $+1$, $+1.414$ for the independent variable " v " and 1, 7, 23, 38 and 45 days corresponding to -1.414 , -1 , 0 , $+1$, $+1.414$ for the independent variable " t ", respectively.

4. Results and discussion

Table 2 shows the normalised values of independent variables and its subsequent dependent variables of the products obtained in the incubation process. Each value is an average of four samples. The deviations for these parameters from their respective means were all $<5\%$.

The equations showed in Table 3 were obtained when the values of the independent variables were substituted for each dependent variable (Table 2) into the polynomial expressions used.

The response surfaces for each dependent variable was plotted (Figs. 1–4) in order to determine the optimum values of each independent variable.

The response surface represented in Fig. 1 shows the variations of pH in relation to the operation time and the amount of vinasse added. It is observed that pH is more influenced

Table 2

Values of the independent variables^a and the experimental results for each parameter measured during incubation process using the proposed experimental design

Normalised values of time and vinasse added		Actual values of time and vinasse added		pH	Kjeldahl-N (%)	OM ^b (%)	C/N	K_b ^c	Kjeldahl-N losses (%)	Gi ^d
X_t ^e	X_v ^f	t ^g	V ^h							
-1	-1	7	11	7.54	1.55	58.30	20.90	19.64	8.16	11
+1	-1	11	11	9.08	1.35	35.10	14.44	68.91	40.20	73
-1	+1	7	69	8.08	1.83	36.40	11.05	12.71	15.34	0
-1	+1	7	69	9.55	1.57	30.20	10.69	34.01	36.56	37
+1.414	0	45	40	8.38	1.53	33.50	12.16	54.05	39.49	71
-1.414	0	1	40	6.25	1.85	52.30	15.71	0.00	0.00	0
0	+1.414	23	80	9.39	1.86	32.00	9.56	17.06	30.27	12
0	-1.414	23	0	8.55	1.39	52.40	20.94	49.61	27.92	53
0	0	23	40	9.12	1.60	42.10	14.62	33.68	28.02	39

^a Each value is the average of four samples, dry weight basis.

^b OM: organic matter.

^c K_b : biodegradability.

^d Gi: germination index.

^e X_t : normalised value of the operation time.

^f X_v : normalised value of the vinasse added.

^g t : operation time (days).

^h V : vinasse added (% (w/w)).

Table 3
Equations yielded for each dependent variable

Equation		r^2	Snedecor F
$\text{pH} = 9.16 + 0.754X_t - 0.82X_t^2 + 0.27X_v$	(4)	0.96	38.83
$\text{KN} = 1.61 - 1.14X_t + 1.45X_v - 0.05X_tX_v$	(5)	0.93	36.70
$\text{OM} = 41.36 - 7.00X_t - 6.96X_v + 4.25X_tX_v$	(6)	0.98	90.80
$\text{KB} = 32.18 + 18.38X_t - 10.98X_v - 6.99X_tX_v$	(7)	0.98	85.53
$\text{CN} = 14.45 - 1.48X_t - 3.71X_v + 1.52X_tX_v$	(8)	0.98	72.27
$\text{NL} = 29.04 + 13.64X_t - 4.42X_t^2 - 2.90X_tX_v$	(9)	0.99	99.14
$\text{Gi} = 32.89 + 24.93X_t - 13.12X_v - 6.25X_tX_v$	(10)	0.98	114.73

Where pH denotes pH; KN the Kjeldahl-N content (%); OM the organic matter content (%); CN the C/N ratio; KB the biodegradability (%); NL the N losses (%); and Gi the germination index. X_t and X_v denote the normalised value of the operation time and vinasse added, respectively. The differences between the experimental values and those estimated by using the previous equations never exceeded 10% of the former.

by the operation time than that by the vinasse added. In the response surface, a maximum of pH is observed at intermediate operation time (23 days). The values found at a longer operation time (45 days) suggest the use of this operation time to ensure a suitable final pH. During the maturation stage the pH should fall towards neutrality in a correct composting process [23].

Kjeldahl-N are more sensitive to changes in operation time than changes in vinasse added (Eq. (5)). Long operation times give rise to low Kjeldahl-N contents, then high vinasse added and short operation time should be used.

Organic matter evolution was similarly affected by operation time and vinasse added (Eq. (6)). An increase of the vinasse added produces a decrease in the organic matter of the initial mixtures, however, similar values of organic matter were observed for all the final mixtures. This fact can be

explained throughout the evolution of the biodegradability in the mixture (Fig. 2). In the mixtures with low contents in vinasse, greater organic matter losses and thus higher biodegradability are observed. To obtain high organic matter losses (high biodegradability), long operation time (38–45 days) and low vinasse addition (0–11%) are recommended. The optimum values of biodegradability and organic matter losses calculated are similar to values reported by other authors in agroindustrial residues [24–26].

In the C/N ratio, the statistical influence of the vinasse added is higher than the operation time (Eq. (8)). The optimum C/N ratio of the initial mixtures is between 25 and 35 [6,9,27,28], therefore, medium-to-low vinasse added (0–40%) was recommended. During incubation, the C/N ratio decreases in mixtures with low vinasse added. The mixtures with low contents in vinasse undergo greater organic

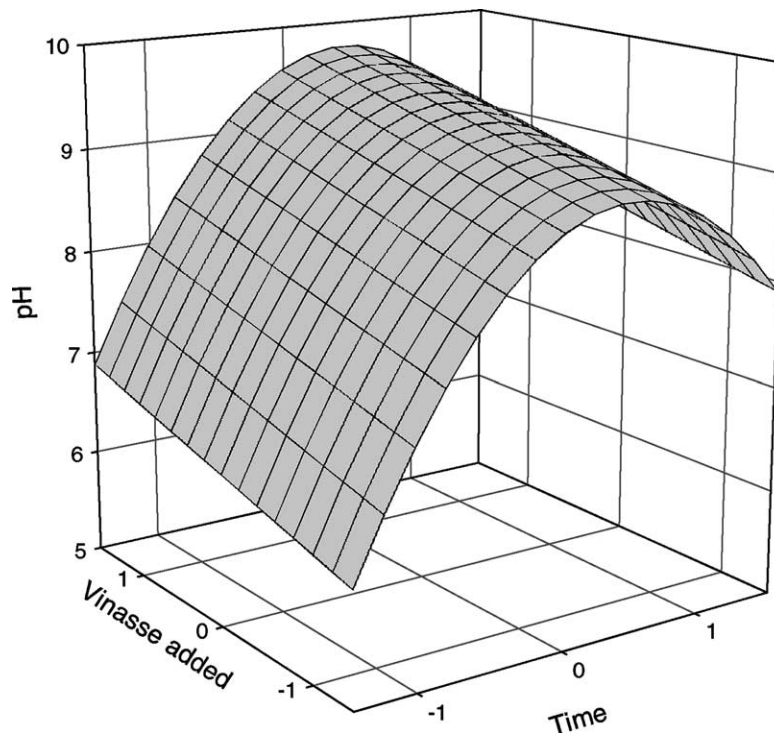


Fig. 1. Variation of pH as a function of operation time and vinasse added.

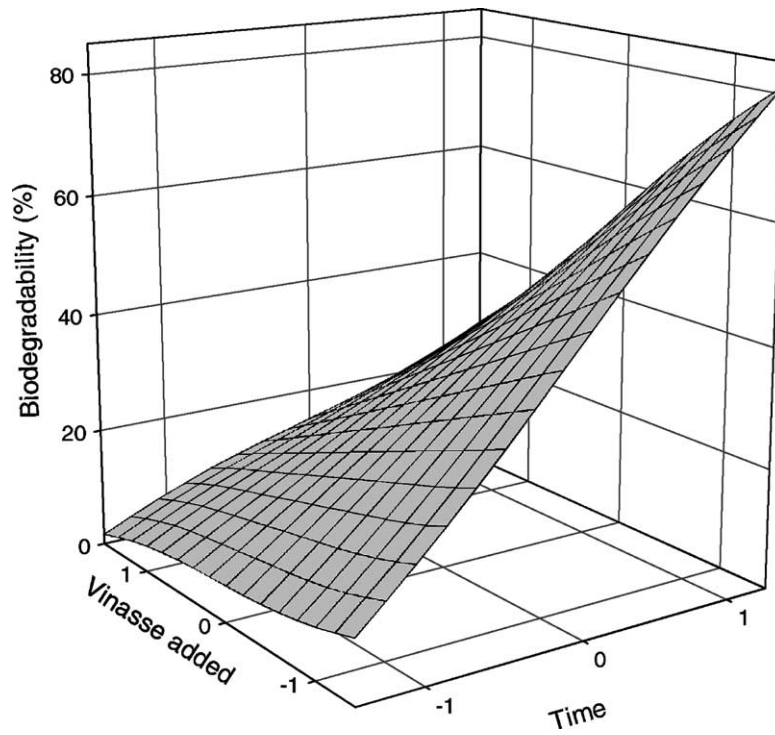


Fig. 2. Biodegradability variation as a function of operation time and vinasse added.

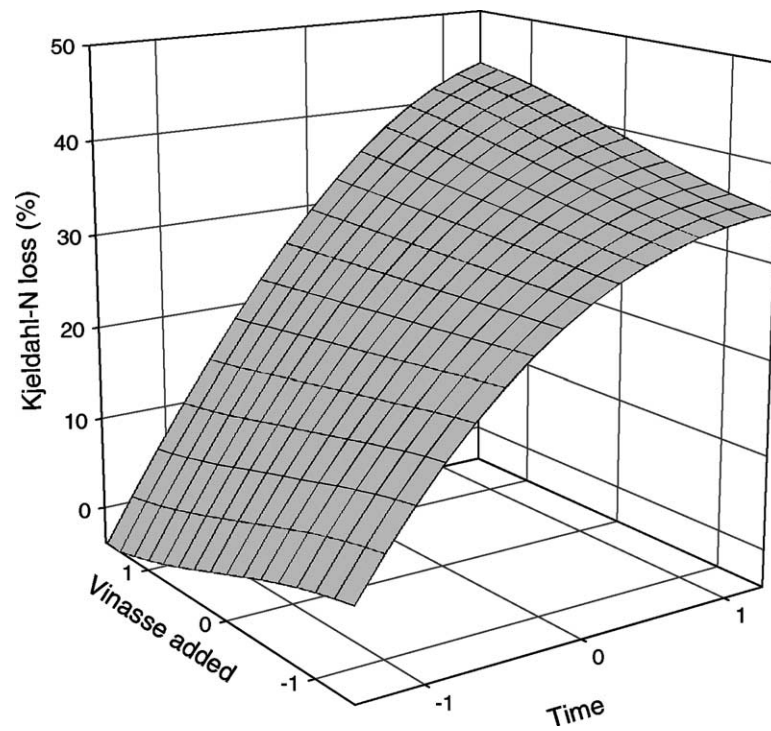


Fig. 3. Kjeldahl-N losses variation as a function of operation time and vinasse added.

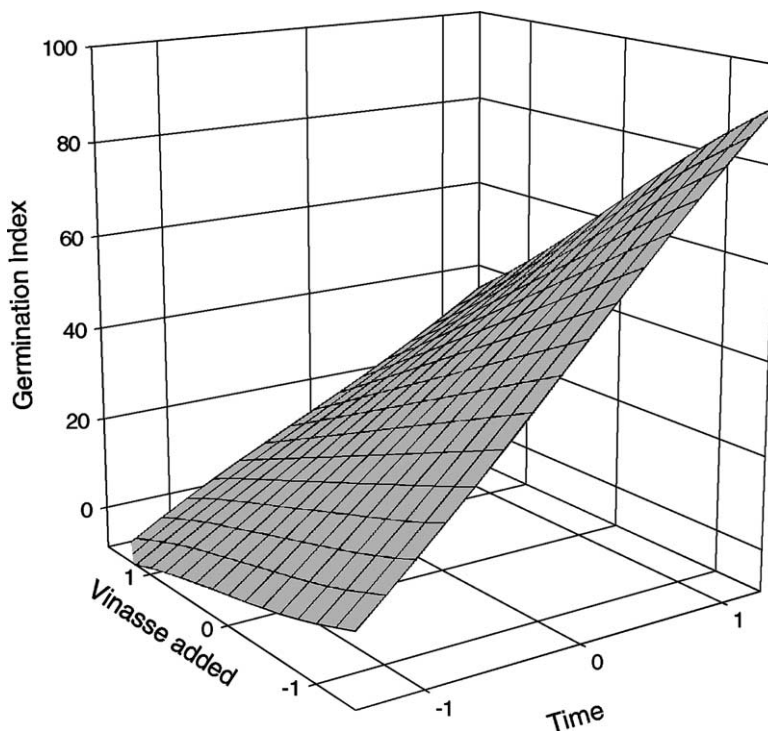


Fig. 4. Germination index variation as a function of operation time and vinasse added.

matter losses and lower N losses (Fig. 3) than those with high contents in vinasse. The mixtures with high vinasse contents suffer greater N losses than organic matter losses, this fact could explain the C/N ratio evolution that occurs in these mixtures. The typical C/N ratio of finished compost is between 10 and 15 [27,29]. To obtain suitable C/N final values, medium-to-low vinasse added (0–40%) should be used. The operation time could be medium-to-short (7–23 days) due to the scarce statistical influence of this independent variable.

According to Fig. 3 and the statistical influence of the quadratic term in the operation time in Eq. (9), the influence of the operation time is higher than that of the vinasse added. Therefore, medium-to-low vinasse added (11–40%) and medium-to-short operation time (7–23 days) may be used in order to ensure minimum Kjeldahl-N losses. The Kjeldahl-N losses found are similar to those found by other authors in similar agroindustrial residues [28,30,31].

Fig. 4 shows the variation of germination index (Gi) as a function of operation time and vinasse added. From

Table 4
Chemical characterisation^a of the final (day 45) mixtures

	V0 ^b		V11		V40		V69		V80	
	AV ^c	S.D. ^d	AV	S.D.	AV	S.D.	AV	S.D.	AV	S.D.
pH	8.20	0.53	8.37	1.14	8.38	1.72	8.53	0.14	8.63	1.59
OM ^e (%)	34.10	5.09	37.70	2.21	33.50	2.75	32.90	5.31	31.30	4.98
N ^f (mg kg ⁻¹)	1.06	0.13	1.16	0.10	1.53	0.53	1.31	0.09	1.33	0.05
NO ₃ -N (mg kg ⁻¹)	195.0	30.70	214.0	42.32	174.0	28.84	141.0	26.33	261.0	21.58
NH ₄ -N (mg kg ⁻¹)	3.30	0.01	2.42	0.00	7.9	0.16	8.4	0.95	19.4	1.15
C/N	17.80	2.77	16.13	1.69	12.16	2.20	14.00	2.80	13.10	0.34
P (g kg ⁻¹)	0.26	0.04	0.25	0.00	0.15	0.01	0.11	0.02	0.09	0.01
K (g kg ⁻¹)	1.45	0.00	1.73	0.19	2.03	0.05	2.39	0.19	2.53	0.50
Ca (g kg ⁻¹)	4.08	0.63	4.68	0.01	3.65	0.21	3.67	0.68	3.07	0.58
Mg (g kg ⁻¹)	0.53	0.10	0.61	0.07	0.70	0.02	0.75	0.03	0.86	0.16

^a Each value is the average of four samples, dry weight basis.

^b V0, V11, V40, V69, V80 are mixtures with 0, 11, 40, 69 and 80% of vinasse, respectively.

^c AV: average.

^d S.D.: standard deviation.

^e OM: organic matter.

^f N: Kjeldahl-N.

Eq. (10), it can be deduced that the positive statistical influence of the operation time is higher than the negative statistical influence of the vinasse added. If a high (G_i) is desired, a medium-to-high operation time (38–45 days) and medium-to-low vinasse added (11–40%) should be used. For low vinasse added mixtures, intermediate operation time (23 days) was enough to ensure a suitable ($G_i > 60$) [19].

In all cases, the content of $\text{NH}_4^+\text{-N}$ in the final products (Table 4) was lower than the value advised for mature compost ($<40 \text{ mg kg}^{-1}$) [32,33].

The initial (day 0) and final (day 45) nutrients content in mixtures are shown in Tables 1 and 4, respectively. As the added vinasse increases, higher values of K, Ca and Mg content were observed. Nevertheless, a decrease in P content was also observed. This fact could limit vinasse addition. The optimum range of C/P ratio is 75–150 [34,35]. Increases in nutrient contents are observed at the end of composting, due to the decrease in organic matter.

Due to economic reasons, medium operation time (23 days) is recommended. Among the products obtained at medium operation time, the corresponding [11% vinasse added, 23 days] point in the experimental design is the one which represents the highest biodegradability/nitrogen loss ratio values and hence could be the most suitable for its subsequent in-vessel composting.

5. Conclusions

The results of the experiments indicated that the incubation process of vinasse and cotton waste is technically feasible in in-vessel systems and could be considered as an ecological way to recycle these wastes.

Results of the incubation experiment showed that the product with acceptable chemical properties entails operating at medium operation time (≈ 23 days) and medium-to-low vinasse percentages (10–40%). Moderate amount of vinasse ($\approx 30\%$) would be the best compromise to compost this waste with cotton waste.

The chemical values found for the products obtained from vinasse/cotton waste are similar (same magnitude order) to other agricultural residues.

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