

## *Monitoring and characterization of compost obtained of sludge of ultra-processed food industry by conventional and spectroscopic analyses*

### **Monitoramento e caracterização de composto obtido de lodo de indústria de alimentos ultra processados por análises convencionais e espectroscópicas**

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#### **Abstract**

Several studies of potentially toxic residues have already been documented in the literature. However, studies focused on treatment of sludge of effluent treatment plant (ETP) from the ultra-processed food industry have been very little addressed. Sludge composting of the ultra-processed food industry in piles was monitored by conventional techniques (temperature, humidity, pH and seed germination index) and spectroscopic analyzes (UV-Vis, FTIR and FAAS). The experiments were carried out with two piles from different sludge combinations from the ultra-processed food industry associated with garden pruning or sawdust. The pile were prepared approximately 1m x 1m x 1m in size and were manually revolved once a week for up to 180 days. The piles presented thermophilic phase for approximately two weeks. After 90 days, the seed germination index (SGI) reached values of 62.5 for pile 1 and 87.5% for pile 2. The spectroscopic analysis indicated the occurrence of degradation of aliphatic/polysaccharide structures and the increase of oxygenated functional groups. The concentration of the metallic ions: Ni, Cd, Cr, Pb, Cu, Mn and Zn in the compounds obtained from the two piles was lower than the values established for use as soil conditioner or organic fertilizer. The composting process in piles was efficient to degrade the sludge of the ultra-processed food industry, associated with garden pruning or sawing, resulting in stabilized compounds.

**Keywords:** Ultra-processed foods. Metallic ions. Organic fertilize.

#### **Resumo**

Vários estudos de resíduos potencialmente tóxicos já foram documentados na literatura. No entanto, estudos voltados para o tratamento do lodo de efluentes de estações de tratamento de efluentes (ETP) da indústria de alimentos ultra processados têm sido pouco abordados. A compostagem de lodo da indústria de alimentos ultra processados em pilhas foi monitorada por técnicas convencionais (temperatura, umidade, pH e índice de germinação de sementes) e análises espectroscópicas (UV-Vis, FTIR e FAAS). Os experimentos foram conduzidos com duas pilhas de diferentes combinações de lodo da indústria alimentícia ultra processada, associada à poda de jardim ou serragem. As pilhas foram preparadas com aproximadamente 1m x 1m x 1m de tamanho e foram revolvidas manualmente uma vez por semana por até 180 dias. As pilhas apresentaram fase termofílica por aproximadamente duas semanas. Após 90 dias, o índice de germinação de sementes (IGS) atingiu 62,5 para a pilha 1 e 87,5% para a pilha 2. A análise espectroscópica indicou a ocorrência de degradação de estruturas alifáticas/polissacarídicas e o aumento de grupos funcionais oxigenados. A concentração dos íons metálicos Ni, Cd, Cr, Pb, Cu, Mn e Zn nos compostos obtidos nas duas pilhas foi menor que os valores estabelecidos para uso como condicionador de solo ou adubo orgânico. O processo de compostagem em pilhas foi eficiente para degradar o lodo da indústria de alimentos ultra processados, associado à poda de jardim ou serragem, resultando em compostos estabilizados.

**Palavras-chave:** Alimentos ultra processados. Íons metálicos. Fertilizante orgânico.

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## Introduction

Ultra-processed foods are products ready for consumption, produced from substances extracted from food or synthesized from synthetic organic materials (COSTA LOUZADA *et al.*, 2015). For the production of this type food, the industry uses high amounts of water daily, producing large volumes of effluent and sludge as the final reject of the process (AWASTHI *et al.*, 2016; VAN DEN HENDE *et al.*, 2016). The sludge from effluent treatment plants (ETP) in food industries has different characteristics, including contaminants from raw materials processed, chemicals and waste from technological additives used in their operations (QASIM; MANE, 2013).

However, the direct disposal of this material in the environment without proper treatment represents a problem for the society, increasing the volume of landfills, contaminating the soil and bodies of water, emitting greenhouse gases, among others (AWASTHI *et al.*, 2016; WANG *et al.*, 2016). The decomposition by composting has already been studied using kitchen-waste (YANG *et al.*, 2015), animal feces (FIALHO *et al.*, 2010; SALEEM *et al.*, 2018; XU; LI, 2017), sludge from breweries (STOCKS; BARKER; GUY, 2002) and urban ETP sludge (DZULKURNAIN *et al.*, 2017; KUMAR AWASTHI *et al.*, 2017; MENG *et al.*, 2016). However, research focused on the treatment of ETP sludge from the ultra-processed food industry is scarce.

Composting is a technology capable of obtaining a stabilized, nutrient-rich compound after the treatment of potentially toxic materials, which can benefit agricultural practices and reduce inorganic fertilizer applications (FIALHO *et al.*, 2010; HE *et al.*, 2011). Composting processes are usually characterized by the decomposition of materials that act as nitrogen sources (domestic organic waste) together with structural materials (plant pruning and sawdust) as a carbon source (MORETTI *et al.*, 2016). Several criteria and parameters were proposed and adopted to test the maturity and stability of the compound (BERNAL; ALBURQUERQUE; MORAL, 2009). Conventional controls (temperature, moisture, pH, C/N and germination index) and spectroscopic analyses (Uv/Vis, FTIR and FAAS) are efficient ways to monitor the stabilization and toxicity of composted material (GUO *et al.*, 2012; MCWHIRT *et al.*, 2012; USMAN *et al.*, 2012).

The stabilized or matured material must have phytotoxicity index above 50%, pH close to neutrality and

reduction of C/N ratio (CHIKAE *et al.*, 2006; SHAFFER, 2010; TIQUIA *et al.*, 1997). In addition, the increase in absorptions between specific wavelengths are proportional to the number of condensed rings. The decrease in the ratio between absorption intensities of 250/365 nm indicates the degradation of simpler molecules. The wavelength ratio 465/665 nm is inversely proportional to the degree of condensation of the unsaturated structures, so that a high ratio of this index indicates a low degree of condensation of carbon-carbon unsaturation and the presence of various olefinic structures (CAMPOS; RESSETI; ZITTEL, 2014). Thus, it is expected that for a stabilized compound, the values of the 250/365 nm ratio decrease and the values of the 465/665 nm ratio increase.

The purpose of this study was to evaluate the efficiency of the composting process, using ETP sludge from the ultra-processed food industry with different structuring materials, by employing conventional and spectroscopic techniques.

## Materials and Methods

### Experiments

Two piles (1 m wide, 1 m long and 1 m high) were mounted. The piles were assembled containing the following materials: Garden Pruning (GP), Sawdust (Sa) and ETP sludge from the ultra-processed food industry. The distribution of the residues in the piles and the characteristics of the materials are presented in Table 1.

**Table 1** – Combination of waste and characteristics of the materials in the piles. (Mean  $\pm$  Sd; n3).

	GP	Sa	Sludge
Pile 1 (kg)	20	0	95
Pile 2 (kg)	0	43	106
pH	7.9	5.6	7.8
MC (%)	5.3	0.6	91.1
Cu (mg kg <sup>-1</sup> )	2.5 $\pm$ 0.4	7.1 $\pm$ 0.8	19.6 $\pm$ 1.0
Mn (mg kg <sup>-1</sup> )	1.5 $\pm$ 0.2	51.3 $\pm$ 6.4	246.4 $\pm$ 2.8
Ni (mg kg <sup>-1</sup> )	< LQ	< LQ	32.6 $\pm$ 3.3
Cd (mg kg <sup>-1</sup> )	< LQ	< LQ	3.3 $\pm$ 0.12
Pb (mg kg <sup>-1</sup> )	< LQ	< LQ	8.4 $\pm$ 1.3
Cr (mg kg <sup>-1</sup> )	< LQ	< LQ	7.9 $\pm$ 1.1
Zn (mg kg <sup>-1</sup> )	54.2 $\pm$ 6.6	17.2 $\pm$ 1.6	384.0 $\pm$ 15.2

GP: Garden Pruning; Sa: Sawdust; Moisture Content: MC; LQ: Limit Quantification.

Source: The authors.

The compost was prepared with two different types of organic waste mixtures according to the quantification presented in Table 1. The initial C/N ratios were calculated from the C and N contents and taking into account the equation proposed by Caricasole *et al.* (2011).

Those amounts were used to ensure C/N ratio of about 46 in pile 1 and 49 in pile 2. Bulking agents were mixed with the substrates to ensure suitable aeration. In pile 1, was added GP, while in pile 2 was added Sa. The piles were prepared in a shed to avoid excess moisture of rain and were manually revolved once a week until the 180 days. When the mixture obtained moisture below 40%, water was added during the revolving.

Samples were collected from seven random locations distributed on the inside and on the surface of the piles, being homogenized until approximately 1.0 kg of sample was obtained for each pile and time. Samples for analysis of C/N, seed germination index, metal ions, Uv-vis and FTIR were ground in Gral and standardized in 18 mesh sieve. The other analyzes were performed with *in - natura* samples. The collections and the composting process were followed up for 180 days. All analyzes were performed in triplicates for each composting time.

#### Moisture, Temperature, pH, C/N ratio

In order to control the moisture, readings of the mixtures in the piles were carried out fortnightly. A digital thermo hygrometer was used for this analysis. The temperature was verified daily in the pile with a mercury thermometer. The pH value was determined fortnightly, according proposed by Fialho *et al.* (2010). The elemental chemical analysis to determine the C/N ratio was a performed the elemental analyzer fisions CHNS-O apparatus.

#### Seed germination index (SGI)

To evaluate the phytotoxicity, 20 g of dry weight compost were extracted with 200 ml of distilled water stirred for 1 h and then centrifuged at 10.000 rpm. Then 5 ml of the aqueous extract was added in petri dishes containing 10 seeds of *Lepidium sativum* each. The sets were incubated in a dark environment at 25°C for 5 days. Control tests were performed, replacing the aqueous extract with distilled water. The SGI was calculated according to the equation proposed by Tiquia, Tam and Hodgkiss (1997).

$$SGI\% = \frac{\text{seed germination-root length of treatment}}{\text{seed germination-root length of control}} \quad (1)$$

#### Uv-Vis spectroscopy

For the Uv-Vis analyses, portions containing 0.02 g of the samples were dissolved in 10 ml of a 0.05 mol L<sup>-1</sup> sodium bicarbonate solution, then the absorption readings were carried out using the apparatus varian Cary 50 bio. Then, the ratios between the absorbance at 250 nm and 365 nm (E<sub>2</sub>/E<sub>3</sub>), 465 nm and 650 nm (E<sub>4</sub>/E<sub>6</sub>) and aromaticity (52.5 - 6.78 E<sub>2</sub>/E<sub>3</sub>) were determined (PEURAVUORI; PIHLAJA, 1997).

#### FTIR spectroscopy

For the FTIR analyses, pellets were prepared by homogenizing 1.0 mg sample with 100 mg of potassium bromide. Then they were inserted into an FTIR spectrometer, brand Shimadzu, model IR prestige 21, and the readings ranged from 400 to 4000 cm<sup>-1</sup> with spectral resolution of 4 cm<sup>-1</sup> (FIALHO *et al.*, 2010). Then the ratios of the transmittances from wavenumbers 1647/2928, 1647/1417 and 1647/1030 were determined.

#### Determination of metallic ions

The samples were submitted to digestion using the method 3050-B (USEPA, 1996). Approximately 0.5 g sample and 5 ml concentrated nitric acid were added. The samples were digested in digester block for 2 h at 95±5 °C.

Then, 1.5 ml of hydrogen peroxide 30% was added, and after another 1h of reaction a further 5 ml concentrated hydrochloric acid was added, followed by a further 1 h digestion. The extracts obtained transferred to 50 ml volumetric flasks and completed with ultrapure water. Reagent blank and triplicate of each sample were evaluated. Ni, Cd, Cr, Pb, Cu, Mn and Zn metallic ions were quantified. The readings were performed in a Varian model 240fs Flame Atomic Absorption Spectrometer (FAAS).

The accuracy of the method was obtained from the reference material of the Institute for Reference Materials and Measurements (IRMM) in Europe and marine sediments of the National Research Council of Canada (NRCC), recovering between 90 and 103%.

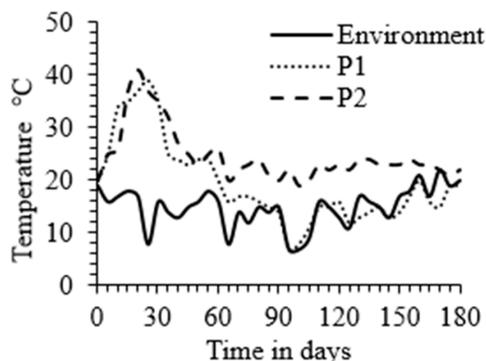
## Results and Discussion

#### Moisture, Temperature, pH, C/N ratio

Controlling physic-chemical parameters is important due to the influence on the conditions necessary for decomposition of the materials during composting. Initially, the moisture content for pile 1 was 50 % and for pile 2 it

was 55 %. For proper development of microbial activity to occur, it is necessary that the initial compound has between 40 and 70 % moisture (EPSTEIN, 2011; KHALID *et al.*, 2011). Thus, Figure 1 presents the temperature values obtained throughout the composting time.

**Figure 1** – Temperature variation at the in piles in 180 days



Source: The authors.

Pile 1 showed temperatures  $> 35^{\circ}\text{C}$  for a period of 15 days, with a peak of 2 days at  $39^{\circ}\text{C}$ . For pile 2, temperatures  $> 35^{\circ}\text{C}$  were observed for 13 days with a peak of 3 days at  $41^{\circ}\text{C}$ . From the third week, the temperatures of the piles declined rapidly to environmental temperature, showing a decrease in microbial activity. The piles reached the thermophilic phase characterized by temperatures in the range of  $35$  to  $65^{\circ}\text{C}$ , indicating increase in microbial activity (INSAM; FRANKE-WHITTLE; GOBERNA, 2010; POLPRASERT; KOOTTATEP, 2017). The thermophilic phase was affected by the decrease in the ambient temperature (Figure 1), due to the loss of heat, which did not allow the composting process to reach higher temperature values. The pH values varied in the range of 6.7 to 7.5, indicating that the composting occurred efficiently according to the ideal values of 5.5 to 8.0 (PIOTROWSKA-CYPLIK *et al.*, 2009). In 90 days of the process the ratio C/N for pile 1 was 10.5, while for pile 2 it was 16.7. Final C/N ratio low values indicate possible material stabilization and low microbial activity (PROVENZANO *et al.*, 2001). According to Bustamante *et al.*, (2008) C/N ratios lower than 20 indicate an optimal degree of maturation. This indicates that the composted materials were stabilized.

#### Seed germination index (SGI)

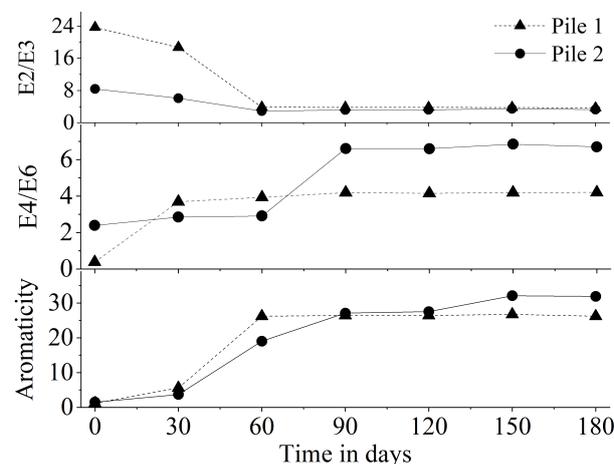
The germination index (SGI) is used to evaluate the toxicity and stabilization of composted material. For a compound to be considered non-phytotoxic, it must

present a germination index  $\geq 50\%$  (CHIKAE *et al.*, 2006). After 90 days of composting, the SGI value for pile 1 was 62.5%, while for pile 2 it was 87.5%. Studies indicate that ETP sludge from the industry contains large concentrations of metallic ions and potentially toxic organic contaminants (FANG *et al.*, 2017). The SGI values of the piles demonstrated that the compounds at 90 days are not phytotoxic, even employing ETP sludge from the ultra-processed food industry as feedstock.

#### Uv-Vis spectroscopy

The results obtained by the Uv-Vis analysis were used to calculate the  $E_2/E_3$ ,  $E_4/E_6$  and total aromaticity ratios, and are presented in Figure 2.

**Figure 2** – Ratio Uv -Vis during composting in 180 days



Source: The authors.

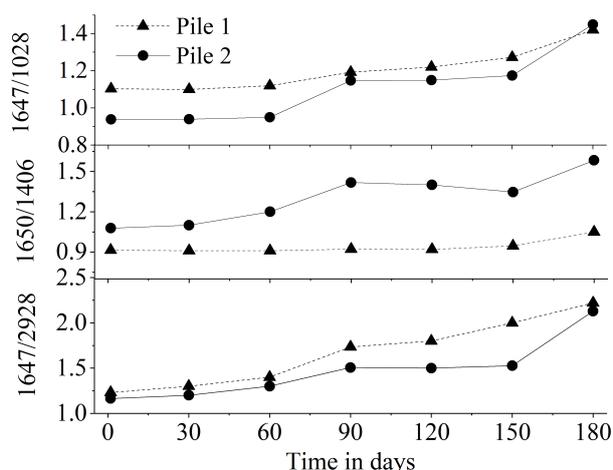
The reduction in  $E_2/E_3$  ratio for the two piles was observed in Figure 2. Between 60 and 150 days it was verified that  $E_2/E_3$  ratio presented stability, for pile 1 the mean ratio was  $3.83 \pm 0.02$ , while for pile 2 the mean was  $3.55 \pm 0.91$ . There was, therefore, degradation of aliphatic molecules, mainly in the first 60 days of the process (FIALHO *et al.*, 2010). Significant increase in total aromaticity values during the first 60 days of composting was observed, which is related to the biological activity of organic matter degradation and stabilization (FIALHO *et al.*, 2010; PEURAVUORI; PIHLAJA, 1997). The  $E_4/E_6$  ratio increased during the composting period, and the highest values observed were for pile 2 after 90 days, stabilizing at the end of the process. Studies indicate that the increase in  $E_4/E_6$  ratio during sludge composting of ETP, is associated with the increase in oxygenated functional groups of humic acids (KIM *et al.*, 2008; PAJACZKOWSKA *et al.*, 2003).

The characteristics of the products after 90 days of the beginning of the process pointed to a stabilized material, which when applied to the soil can increase the amount of organic matter, reducing the need to apply inorganic fertilizers.

#### FTIR spectroscopy

Figure 3 shows the ratios of wavenumbers 1647/2928, 1647/1406 and 1647/1030 for FTIR analyses.

**Figure 3** – FTIR ratio during composting in 180 days



Source: The authors.

The ratio 1647/2928 demonstrates the relationship between the aromatic/aliphatic carbons. For this ratio, pile 1 was seen to increase from 1.23 to 2.22, while pile 2 increased from 1.16 to 2.13 during the controlled composting period. According to Sen Chandra, (2007) this result is due to the formation of condensed polymers from the degradation of aliphatic structures, which is in agreement with the  $E_2/E_3$  ratio and total aromaticity. The 1647/1417 ratio demonstrates the ratio of aromatic/carboxylic carbons, which increased from 0.91 to 1.5 for pile 1, while for pile 2 the increase was from 1.07 to 1.58. According to He *et al.*, (2011), during the composting process the increase in aromatic and carboxylic carbons occurs. From the analysis of Figure 3, a significant reduction of the 1647/1417 ratio in relation to the 1647/2928 ratio can be observed, which may indicate higher formation of carboxylic acids, which is in agreement with the  $E_4/E_6$  ratio previously found. The 1647/1030 ratio demonstrates the relationship between aromatic carbons/polysaccharides and Figure 3 shows the increase from 1.10 to 1.42 in pile 1 and from 0.93 to 1.45 in pile 2. This increase was possibly caused by the degradation of polysaccharides metabolized by microorganisms during compost-

ing, producing new aromatic substances (DROUSSI *et al.*, 2009; HE, XIAO-SONG *et al.*, 2012; SONG *et al.*, 2015). Spectroscopic results indicated degradation of aliphatic compounds and polysaccharides during the composting process.

#### Determination of metallic ions

The concentrations of Ni, Cd, Cr, Pb, Cu, Mn and Zn metallic ions were evaluated in the product of 120 days and results are presented in Table 2.

**Table 2** – Concentration of metallic ions in the two piles in the period of 120 days and maximum limits allowed. (Mean  $\pm$  Sd; n3).

	Pile 1	Pile 2	USDA, (1980)	CCME, (2005)
Ni	<LQ	<LQ	200	62
Cd	<LQ	<LQ	10	3
Cr	<LQ	<LQ	1000	210
Pb	3.55 $\pm$ 0.43	5.248 $\pm$ 0.23	250	150
Cu	22.41 $\pm$ 0.02	21.64 $\pm$ 0.06	1000	400
Mn	135.48 $\pm$ 0.07	224.93 $\pm$ 0.11	-	-
Zn	458.58 $\pm$ 0.05	261.62 $\pm$ 0.02	2500	700

Concentration in  $\text{mg kg}^{-1}$ ; LQ: limit quantification.

Source: The authors.

The metallic ions Ni, Cd and Cr were below the limit of quantification of the equipment 0.157, 0.29 and 0.53  $\text{mg kg}^{-1}$  respectively. The total concentration of Ni, Cd, Cr, Pb, Cu, Mn and Zn metallic ions was below the control values established by the U.S department of agriculture and the Canadian Council of Ministers of the Environment for the use organic compounds in agriculture (CCME, 2005; USDA, 1980), indicating that the compound can be used according to these parameters. The concentrations of these metallic ions in biosolids are regulated by the characteristics of the composted material and the type of process used during the treatment (WUANA; OKIEIMEN, 2014). Although previous studies (AWASTHI *et al.*, 2016; KULIKOWSKA; GUSIATIN, 2015; MORETTI *et al.*, 2016) concluded that ETP sludge from the industry are sources of high concentrations of potentially toxic metals, the composted blends of ETP from the ultra-processed food industry obtained values below the limit established for the production of organic fertilizer. Therefore, it can be used according to international control standards.

## Conclusions

According to the results of the seed germination index, the final compound did not present phytotoxicity. The C/N ratio decreased to levels below 20 within 90 days, indicating stabilization of the compound. Monitoring of variation in Uv-vis and FTIR ratios indicates that the compound reached the degree of stabilization. The metal ion concentrations of the final compound are in accordance with permitted limits for the production of organic fertilizers. Therefore, the compost obtained from the sludge composting of the ultra-processed food industry can be considered as a fertilizer.

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