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# COMPOSTING OF SEIZED CIGARETTE TOBACCO AND INDUSTRIAL SEWAGE SLUDGE WITH SAWDUST OR GARDEN PRUNING

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#### Abstract

The composting of seized cigarette tobacco (SCT) and industrial sewage sludge (ISS) mixed with sawdust or garden pruning was evaluated using physical-chemical, phytotoxic and spectroscopic parameters. The temperatures reached peaks above  $55^{\circ}$ C in the five windrows and were sufficient to achieve the stability of the compounds. The moisture content remained mostly in the range of 50 to 70% indicated for efficient microbial activity during the decomposition process. The pH remained between 7.0 and 9.0, considered ideal for the stability of microorganisms that act in the various stages of composting. The loss of organic matter indicated an increase in the mineralization of the compost. After 180 days, the seed germination index (SGI) was above than 50% in the five windrows. The E<sub>2</sub>/E<sub>4</sub>, E<sub>4</sub>/E<sub>6</sub> (UV-Vis) and 1650/2930 cm<sup>-1</sup>, 1650/2850 cm<sup>-1</sup> and 1650/1711 cm<sup>-1</sup> (FTIR) ratios indicated the degradation of lignin, formation of aliphatic structures, oxygenated groups and aromatic carbon at different stages of maturation of the compounds. The windrow composting process was efficient to degrade different proportions of SCT and ISS, resulting in matured compounds.

Keywords: industrial sewage sludge; seized cigarette tobacco; solid residues.

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#### Introduction

The growing increase in the generation of organic solid waste by anthropic activities has resulted in great concern regarding its deposition in the environment (Zittel *et al.* 2018). A considerable part of the solid waste comes from seized cigarette tobacco (SCT) of illicit origin (Silva *et al.* 2014; Zittel *et al.* 2017) and the industrial sewage sludge (ISS) generated by various industrial processes (Silva *et al.* 2019).

Worldwide, 48.3 billion units cigarettes were seized in 2016, representing approximately 31.2 thousand tons of tobacco (Zittel *et al.* 2018). In Brazil in 2019, 235 million packs of illegal cigarettes were seized (BFR 2020). The generation of sludge in the food processing industry in 2015 was 21 million tons in European Union countries (Villar *et al.* 2016), 3 million tons in China (Meng *et al.* 2017) and 162 thousand tons in Brazil (Zittel *et al.* 2018).

Destruction of SCT by incineration (Silva *et al.* 2016) as the disposal of ISS in landfills or its direct application to the soil are pollution sources (Meng *et al.* 2017; Zhao *et al.* 2017). In addition, the improper disposal of these solid residues can result in the emission of methane gas, carbon dioxide and nitrogen compounds (Zittel *et al.* 2018).

A sustainable and low-cost strategy is the composting process (Silva *et al.* 2019; Zittel *et al.* 2018), as it significantly reduces the weight and volume of waste and results in safe products (Ayilara *et al.* 2020). In addition, the recycling of waste through the composting process leads to the rationalization of residual biomass, guaranteeing the supply of humidified organic material, minerals, improving moisture retention and microbial consortia beneficial to the soil and plants (De Corato 2020).

Composting can be summarized in three distinct phases (i.e., mesophilic, thermophilic and maturation phases), where each phase provides conditions of temperature, pH and availability of nutrients for the development of different microorganisms (Nafez *et al.* 2015). The addition of bulking agents (i.e., wheat straw, sawdust, rice bran, among others) is necessary to adjust the moisture content and the chemical composition of the mixture. They contain a high proportion of carbon/nitrogen, being able to absorb excess moisture from organic waste (i.e., sludge, animal manure, food waste), while adding structure to the mixture favoring the circulation of oxygen to the aerobic microorganisms (Palaniveloo *et al.* 2020). In addition, the duration of each phase depends on the initial composition of the residue, moisture content, quantity and composition of the microbial community.

The basic safety requirement for using the substrate resulting from composting is the degree of stability, which refers to the absence of animal and plant pathogens, as well as phytotoxic compounds present in precursor organic waste (Cesaro *et al.* 2019). Conventional control



methods (i.e., temperature, pH, moisture content, loss organic matter and seed germination index) associated to the spectroscopic analyses (i.e., UV/Vis, FTIR and FAAS) are excellent ways of monitoring decomposition, stabilization and material toxicity (Zittel *et al.* 2018).

The disposal of SCT and ISS requires an appropriate treatment to reduce the impact on the environment through sustainable practices of final disposal as solid waste. In this sense, the literature on sustainable recycling of SCT and ISS is still scarce.

The objective of this work was to investigate the treatment and recycling process of SCT and ISS by composting in windrows with manual turning aeration using physical-chemical, phytotoxicity and spectroscopic analyzes.

### **Material and methods**

The industrial sewage sludge (ISS) used in the experiments was obtained at the effluent treatment station from the aeration tank of a food processing plant. The treated effluent has as its initial composition washing water from the industrial processing system for processed foods and domestic wastewater. The seized cigarette tobacco (SCT), were obtained by seizure in police inspections, being cigarettes were removed from their boxes and crushed, then the tobacco was separated from the paper and the filter through a 6.0 x 6.0 mm sieve. The main properties of the feedstock are shown in Table 1.

Waste	Carbon/Nitrogen	Moisture (%±SD)	pH ± SD	Particle size (mm)
Garden pruning	23.72	5.31 ± 1.02	7.92 ± 0.02	>10
Seized cigarette tobacco	11.49	5.36 ± 0.81	5.34 ± 0.03	0.5 to 4.8
Industrial sewage sludge	6.01	91.14 ± 0.23	7.88 ± 0.1	Pasty
Sawdust	157.8	0.59 ± 0.05	5.64 ± 0.06	4.8 to10

Table 1. Properties of the feedstocks used in composting (dry weight).

The feedstocks SCT, ISS, Sawdust (Sa) and Garden Pruning (GP) were mixed in different proportions in order to obtain carbon/nitrogen ratios (10, 20 and 30) for windrows containing Sa (W1, W2, W3) and ratios (10 and 20) for windrows containing GP (W4 and W5), all subsequently submitted to composting for 180 days. The quantities of feedstocks used in each experiment are shown in Table 2.

Carbon and Nitrogen contents were obtained via elemental analysis using the TrucSpec CN Analyser equipment. The initial C/N ratios were calculated from the C and N content and considering the equation proposed by Caricasole *et al.* (2011).



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Feedstock	Windrow 1	Windrow 2	Windrow 3	Windrow 4	Windrow 5
Sawdust (kg)	47.5	47.5	95.0	0.0	0.0
Industrial sewage sludge (kg)	85.0	17.5	18.0	30.0	2.0
Seized cigarette tobacco (kg)	30.0	15.0	15.0	25.0	3.0
Garden pruning (kg)	0.0	0.0	0.0	30.0	36.0
Initial size base area of windrow (m <sup>2</sup> )	1.87	1.95	1.92	1.76	1.54
Initial moisture content (%)	75.2	71.2	74.2	70.5	75.0

 Table 2. Initial proportion of feedstock and of mixtures (wet weight)

For each windrow assembled, a specific amount of each residue was used, in order to initially obtain a specific C/N ratio. The windrows with a C/N = 10 ratio were intended to use materials with a higher nitrogen content and increase the volume of SCT and ISS treated. The windrows with a C/N  $\ge$  20 ratio followed what is recommended in the literature for composting with similar materials (Fialho *et al.* 2010).

The samples were collected in two sessions. The first sampling to verify the pH, was carried out from the collection of the material of seven randomly distributed points in the windrow (Fialho *et al.* 2010). The second sampling followed the solid residue sampling guidelines by ABNT-NBR: 10007 (ABNT 2004) for the remaining analysis. The samples collection and composting processes were observed for 180 days.

The windrows were assembled in conical shapes protected from weathering (closed shed). The system was operated with manual turning of windrow, being carried out e<zavery 15 days of composting from the beginning of the process up to 180 days.

### Temperature, Moisture content, pH

Temperature were verified daily throughout the whole period of composting with a digital thermometer. The moisture content was determined through gravimetric analysis. The pH value was also determined daily up to the 15<sup>th</sup> day, after that period it was verified each fortnight, with a digital pH meter.

### Organic Matter loss (OM Loss) and Seed Germination Index (SGI)

Ash content was determined using gravimetric. The decreases in organic matter were calculated using the initial (X<sub>1</sub>) and final (X<sub>2</sub>) Ash contents, with the following equation (Jara-Samaniego *et al.* 2017):

OM loss (%) = 
$$100 - 100 \times \frac{[X_1(100 - X_2]]}{[X_2(100 - X_1]]}$$



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Phytotoxicity was evaluated with watercress seeds (*Lepidium sativum*), being SGI was calculated according to the following equation:

 $SGI(\%) = \frac{[NGext \ x \ LRetx]}{[NGwater \ x \ LRwater]} \times 100$ 

Where: NGext is the number of seeds germinated in the aqueous extract. NGwater is the number of seeds germinated in the control, LRext is the length of the existing roots in the aqueous extract, LRwater is the length of the existing roots in the control (Mari *et al.* 2003).

## Metal determination

The total concentration of lead (Pb), cadmium (Cd), nickel (Ni), chrome (Cr), iron (Fe), manganese (Mn), copper (Cu) and zinc (Zn) was determined in triplicate in the 180-day compost obtained from the five windrow, they were also determined in triplicate for the SCT and ISS residues, individually. The digestion process was carried out following the method 3050B (US.EPA 1996). A flame atomic absorption spectrometer (FAAS), brand Varian, model 240 FS was employed for the determinations. The accuracy of the method was verified from the reference material analysis by the Canada Research National Council (marine sediment- ERMCE278), obtaining from 90% to 103% recovery of the metals.

### UV-VIS and FTIR spectroscopic analyses

For the UV-Vis analyses, 0.03 g sample (dry) was dissolved in 10 ml sodium bicarbonate solution at 0.05 mol/L. The wavelength determinations were carried out in scan mode in the range 200 to 800 nm and using the Varian Cary 50 equipment. After the determinations, the  $E_2/E_4$  ratios was calculated (270/407 nm) and  $E_4/E_6$  (465/665 nm) (Albrecht *et al.* 2011; Budziak *et al.* 2004).

The FTIR analyses were carried out with 0.1 g sample with 0.1 g potassium bromide. The readings were performed using IR Prestige-21 equipment in scan mode in the range 400 to 4000 cm<sup>-1</sup>. After obtaining the spectra, the ratios  $1650/2930 \text{ cm}^{-1}$ ,  $1650/2850 \text{ cm}^{-1}$  and  $1650/1711 \text{ cm}^{-1}$  was calculated (Castaldi *et al.* 2005).

### **Results and discussion**

# Temperature, Moisture content and pH

Figure 1 shows that during the period of 180 days in which the windrows were studied, the ambient temperature ranged from 9 °C to 32 °C. In the studied windrow, all the different characteristic phases of composting were observed: mesophilic, thermophilic and maturation phases. For W1, the mesophilic phase lasted 2 days, where the temperature in this period remained close to the environment temperature (20 °C). The thermophilic phase for this windrow



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lasted 10 days, when the temperature of W1 remained above 55 °C for 8 days. For W2, the mesophilic phase also lasted 2 days, with the first day remaining at 17 °C and the second day 29.7 °C. The thermophilic phase of this windrow lasted 6 days, being 5 days above 55 °C. For W3, the mesophilic phase lasted two days, the first with a temperature of 17 °C and the second with 29.5 °C. The thermophilic phase lasted 6 days, being 5 of them with a temperature above 55 °C for W4, the mesophilic phase lasted 24 hours, with a temperature of 15 °C. On the second day of composting, the temperature increased to thermophilic values, which lasted 16 days, being 11 days above 55 °C. For W5, the mesophilic phase lasted 24 hours with temperature of 21 °C. The following day the temperature reached 62.7 °C, starting the thermophilic phase, which remained for 9 days, being 8 days with temperatures above 55 °C.



Figure 1. Temperature evolution for the five windrows in 180 days of composting process.

The thermophilic phase is important for the decomposition of organic matter, destruction of pathogenic agents and potentially toxic compounds present in the precursor materials (Zittel *et al.* 2017). All windrows showed an increase in temperature at the beginning of the composting process, being the elimination of pathogens is related to temperatures ranging from 40-65 °C in one week (Chan *et al.* 2016). After 60 days, the temperature of all windrows reached values close



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to room temperature, which suggests low microbial activity and that the compounds reached stability (Jiang *et al.* 2015). Microorganisms that degrade organic matter act in composting at different temperature ranges. Bacteria are frequent at the beginning of the process and fungi during the entire process except at temperatures >60 °C. Actinomycetes are more present in the maturation phase, degrading resistant polymers together with fungi (Wei *et al.* 2019). Therefore, the temperatures obtained in all windrows revealed that composting can contribute to the degradation of several compounds present in the waste and it was possible to obtain the ideal conditions for the microbiological activity and the total sterilization of the compound.

Figure 2 shows the moisture variations observed for the studied composting systems.



Figure 2. Moisture evolution for the five windrows in 180 days of composting process.

Figure 2 showed to an increase in moisture content for all windrows at the initial composting process. It is known that a windrow can produce additional moisture to the system due to the biological oxidation of organic compounds and as a result of microbial activity (Anand & Apul 2014). After 15 days, a significant reduction in moisture content was observed and it remained in the interval considered ideal (50 to 70%) for most of the time (Zittel *et al.* 2017).



The moisture content is an important parameter to be controlled during composting, which is responsible for the transport of dissolved nutrients necessary for the physiology and biological activities of microorganisms (Guo *et al.* 2012). Therefore, the moisture content obtained in all windrows provided the ideal conditions for microbiological activity and subsequent stabilization of the compost.

Figure 3 shows the pH variations observed for the studied composting systems.



Figure 3. pH evolution for the five windrows in 180 days of composting process.

From the results presented in Figure 3, an increase in pH was observed for almost all windrows except for W4. This change in pH can be indicative of microbiological activity and degradation of the compounds in the mixtures. The increase in pH may be the result of decomposition of acidic organic compounds, formation of ammonia and mineralization of organic nitrogen (Nobelen *et al.* 2016). Acceptable pH ranges must be within tolerable levels (i.e., bacteria need a pH range of 6.0 to 7.5, fungi can tolerate a range of 5.5 to 8.0 and actinomycetes 5.0 to 9.0) for the microorganisms to perform their activities fully (Gómez-Brandón *et al.* 2008). All windrows under study, pH values can be considered adequate for good microbial activity. In addition, these pH values favor the composting process and the quality of the compost, since the organic nitrogen present in both sludge and tobacco can be converted by nitrification processes into elementary forms such as nitrite and nitrate that can used by plants (Zittel *et al.* 2017).



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### Organic Matter loss (OM Loss) and Seed Germination Index (SGI)

Figure 4 shows variations in OM Loss and SGI in percentage from the first to 180<sup>th</sup> day of composting.



Figure 4. OM Loss (%) and Seed germination index (%) of composts in the five windrows.

Figure 4A shows that there was an increase in OM Loss during the composting time for all the studied windrows. With the decomposition of organic matter, loss of mass in the form of carbon dioxide and water was observed. The increase in OM loss is indicative of mineralization during composting and consequent material stabilization (Caricasole *et al.* 2011). The OM is mineralized due to degradation of protein, cellulose, hemicellulose and lignin, which are used as C and N sources by the microorganisms (Fialho *et al.* 2010). The windrows that contained the largest amount of initial sludge were those that presented the highest of OM loss. This decrease can be attributed to the ISS mineralization facility, when compared to volume agents (Sa and GP), which have a more complex composition and less susceptible to attack by microorganisms (Nafez *et al.* 2015).

Figure 4B shows that in 180 days of composting all composts presented SGI over 50%, being considered stabilized and phytotoxicity free (Wang *et al.* 2014). However, a smaller amount of ISS used in the process required less time to reduce phytotoxicity, as seen in windrows W2, W3 and W5. In addition, a large increase in the volume of Sa and GP can decrease the efficiency of the process. Thus, the SGI appears as a sensitive parameter to evaluate toxicity and the degree of stabilization in process of composting (Sellami *et al.* 2008).



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The unstable compost presents phytotoxicity caused by different factors such as lack of oxygen, due to the intense microbial activity, the accumulation of toxic compounds (alcohols, phenolic compounds, low molecular weight organic acids and ammonia) and the presence of excess heavy metals and salts minerals (Zittel *et al.* 2017). According to the results obtained, the composting of SCT and ISS is a viable alternative for the treatment of these residues, in addition to obtaining a toxic free compound.

#### Metal determination

Table 3 shows the concentrations of metals related to the initial material SCT and ISS and the composts obtained after 180 days of treatment, in addition to guideline values for the use of organic composts in agriculture.

	SCT	ISS	W1	W2	W3	W4	W5	CCME	USDA
Fe*	1.73±0.01	4.48±0.012	1.40±0.02	10.24±0.02	9.48±0.01	18.84±0.03	8.97±0.02	-	-
Mn*	1.08±0.003	0.39±0.002	1.51±0.005	1.31±0.004	1.19±0.004	1.18±0.004	0.99±0.004	-	-
Pb	20.63±1.82	23.21±1.78	39.38±1.91	33.46±1.93	33.24±2.03	32.65±1.98	30.09±1.81	150	250
Cd	1.41±0.058	0.52±0.05	1.67±0.053	1.44±0.06	1.33±0.05	0.52±0.05	0.66±0.06	3.0	10
Ni	4.07±0.09	17.75±0.39	8.17±0.286	8.86±0.64	10.49±0.53	7.62±0.83	5.45±0.69	62	200
Cr	0.62±0.06	8.92±9.66	11.72±1.01	5.09±0.53	4.98±0.58	1.24±0.12	1.91±0.28	210	1000
Cu	11.33±0.85	10.87±0.86	14.61±1.26	12.25±1.06	14.56±1.05	12.29±1.08	9.61±0.76	400	1000
Zn	33.57±0.12	168.51±0.22	72.59±0.19	57.44±0.18	82.23±0.21	59.71±0.21	41.76±0.1	700	2500

Note: Fe\* and Mn\*: g/kg; other metals: mg/kg; USDA: United States Department of Agriculture; Canadian Council of Ministers of the Environment.

The results present in Table 3 showed that the concentration of metals Fe, Mn, Pb, Cd, Ni, Cr, Cu and Zn is below the values recommended by the international norms suggested by the United States Department of Agriculture and Canadian Council of Ministers of the Environment (CCME 2005; USDA 1980). Among the organic composts used, SCT and ISS are rich in metals. However, the proportion of residues employed in the initial mixtures made it possible to obtain final composts with metal concentrations below the limits set forth by international norms for the quality of the composting product.

### UV-VIS and FTIR spectroscopic analyses

After carrying out the UV-Vis analysis, it was possible to calculate the  $E_2/E_4$  and  $E_4/E_6$  ratios presented in Table 4.



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Table 4	$\mathbf{L}$ $\mathbf{E}_2/\mathbf{E}_4$ and $\mathbf{E}_2$	<sub>4/E6</sub> ratio ii	n the windrows as a	function of th	he composting time.	
	Patio	Timo	\\/1	\\/2	\\/2	\\//

Ratio	Time	W1	W2	W3	W4	W5
E <sub>2</sub> /E <sub>4</sub>	1 day	4.086	4.180	5.225	4.051	3.793
	180 days	4.602	4.478	3.446	3.820	3.573
$E_4/E_6$	1 day	3.023	9.281	3.042	3.167	2,544
	180 days	3.214	5.352	3.481	3.921	2.561

The results presented indicate that the windrows that decreased the  $E_2/E_4$  ratios during composting, degraded the lignin and formed porphyrinic structures, associated with a depolymerization and degradation of monomeric compounds by microorganisms (Abid *et al.* 2020). However, some windrows showed an increase in the same ratio, which suggests the consumption of porphyrinic structures originating in the thermophilic phase and formation of non-condensed aromatic structure linked to oxygenated functions (Budziak *et al.* 2004; Maia *et al.* 2012).

The  $E_4/E_6$  ratio indicates the degree of condensation and polymerization of aromatic structures during the composting process (Albrecht *et al.* 2011). In windrow W2, decrease was observed in the  $E_4/E_6$  ratio, which suggests the mineralization of carbohydrates and quinones, oxidation of phenolic compounds linked to metoxyl groups and/or aliphatic lateral chains in humic substances (Sellami *et al.* 2008). The results revealed an increase in the  $E_4/E_6$  ratio in W1, W3, W4 and W5. This characteristic suggests the presence of small sized organic molecules, lower degree of condensation of aromatic structures and larger number of functional groups as carboxyl (Fialho *et al.* 2010).

Table 5 showed the ratio between 1650/2930 cm<sup>-1</sup> (C-aromatic/C- aliphatic), 1650/2850 cm<sup>-1</sup> (C-aromatic/C-aliphatic) and 1650/1711 cm<sup>-1</sup> (C-aromatic/C-carboxyl) (Castaldi *et al.* 2005) in the windrows at the beginning and at the end of the study period.

Table 5. Ratio between in the windrow analyzed in different phases of the compost stabilization.							
Ratio	Time	W1	W2	W3	W4	W5	
1650 cm <sup>-1</sup> / 2930 cm <sup>-1</sup>	1 day	0.902	0.886	0.962	0.892	0.909	
	180 days	0.945	0.883	0.874	0.949	0.878	
1650 cm <sup>-1</sup> / 2850 cm <sup>-1</sup>	1 day	0.871	0.851	0.913	0.853	0.889	
	180 days	0.909	0.884	0.837	0.887	0.816	
1650 cm <sup>-1</sup> / 1711 cm <sup>-1</sup>	1 day	0.870	0.840	0.831	0.844	0.889	
	180 days	0.900	0.844	0.828	0.850	0.845	

Table 5. Ratio between in the windrow analyzed in different phases of the compost stabilization.



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In windrows W1 and W4, an increase in the ratios 1650/2930 cm<sup>-1</sup> and 1650/2850 cm<sup>-1</sup> was observed. Such characteristic agrees with the aliphatic carbon reduction and/or formation of humic polymer in the composts obtained from these windrow (Zittel *et al.* 2018). The reduction in the values of 1650/2930 cm<sup>-1</sup> and 1650/2850 cm<sup>-1</sup> ratios observed in W3 and W5 is due to the formation of aliphatic structures resulting from the microbial activity (Castaldi *et al.* 2005).

The increase in the levels of aromatic carbon can be seen due to the increase in the  $1650/1711 \text{ cm}^{-1}$  ratio in W1, W2 and W4, complementing the information obtained by the  $1650/2930 \text{ cm}^{-1}$  and  $1650/2850 \text{ cm}^{-1}$  ratio. The increase in the aromatic carbon amount is an indicative of the stabilization of the compost formed from this windrows (Varma *et al.* 2017).

The results obtained with these FTIR ratios agree with that observed in the results of the  $E_4/E_6$  ratios. In W1 and W4, for example, an increase in the  $E_4/E_6$  ratio was observed and indicated lower condensation of the aromatic structures and higher number of oxygenated groups. Such characteristic was confirmed, for example, by the increase in the 1650/1711 cm<sup>-1</sup> ratio, suggesting a higher number of carboxyl groups (oxygenated groups).

The spectroscopic results obtained from the UV-Vis and FTIR analyses demonstrated that W1 and W4 presented higher amount of aromatic carbon at the end of the composting when compared to W2, W3 and W5. However, the formation of aromatic carbon occurred in all windrow at different times. Since the formation of aromatic carbon is an indicative of the compost stabilization (Varma *et al.* 2017).

### Conclusions

Regarding the five treatments, the results showed that the period of thermophilic temperature contributed to the stability of the compounds, that the values of humidity and pH are considered favorable to the activity of the microorganisms. The loss of organic matter in the five compounds indicated an increase in maturity and mineralization of nutrients. The five treatments obtained SGI above 50% in 180 days of process. The metal concentration in all windrows was in accordance with the specification for use of organic compounds as a fertilizer. The results of UV-Vis and FTIR indicated degradation of lignin, formation of aliphatic structures linked to oxygenated groups, in addition to aromatic carbon in different stages of compound stabilization. Therefore, the composting process proved to be efficient in the degradation of SCT and ISS residues, and the final compost from the five processes showed characteristics that can contribute to the better agricultural conditioning of the soil and providing nutrients to the plants.



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