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Thermal analysis of growing media obtained from mixtures of paper mill waste materials and sewage sludge

A. Méndez · S. Barriga · F. Guerrero · G. Gascó

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Abstract This study deals with thermal analysis of growing media obtained from mixtures of paper will waste materials (one de-inking paper sludge, HP, and one reject from paper mill producing paper from virgin wood, RT) with sewage sludge. For the growing media formulation, one sewage sludge (L) was mixed with both paper mill waste materials at 10, 20, and 30% in volume. An incubation experiment was designed in order to study their carbon mineralization. Addition of sewage sludge significantly increases the carbon mineralization of growing media based on RT. In case of HP, carbon mineralization increases after addition of sewage sludge in 30% (HP + 30L treatment). Thermogravimetric analysis (TG and DTG) of growing media was performed before and after incubation experiment in order to study the organic matter transformation. The higher the carbon mineralization, the greater the difference between TG curves of samples before and after the incubation. The WL_3/WL_2 ratio increases after incubation of samples as a result of organic matter stabilisation. For HP growing media, the highest value corresponds to HP + 30L whereas HP + 20L, and HP + 10L show similar values. In case of RT, the WL_3/WL_2 index shows a progressive increase with sewage sludge content.

A. Méndez (✉)
Departamento de Ingeniería de Materiales,
E.T.S.I. Minas, Universidad Politécnica de Madrid,
C/Ríos Rosas n°21, 28003 Madrid, Spain
e-mail: anamaria.mendez@upm.es

S. Barriga · F. Guerrero · G. Gascó
Departamento de Edafología, E.T.S.I. Agrónomos,
Universidad Politécnica de Madrid, Ciudad Universitaria,
28004 Madrid, Spain

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Introduction

Peat or natural soils are commonly used in the production of growing media for ornamental plants, leading to important environmental problems. Different studies have been carried out in order to fully or partially replace these natural materials by organic-rich wastes such as sewage sludges or pine bark [1–4].

Paper industry generates large quantities of organic wastes with high cellulose content [5]. Some of them could be used as soil amendment due to their high content in organic matter. For example, deinking sludge amendments improve or restore soil fertility and biological functioning [6, 7] increasing yield and plant growth and improving soil properties such as organic matter content, water holding capacity or cation exchange capacity [8]. So, deinking sludges provide materials serving as a long-term source of organic matter [7]. However, since paper wastes are poor in phosphorus and nitrogen, supplemental sources of both elements must be added to facilitate the growth of woody species [9]. Moreover, sewage sludges contain significant amounts of N and P, making them of interest as soil amendments and in the growing media production [2, 10].

This study deals with the use of paper mill waste materials and sewage sludge mixtures in the growing media formulation. One incubation experiment was designed to study carbon mineralization of samples during 21 days and thermogravimetric analysis of growing media was performed before and after incubation process. Characterization of growing media was completed by physical and chemical analysis.

Experimental

Treatments

The growing media tested were obtained from one de-inking paper sludge (HP) and one reject from paper mill producing paper from virgin wood (RT) mixed with increasingly amounts of sewage sludge (L) at 0, 10, 20, and 30% in volume leading to HP, HP + 10L, HP + 20L, and HP + 30L treatments, respectively for HP and RT, RT + 10L, RT + 20L, and RT + 30L for RT.

Chemical properties

Raw waste materials (HP, RT, and L) and growing media were air-dried, crushed and passed through a 2-mm sieve. pH and electrical conductivity (EC) were determined in a ratio sample: water 1:2.5 (m/v) using a Crison micro-pH 2000 and a Crison 222 conductivity meter for pH and EC determination, respectively [11, 12]. Cation exchange capacity (CEC) was determined with $NH_4OAc/HOAc$ pH 7.0 [13]. Total organic matter (TOM) was measured by the dry combustion method at 540 °C [14]. N content was analysed by the Kjeldahl method [15] with a Büchi 435 digestor. Metal content was determined using a Perkin Elmer 2280 atomic absorption spectrophotometer after sample extraction by digestion with 3:1 (v/v) concentrated HCl/HNO_3 following 3,051a method [16].

Physical characteristics

The hydrophysical characteristics of growing media were determined using the method described by De Boodt and Verdonk [17] and Bunt [18] for measuring the water desorption curve of samples. According to this method, porosity_0 (% vol.) was the total pore space determined at 0-cm water suction; airspace_{10} (% vol.) was the difference in volume between porosity and the moisture content at 10-cm suction; and $\text{microporosity}_{100}$ was the moisture content (% vol.) at 100-cm suction. Two intervals of available water (AW) commonly used for horticultural purposes were also determined [19]: $AW_{10–50}$ (% v/v) was related to the water released from the substrate when the suction increases from 10 to 50-cm and $AW_{50–100}$ (% vol.) was the water released from the substrate when the suction increases from 50 to 100-cm water tension. Finally, R was defined as the suction (cm) which the air volume is equal to water volume in the growing media. Optimal R values range from 10 to 30 [20].

C mineralization

The biological activity of different growing media was evaluated by carbon mineralization (cumulative CO_2 evolution) and total mineralization coefficient (TMC). 200 g of each growing media were put in a glass vessel and the CO_2 evolved by each samples was evaluated during 21 days at a temperature of 28 ± 2 °C. The decomposition rate was determined by passing CO_2 and NH_3 free air through the respiration vessels, trapping the evolving CO_2 in 50 mL of 1 M NaOH. Periodic titration of the CO_2 trapped was performed with 1 M HCl after $BaCl_2$ precipitation of carbonates. Triplicate CO_2 measurements of each growing media were taken periodically. Another three vessels without growing media were used as blanks for each measure of evolving CO_2 [5]. Total mineralization coefficient (TMC) was calculated according to Díez et al. [21] as follows:

$$\text{TMC}\ (\text{mgC--CO}_2/\text{gC}) = \sum \text{C} - \text{CO}_2\text{evolved}/\text{initial TOC}$$

where C–CO_2 evolved is expressed as mg C–CO_2/100 g soil and initial TOC is expressed as g C/100 g soil.

Thermal analysis

Thermogravimetric analysis (TG and DTG) of growing media before and after the incubation experiment were carried out in a thermobalance Labsys Setaram. About 30 mg of sample was heated at 15 °C min^{-1} until 700 °C in air atmosphere using a flow rate of 40 mL min^{-1}.

Thermogravimetric results were quantified as the weight loss of samples attributed to the main peaks: WL_1 from 25 to 150 °C; WL_2 from 200 to 375 °C and WL_3 from 375 to 600 °C. WL_1 was related with water release from sample whereas WL_2 and WL_3 correspond to weight loss associated to organic matter combustion (Worg = $WL_2 + WL_3$). In addition, weight loss between 200 and 375 °C (WL_2) can be attributed to combustion of carbohydrates such as cellulose and lignocellulosic substances whereas weight loss between 400 and 600 °C (WL_3) was related to combustion of more complex and condensed structures with higher molecular weight [22]. The contribution of WL_2 and WL_3 to the total organic matter content could be quantified by the ratios WL_2/Worg and WL_3/Worg, respectively. Finally, the ratio between WL_3 and WL_2 was previously established as the thermostability index representing the relative amount of the thermally stable organic matter fraction compared to the less stable one [23].

Results and discussion

Table 1 summarises main properties of raw materials used in the growing media formulation. Paper mill waste materials (HP and RT) show basic pH (7.93 and 8.57, respectively) whereas sewage sludge (L) shows slightly acid pH (6.88). Electrical conductivity (EC) and cation

Table 1 Main properties of raw materials

Raw material	pH	EC/ mS cm^{-1}	CEC/ $cmol_{(+)}Kg^{-1}$	TOM/%	N/%	C/N	Cu/ mg kg^{-1}	Ni/ mg kg^{-1}	Cd/ mg kg^{-1}	Zn/ mg kg^{-1}	Pb/ mg kg^{-1}	Cr/ mg kg^{-1}
HP	7.93	0.79	25.37	47.2	0.43	63.9	367	170	9.8	1,918	57	11.1
RT	8.57	0.78	19.10	88.7	0.10	516.0	_	3	24.8	nd	14	nd
L	6.88	5.81	64.90	51.6	3.21	9.3	225	30	1.6	785	114	32

Table 2 Composition of growing media from HP and RT

Growing media	Composition			
	Volume/%		Weight/%	
	PW	SL	PW	SL
HP	100	0	100	0
HP + 10L	90	10	83.1	16.9
HP + 20L	80	20	68.6	31.4
HP + 30L	70	30	56.0	44.0
RT	100	0	100	0
RT + 10L	90	10	71.1	28.9
RT + 20L	80	20	52.2	47.8
RT + 30L	70	30	38.9	61.1

exchange capacity (CEC) of HP and RT are significantly lower than those of L. Sewage sludge shows a nitrogen content of 3.21 wt% much higher than that of paper waste materials (0.10 wt% for RT and 0.43 wt% for HP). The higher content of total organic matter (TOM) and highest C/N ratio correspond to RT (88.7% and 516, respectively). Finally, metal content of three raw materials is below to the limit established for use in agriculture [24].

Compositions of different growing media (in wt% and vol%) obtained from mixtures of L with HP or RT are summarised in Table 2. The weight proportion of HP in the growing media is higher than that of RT due to higher bulk density of HP (Table 3) according to the elevated mineral content of de-inking paper sludges [5].

Physical properties are of great interest in the growing media formulation. Table 3 summarizes the more important. Bulk density increased with the addition of sewage sludge [2, 25] being the higher values for HP than for RT based growing media. Also, $porosity_0$ slightly decreased with the sewage sludge addition [3] according to density values, although, these values are over 85% the optimal value obtained previously for growing media [20]. $Airspace_{10}$ values are higher than optimal values, 20–30%, reported by Abad et al. [20] according to the low available water (AW) values and R values, except for HP + 30L that show an $airspace_{10}$ value of 37%. The total AW ($AW_{10–50}$ + $AW_{50–100}$) of HP + 30L was 19% being near to optimal value proposed by Abad et al. [26] for growing media. Indeed, R value of HP + 30L is over 10 ensuring a good aeration and moisture of the growing media. However, R values lower than 10, as obtained for the rest of substrates, indicate that they must be frequently irrigated. In summary, HP + 30L shows the better hydro-physical properties and these were within the recommended values for ornamental plants production.

Table 4 shows main chemical properties of growing media. The addition of sewage sludge, L, decreases the pH of the substrates from HP and especially from RT, being pH over 7.00 and suitable in all cases for growing media uses [25]. This result was related to the decrease observed in the substrates $CaCO_3$ content with addition of sewage sludge. The EC increases with the addition of L, especially for RT. In addition, CEC values so increases with the addition of L, especially for HP, and were optimal to ensure a good retention of nutrients. Addition of sewage sludge increased the TOM of HP based growing media (from 47.22 to 53.17 wt% for HP and HP + 30L, respectively) due to higher organic matter content of L (Table 1). So, the addition of L notably increased the total nitrogen content of these substrates leading to an important decrease in the C/N ratio reaching to optimal values. For example, Ingelmo et al. [3] obtained optimal *Cupressus sempervirens* growth with C/N ratios around 25 and Hernández-Apaolaza et al. [27] described optimal *Cupressus arizonica, Pinus Pinea*, and *Cupressus sempervirens* growth with C/N ratios ranged form 9.7 to 53.0. With respect to RT-based growing media, the addition of sewage sludge reduced the TOM significantly, but increased the total nitrogen content from 0.1% in RT to 0.91% in RT + 30 L leading to a significant decrease in the C/N ratio from 516 to 46, respectively and reaching optimal values described previously by Hernández-Apaolaza et al. [27]. Indeed, the reduction in C/N ratio for RT-based growing media could have a positive effect in substrate respiration as shown below. Nevertheless, Inbar et al. [28] have cautioned that the C/N ratio of organic wastes is only one technique by which maturity should be gauged and other factors such as inorganic and organic content and degree of decomposition are so important in growing media formulation.

Experimental results obtained during incubation process of growing media show that all treatments presented the same mineralization pattern which was satisfactorily described by means of a power model CO_2–C=C=a · t^b [29,

Table 3 Main physical characteristics of growing media

Growing media	Bulk density/g L^{-1}	Porosity$_0$/%v/v	Airspace$_{10}$/%v/v	$AW_{10–50}$/%v/v	$AW_{50–100}$/%v/v	Microporosity$_{00}$/%v/v	R/cm	pH	EC/dS cm^{-1}	$CaCO_3$ eqv/%	CEC/cmol$_{(c)}$ Kg^{-1}	TOM/%	N/%	C/N
HP	210	89	61	6	2	21	<10	7.93	0.79	24.44	25.37	47.22	0.43	64
HP + 10L	260	89	56	7	2	24	<10	7.84	1.83	22.50	28.77	48.97	1.32	22
HP + 20L	300	87	51	8	2	25	<10	7.64	2.28	17.00	29.44	51.11	1.72	17
HP + 30L	352	85	37	14	5	30	25	7.55	2.90	16.30	37.67	53.17	1.87	16
RT	131	92	53	8	2	29	<10	8.88	0.78	6.12	19.10	88.74	0.10	516
RT + 10L	160	91	54	8	1	28	<10	7.82	1.94	5.60	20.16	80.76	0.39	120
RT + 20L	196	90	53	10	3	24	<10	7.23	3.69	4.50	22.61	75.42	0.60	73
RT + 30L	213	89	52	11	3	23	<10	7.30	4.07	4.30	25.07	71.84	0.91	46

Table 4 Main chemical characteristics of growing media

Growing media	pH	EC/dS cm^{-1}	$CaCO_3$eqv/%	CEC/cmol$_{(c)}$ Kg^{-1}	TOM/%	N/%	C/N
HP	7.93	0.79	24.44	25.37	47.22	0.43	64
HP + 10L	7.84	1.83	22.50	28.77	48.97	1.32	22
HP + 20L	7.64	2.28	17.00	29.44	51.11	1.72	17
HP + 30L	7.55	2.90	16.30	37.67	53.17	1.87	16
RT	8.88	0.78	6.12	19.10	88.74	0.10	516
RT + 10L	7.82	1.94	5.60	20.16	80.76	0.39	120
RT + 20L	7.23	3.69	4.50	22.61	75.42	0.60	73
RT + 30L	7.30	4.07	4.30	25.07	71.84	0.91	46

30] (Table 5 and Fig. 1). In the case of HP-based growing media, the mineralization rate did not significantly increase with the addition of L, except for HP + 30 L. For this reason, the highest TMC was obtained for HP + 30L. For RT-based growing media, the percentage of CO_2 evolved significantly increased with the proportion of L, according to the mineralization rate values. This fact could be related to the decrease of the ratio C/N [31] (Table 3) due to the addition of L. The important differences in the respiration between HP- and RT-based growing media could be due to higher TOM content of RT.

Many tests have been used to study the maturity and stability of the organic matter during composting or incubation processes such as C/N ratio or concentration of water soluble organic carbon [27], humification index, total mineralization coefficient [29] and spectroscopic measurements including UV–Vis, FTIR, and NMR [31]. In the last years, thermal analysis has been proposed as an interesting technique in the characterization of organic matter stabilization processes [23, 32–35] that has the advantage of providing information on the chemical characteristics of the sample without any extraction step.

Figure 2 shows TG curves in air of growing media from HP before and after incubation process. It could be observed that weight loss during combustion is lower after incubation process, especially as the L content increases. It was showed that carbon mineralization of growing media from HP increased with the sewage sludge content (Fig. 1 and Table 5) and consequently, this is reflected in the TG curve by an increase in the combustion residue of the sample linked to a decrease in the weight associated to organic matter combustion [34]. Therefore, the higher the mineralization of organic matter the greater the difference between the TG curves before and after the incubation.

TG curves of RT-based growing media (Fig. 3), in comparison with HP (Fig. 2), showed more differences before and after incubation process as the L content increases. This result was according with the higher sewage sludge effect observed in carbon mineralization (Fig. 1). Growing media from RT without sewage sludge addition shows low carbon mineralization and similar TG curves were obtained before and after incubation. However, the addition of sewage sludge increased the carbon mineralization (Fig. 1 and Table 5) and leads to important differences between TG curves obtained before and after incubation.

Figure 4 shows DTG curves of growing media from HP before and after incubation process. In general, three areas could be observed. Fist peak between 25 and 150 °C was due to moisture released from samples. Then, from 200 to 600 °C combustion of organic matter was produced and two areas

Table 5 Parameter estimates for cumulative CO_2–C evolved (mg C kg^{-1} dry weight) for the different treatments

Growing media	$C = a \cdot t^{b}$	r	Mineralization rate	CO_2 evolved/ mg C–$CO_2$100 g^{-1}	TMC/mgC–CO_2gC^{-1}
HP	$C = 89.35 \cdot t^{0.70}$	0.99	$dC/dt = 62.65\ t^{-0.30}$	757	27.6
HP + 10L	$C = 95.48 \cdot t^{0.68}$	0.99	$dC/dt = 64.70\ t^{-0.32}$	804	28.2
HP + 20L	$C = 96.45 \cdot t^{0.70}$	0.99	$dC/dt = 67.67\ t^{-0.30}$	836	28.1
HP + 30L	$C = 130.98 \cdot t^{0.66}$	0.99	$dC/dt = 86.47\ t^{-0.34}$	1,011	32.7
RT	$C = 3.73 \cdot t^{1.60}$	0.98	$dC/dt = 5.98 * t^{0.60}$	300	5.8
RT + 10L	$C = 16.69 \cdot t^{1.51}$	0.98	$dC/dt = 25.22 * t^{0.51}$	1,085	23.1
RT + 20L	$C = 36.58 \cdot t^{1.36}$	0.99	$dC/dt = 49.75 * t^{0.36}$	1,758	40.1
RT + 30L	$C = 95.74 \cdot t^{1.07}$	0.99	$dC/dt = 102.44 * t^{0.07}$	2,203	52.7

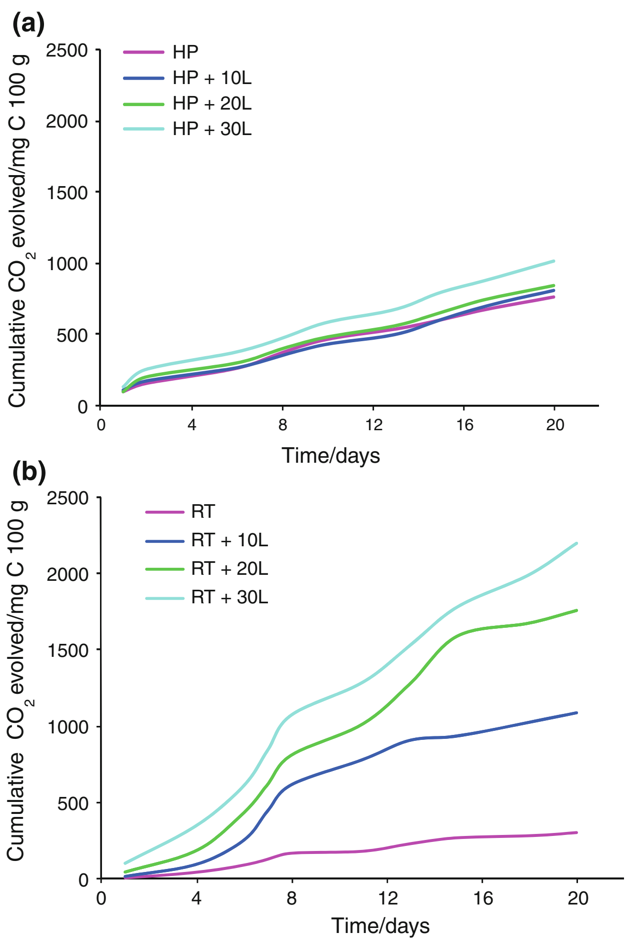

Fig. 1 Cumulative curve of CO_2 evolved during the incubation experiments

could be differenced. The first one between 200 and 375 °C could be attributed to combustion of carbohydrates such as cellulose and lignocellulosic substances whereas the second one between 400 and 600 °C was related to combustion of more aromatic structures with higher molecular weight [22]. Weight loss at temperatures higher than 600 °C indicates oxidation of refractory C [36] as well as the decomposition of minerals such as carbonates and clays from paper waste materials [5]. Generally, the peak related to cellulose combustion decreases after incubation of samples whereas the weight loss continues until higher temperatures. Similar effect was observed in the growing media from RT (Fig. 5). The lower carbon mineralization of RT without sewage sludge addition (Fig. 1b and Table 5) leads to small differences in their organic matter combustion and similar DTG curves were obtained for samples before and after the incubation experiment. For the rest of RT-based growing media it was observed that after the incubation, the organic matter that is oxidised at lower temperatures such as cellulose significantly decreases whereas weight loss continues until highest temperatures [35].

Analysis of weight losses produced during combustion of HP- and RT-based growing media at different temperatures is showed in Table 6. Addition of L to HP slightly decreases WL_2 corresponding to weight loss between 200 and 375 °C whereas WL_3 values corresponding to weight loss between 400 and 600 °C were similar for all growing media from HP. Comparing growing media before and after incubation, WL_3 and specially WL_2 decreases due to stabilization of samples. As a result of this evolution, WL_2/Worg decreases whereas WL_3/Worg increases specially for HP + 30L that show the highest carbon mineralization of growing media from HP (Fig. 1a). The same trend was observed for RT-growing media. As sewage sludge addition increases highest is the reduction of WL_2/Worg and the increase in WL_3/Worg after incubation of samples. This evolution can be quantified using the WL_3/WL_2 ratio, named thermostability index that was previously identified as a reliable parameter for evaluating the level of stability of organic maturation in organic compost [23, 34, 36]. WL_3/WL_2 indicates the relative amount of the thermally more stable fraction of organic matter with respect to less stable one. Generally, addition of sewage sludge to HP and RT increases the WL_3/WL_2. After incubation of samples, the WL_3/WL_2 increases, especially the higher is the sewage sludge content. For HP-growing

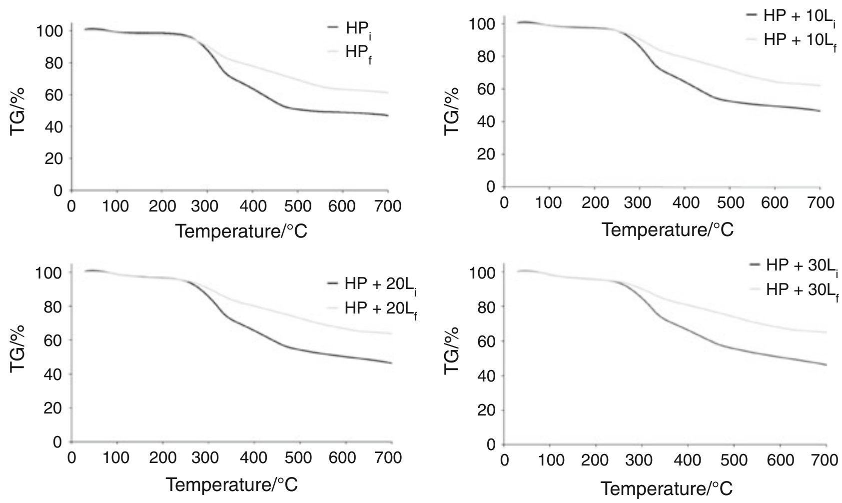

Fig. 2 TG curves of growing media from HP (*i* before the incubation process, *f* after the incubation process)

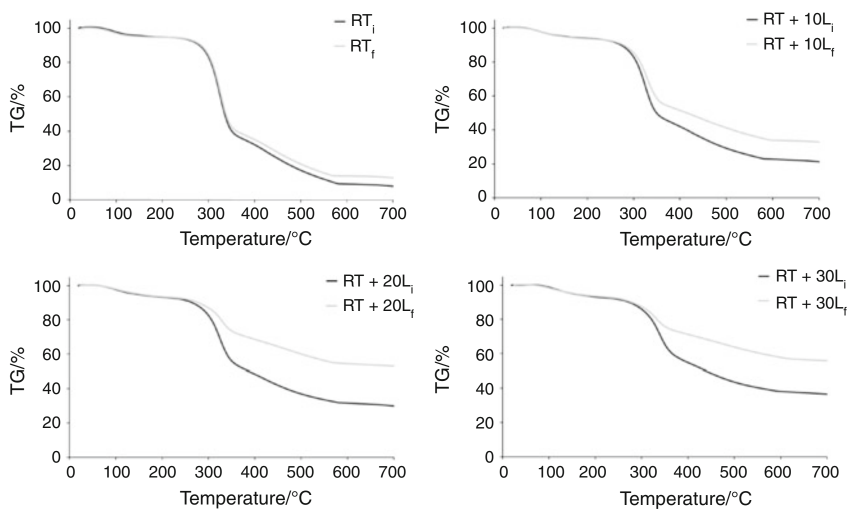

Fig. 3 TG curves of growing media from RT (*i* before the incubation process, *f* after the incubation process)

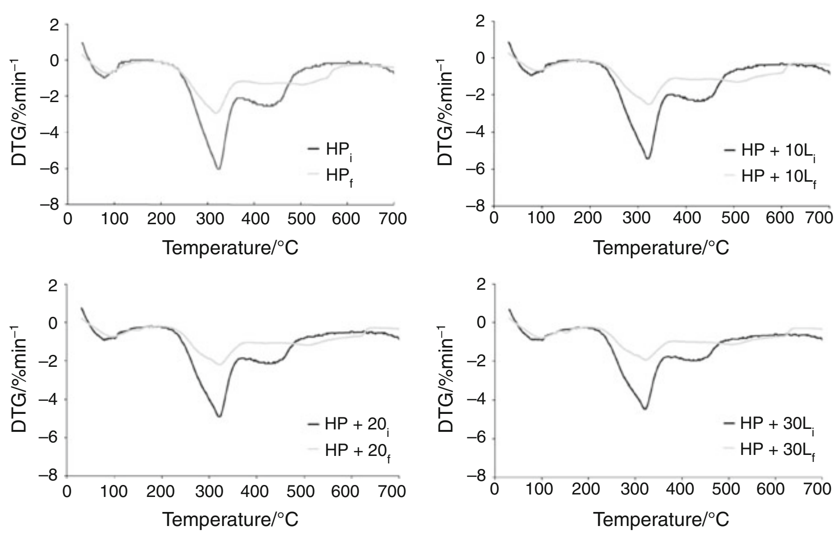

Fig. 4 DTG curves of growing media from HP (*i* before the incubation process, *f* after the incubation process)

media the highest value was obtained for HP + 30 L whereas similar values were obtained for HP + 20 L and HP + 10 L. In case of RT, the WL_3/WL_2 index shows a progressive increase with L content. These results were according to carbon mineralization of samples observed in Fig. 1 and TMC showed in Table 5.

Fig. 5 DTG curves of growing media from RT (*i* before the incubation process, *f* after the incubation process)

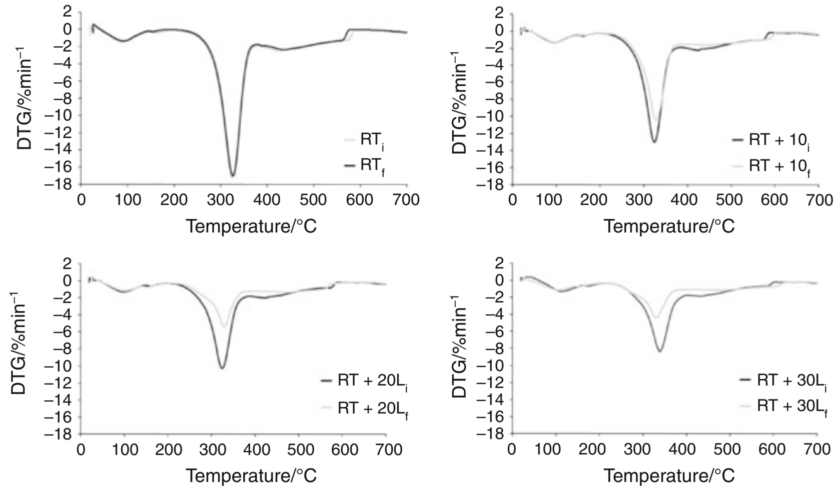

Table 6 Weight loss (wt%) and thermal parameters corresponding to the main peaks observed during combustion of growing media

Growing media	WL_1/25–150 °C	WL_2/200–375 °C	WL_3/375–600 °C	WL_2/Worg	WL_3/Worg	WL_3/WL_2
HP_i*	1.50	30.86	18.66	0.62	0.38	0.60
HP_f**	2.47	17.49	16.5	0.51	0.49	0.94
HP + $10L_i$	2.20	29.41	18.46	0.61	0.39	0.63
HP + $10L_f$	2.58	15.73	16.57	0.49	0.51	1.05
HP + $20L_i$	2.79	27.78	18.59	0.60	0.40	0.67
HP + $20L_f$	2.87	14.57	15.23	0.49	0.51	1.05
HP + $30L_i$	3.30	26.44	18.65	0.59	0.41	0.71
HP + $30L_f$	3.39	13.17	14.59	0.47	0.53	1.11
RT_i	4.38	59.42	26.11	0.69	0.31	0.44
RT_f	4.77	56.65	24.21	0.70	0.30	0.43
RT + $10L_i$	4.88	48.70	22.33	0.69	0.31	0.46
RT + $10L_f$	5.15	39.60	19.96	0.66	0.34	0.50
RT + $20L_i$	5.20	41.71	19.88	0.68	0.32	0.48
RT + $20L_f$	4.74	22.85	16.32	0.58	0.42	0.71
RT + $30L_i$	4.80	35.06	20.03	0.64	0.36	0.57
RT + $30L_f$	4.71	20.56	15.30	0.57	0.43	0.74

* *i* before the incubation experiment

** *f* after the incubation experiment

Conclusions

(1) It is possible to prepare growing media from adequate mixtures of paper mill waste materials and sewage sludge. The best hydrophysical properties were obtained mixing de-inking paper sludge with 30% in volume of sewage sludge. The rest of growing media should be frequently irrigated due to the low R values.

(2) Addition of sewage sludge significantly increases the carbon mineralization of growing media based on RT and in growing media from HP with 30% in volume of sewage sludge.

(3) Thermogravimetric analysis is an interesting tool to evaluate the organic matter transformation of growing media during incubation process. Experimental results from thermal analysis were according to carbon mineralization of samples. The higher the carbon mineralization, the greater the difference between the TG curves of samples before and after the incubation. Carbon mineralization of growing media from HP slowly increased with the sewage sludge content, except for HP + 30L, and this is reflected in the TG curve by a lightly growth in the combustion residue of the sample linked to a slowly decrease in the weight

associated to organic matter combustion. This effect is more noticeable in the RT-based growing media.

(4) Analysis of DTG curves of HP- and RT- based growing media show that generally, the peak related to cellulose combustion decreases after incubation of samples, whereas the weight loss continues until higher temperatures as a consequence of organic matter stabilization during the incubation process.

(5) After incubation of samples, the WL_3/WL_2 ratio increases as a result of organic matter stabilisation. For HP growing media, the highest value corresponds to HP + 30L whereas HP + 20L and HP + 10L show similar values. In case of RT, the WL_3/WL_2 index shows a progressive increase with sewage sludge content.

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