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## **Physico-chemical and spectroscopic quality assessment of compost issued from date palm waste valorization**

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### ► **To cite this version:**

Wadii Abid, Imen Ben Mahmoud, Saoussan Masmoudi, Mohamed Ali Triki, Stéphane Mounier, et al.. Physico-chemical and spectroscopic quality assessment of compost issued from date palm waste valorization. *Journal of Environmental Management*, 2020, 264, pp.110492. <10.1016/j.jenvman.2020.110492>. <hal-02559715>

**HAL Id: hal-02559715**

**<https://hal.science/hal-02559715v1>**

Submitted on 30 Apr 2020

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28biodegradation and mineralization. A total of 12 fluorescence excitation emission matrices  
29of composts were successfully decomposed into a four-factor model using PARAFAC  
30analysis. For the 4 main evidenced components, the excitation/emission (Ex/Em) wavelengths  
31were of 350/450 nm, 400/500 nm 450/530 nm and 325/400 nm attributable respectively to  
32humic-like and fulvic-like substances.

33**Keywords:** Date palm waste, goat manure, compost, FTIR, fluorescence spectroscopy,  
34PARAFAC analysis.

35

### 136Introduction

37

38 At present, organic waste valorization is an important strategy for human and environment  
39safeguard, reducing agricultural costs and chemical products use. Composting was considered  
40as an effective biological, economical and sustainable process to reuse the organic matter  
41wastes (Sellami et al., 2008; Hachicha et al., 2009a; Masmoudi et al., 2013; El Fels et al.,  
422016). Indeed, the composting process stabilizes the organic waste by its conversion into  
43humic substances and inactivate pathogens, allowing compost use for soil amendment  
44(Hachicha et al., 2006; Zhang and Sun, 2015). Several studies had developed the organic  
45agricultural wastes composting such as olive mill waste (Hachicha et al., 2006; Hachicha et  
46al., 2008; Masmoudi et al., 2013), sludge issued from the olive mill waste evaporation ponds  
47and poultry manure (Hachicha et al., 2009b), horticultural wastes (Gavilanes-Terán et al.,  
482015), green waste (Abid et al., 2015; Zhang and Sun, 2015) and *Acacia* residues (Brito et al.,  
492015). Consequently, the composting process and the final product quality depend on many  
50factors such as the raw materials nature and their formulation, the windrow and the ambient  
51temperatures, the windrow materials water content and returning frequency and the microbial  
52activity (Masmoudi et al., 2013; Cesaro et al., 2015). Previous studies established that

53compost quality was especially related to its stability and maturity (Hachicha et al., 2009a;  
54Cesaro et al., 2015; Nikaeen et al., 2015). Further, compost quality was assessed with  
55different physico-chemical (pH, electrical conductivity (CE), carbon measure, C/N,  
56temperature and moisture) and biological (microbial population, respiration, germination  
57index, solvitan test) parameters, associated to spectroscopic techniques (FTIR, UV-visible and  
58fluorescence spectroscopy) (Sellami et al., 2008; Yu et al., 2011; Masmoudi et al., 2013; El  
59Fels et al., 2014; Mehta et al., 2014; Song et al., 2015; Kulikowska, 2016).

60 In the Mediterranean basin, date palm is largely cultivated. Indeed, in Tunisia, there are  
61more than 4.5 million trees producing an important waste quantities essentially palm leaf  
62waste (16 kg/tree yearly) (Ben Salah, 2014).

63 In the present study, an industrial ecological interest was considered in the date palm waste  
64valorization by its co-composting with goat manure and compost quality assessment, using  
65physico-chemical and biological parameters associated to spectroscopic techniques (FTIR,  
66UV-visible and fluorescence spectroscopy) to evaluate the organic matter biodegradation. The  
67final product availability would contribute to a sustainable development.

68

## 69 **2. Materials and methods**

70

### 71 *2.1. Composting procedure*

72

73 In this study, goat manure and crushed date palm waste composed essentially of palm  
74leaflets both collected from the Oasis of Cheneni-Gabes (South of Tunisia) were used for the  
75composting procedure. The physico-chemical characteristics of the mentioned materials are  
76presented in Table 1.

77 The composting procedure was carried out at the Chenini Oasis Protection Association  
78composting site, as described in Abid et al. (2016). The windrow was watered with the  
79previous date palm waste soaking-water, characterized by ~~aeidie~~low pH (5.83) and its  
80important EC (5.61 mS cm<sup>-1</sup>) and mesophilic flora (4.1 x 10<sup>7</sup> ufc mL<sup>-1</sup>) to keep a moisture  
81range of around 50%.

82 During the composting procedure, homogeneous and representative samples were collected  
83from the windrow and refrigerated until analysis. Each sample consists of a mixture of 9 sub-  
84samples taken at different points of the windrow. Before analysis, the samples were dried at  
85105 °C, and then mechanically crushed to obtain homogeneous powder.

86

#### ~~287~~ *Physico-chemical analysis*

88

89 The windrow temperature was measured using digital thermometer (type Alla, France)  
90weekly during the mesophilic and the thermophilic composting phases and monthly until its  
91maturity. The presented temperature value consists of a mean of 12 measurements at different  
92points and depths of the windrow.

93 All the dried and crushed samples were submitted to physico-chemical analysis using  
94standard methods. The pH and EC values were measured in a solution consisting of 20 g of  
95sample in 100 ml of distilled water. Organic matter (OM) was determined by dry matter and  
96weight loss on ignition at 550 °C for 4 h in a muffle furnace (Hachicha et al., 2009b). The  
97total Kjeldahl nitrogen (TKN) was determined according to AFNOR (1981). Phosphorus  
98content, macro and micro elements were determined using standard methods as mentioned in  
99our previous study (Abid et al., 2016).

100

#### ~~101~~ *Dissolved organic matter extraction (DOM)*

102

103 The organic matter extraction was achieved according to Song et al. (2015) with a slight  
104 modification. All the dried compost samples were dissolved in distilled water (solid to water  
105 ratio of 1:10, w/v) for 24 h in a rotary shaker (Heidolph reax 20) at room temperature. The  
106 suspensions were then centrifuged at 5000 rpm for 15 min, filtered through a 0.22 µm  
107 membrane filter and refrigerated until spectroscopy analysis. The Dissolved Organic  
108 Carbon (DOC) was measured using multi TOC/TN analyzer type (Analytic Jena multi N/C  
109 2100S).

110

111 2.4.

#### *Biological analysis*

112

#### *2.4.13 Microbial respiration*

114

115 Microbial respiration was assayed according to Pallo et al. (2009) and Yu et al. (2011) with  
116 some modifications. A sample of 50 g compost was incubated for 24 h in bottle of 1 L where  
117 a tube containing 10 mL of NaOH (0.1 N) was placed. The bottles have the same windrows  
118 temperature when the sample was collected. The method involves the CO<sub>2</sub> trapping by sodium  
119 hydroxide (0.1 N NaOH), and then titration with hydrochloric acid (0.1 N HCl) with the  
120 phenolphthalein as a pH indicator. The amount of CO<sub>2</sub> released (C-CO<sub>2</sub>) is given by the  
121 following formula:

$$122 \text{CO}_2\text{-C (mg /50 g of compost)} = (\text{Control } V_{\text{HCl}} - \text{Sample } V_{\text{HCl}}) \times 2.2;$$

123 Control  $V_{\text{HCl}}$  (ml): the volume of HCl (0.1 N) used for the titration of NaOH in the control;

124 sample  $V_{\text{HCl}}$  (ml): the volume of HCl (0.1 N) used for compost sample titration; 2.2 = a

125 coefficient corresponding to 1 mL of HCl (0.1 N) CO<sub>2</sub> (g) determined.

126

#### 127 4.2. *Microbial counts*

128

129 The microbial count was achieved by number of colony forming units count (cfu). A 10 g  
130 mass of matured compost was suspended in 90 mL of a sterile peptone water solution and  
131 stirred at 150 rpm for 10 min at 28 °C. The suspension was used for serial successive decimal  
132 dilutions ( $10^{-1}$  to  $10^{-7}$ ) and 1 mL was used to inoculate Plate Count Agar medium (PCA). The  
133 cell enumeration was for 72 h  $\pm$  4 h at 37 °C  $\pm$  1 °C (Hachicha et al., 2006).

134

#### 135 4.3. *Phytotoxicity test*

136

137 The compost phytotoxicity was evaluated using the germination index (GI) according to  
138 the method described by Cesaro et al. (2015). This was determined as follows: 20 g of dry  
139 weight compost were extracted with 200 mL of distilled water, stirred for 2 h and then  
140 centrifuged at 9000 rpm. Ten *Lipidium sativum* seeds were evenly distributed on filters papers  
141 in Petri dishes (10 cm diameter) and moistened with 10 mL of the compost extract. For each  
142 sample, three replicate dishes were incubated at 25 °C for three days in the dark. The numbers  
143 of germinating seeds per dishes were counted and the length of each seed root was measured.  
144 For the control, 10 mL of distilled water were used to moisten the filter. The GI was  
145 calculated using the following formula:

146  $GI (\%) = [(Seed\ germination \times Treated\ root\ length) / (Seed\ germination \times Control\ root\ length)]$   
147  $\times 100.$

148

#### 149 2.5. *Spectral analysis*

150

##### 151 5.1. *UV-visible*

152

153 The Dissolved Organic Matter (DOM) UV absorbance measurements from 200 to 800 nm  
154 were recorded on the Shimadzu UV-1800 spectrophotometer. The base line was carried out  
155 with osmosis water used for organic matter extraction. The analyses were performed at  
156 neutral pH in quartz cuvettes (1 mm path length) at room temperature. The spectra were  
157 recorded in the range of 200 to 800 nm. To characterize the DOM, three quality indices were  
158 determined: these were Specific Ultraviolet Absorbances (SUVA) at 254 nm (SUVA<sub>254</sub>),  
159 269 nm (SUVA<sub>269</sub>) and 280 nm (SUVA<sub>280</sub>). The SUVA<sub>254</sub>, SUVA<sub>269</sub> and SUVA<sub>280</sub> were  
160 calculated as ABS(254)/DOC, ABS(269)/DOC, and ABS(280)/DOC (Song et al.,  
161 2015). The ratio of the absorbance at 250 nm to that at 365 nm ( $E_2/E_3$ ) was determined to  
162 describe the humification process intensity (reference). The ratio of the absorbance at 280 nm  
163 to that at 472 nm ( $E_2/E_4$ ) was used as an indicator of the relative lignin amounts at the  
164 beginning of the humification (reference). Finally the ratio between the absorbance at 472 and  
165 that at 664 nm ( $E_4/E_6$ ) was the most indicative parameter related to the humification degree  
166 (Sellami et al., 2008).

167

## 168 5.2. *Fourier transform infrared spectroscopy*

169

170 The dried compost samples were analyzed using an infrared spectrometer type FTIR  
171 Nicolet iS50 (Thermo Scientific, USA). The spectra were scanned over a wavenumber range  
172 of 4000-400  $\text{cm}^{-1}$  with a resolution of 4  $\text{cm}^{-1}$ .

173

## 174 5.3. *Excitation-emission matrix (EEM) fluorescence spectra*

175

176 The EEM spectra were achieved on the DOM of the different samples collected during the  
177composting process using a spectrofluorimeter HITACHI F4500 with scanning emission (Em)  
178and excitation (Ex) wavelengths spectra from 200 to 800 nm at 10 nm increments and the  
179scan speed was set at 2,400 nm·Min<sup>-1</sup>. Excitation and emission slit were fixed at 10 nm and  
180the detector time response in automatic mode. Fluorescence spectroscopy was carried out on  
1814 two time diluted extract to ~~avoid~~correct the inner filter effect ~~(Luciani)~~. The obtained  
182fluorescent EEM were processed by Parallel Factor analysis (PARAFAC) and  
183PROGMEEF~~progMEFF~~ interface provided by PROTEE laboratory, Univ. Toulon (France),  
184allowing the extraction of the most representative fluorescent components to characterize the  
185source and nature of the sample organic matter (Yu et al., 2011; Mouloubou et al., 2016).

186

## 187 2.6. *Statistical analysis*

188

189 The data were submitted to variance analysis using Portable IBM SPSS statistics version  
19019. All analyses were performed in triplicate. Principal component analysis (PCA) was used  
191to examine multivariate relationship between the physico-chemical and biological parameters  
192progress during composting. Pearson's correlation coefficient (R) was used to evaluate the  
193linear correlation between two parameters at a confidence level of 95% ( $p < 0.05$ ).

194

## 195 **3. Results and discussion**

196

### 197 3.1. *Composting performance*

198

#### 199 3.1.1. *Temperature profiles*

200

201 Temperature is one of the main parameters used to monitor the composting process. As  
202 shown in Figure 1, the temperature profile of the date palm waste and goat manure co-  
203 composting was divided into 4 phases: mesophilic, thermophilic, cooling and maturity.

204 During the first 6 days of the composting process, windrow temperature showed a rapid  
205 increase and exceeded the 40 °C (Fig. 1). This period, represented mesophilic phase,  
206 characterized by the easily degradation of the organic matter by mesophilic microorganisms.  
207 After this period, the temperature rose to above 53 °C, corresponding to the thermophilic  
208 phase and remained approximately at 50 °C for 7 days. This high temperature enhanced the  
209 windrow sanitation (Zhang et al., 2014; Zhang et al., 2015). This temperature increase was  
210 due to the intense microbial activity.

211 By the 20<sup>th</sup> day of composting, the temperature started to decrease announcing the cooling  
212 phase, characterized by the mesophilic microorganism colonization which contributed to the  
213 cellulose and lignin degradations and the humus formation (El Fels et al., 2014). According to  
214 Zhang et al. (2014), the traditional composting requires between 90 and 270 days to produce  
215 mature compost. In this study, the maturity was reached after 190 days of composting; this  
216 fact may be attributed to the effect of the initial date palm waste soaking step. However, in  
217 another study date palm waste composting was held in 14 months (El Ouaquoudi et al., 2015).  
218 At the maturity, the temperature was 35 °C. At the 62 and 133 days, the two observed  
219 temperature increases were the result of the windrow return reinforcing the aeration, which  
220 stimulate the microorganism activity (Gavilanes-Terán et al., 2015). Also, at the maturity  
221 phase, the windrow temperature was highly related to the ambient temperature which  
222 increased by the summer beginning from the day 62 until the process end.

223

#### 224 1.2. *pH and EC progress*

225

226 During the date palm waste and goat manure co-composting process, the pH value showed  
227a high correlation with the composting time ( $R^2 = 0.91$ ) and varied slightly but remained  
228globally neutral while an increase in EC value was noticed with a linear model and also high  
229correlation with the composting time ( $R^2 = 0.89$ ). However, a pH decrease by 0.43 units was  
230registered at day 84 (Table 2). According to the literature, this decrease was explained by the  
231raw material degradation essentially lignicellulose, and the organic acids release (Hachicha et  
232al., 2008; Tripetchkul et al., 2012; El Fels et al., 2014). After this fluctuation, the pH value  
233was stabilized with the humic substances biosynthesis, these acted as pH buffer (El Fels et al.,  
2342014). It could be noticed that the pH profile was different from that found by El Fels et al.  
235(2014) who mixed palm waste with sewage sludge characterized by an acidic pH. The  
236observed difference would be attributed to the composted raw materials typology and  
237pretreatment. The mature compost pH value was in the range of the compost standard value  
238(between 6 and 7.5), confirming its suitability for agricultural use as specified by Gavilanes-  
239Terán et al. (2015).

240 The EC was also measured during the date palm waste co-composting and reflected the  
241compost [salinity ion content](#) and its importance for plant growth (Gavilanes-Terán et al., 2015;  
242Singh and Kalamdhad, 2015). The EC values increased during composting process from 2.71  
243to 4.33  $\text{mS cm}^{-1}$  (Table 2) reflecting the OM mineralization and the ammonium ions and  
244mineral salts release (El Fels et al., 2014; Gavilanes-Terán et al., 2015).

245

### 246 1.3. *Organic matter degradation and C/N progress*

247

248 During the composting process, the OM and C/N progress were the important key  
249parameter reflecting raw materials biodegradation and transformation. At the process end, the  
250OM was reduced by 63% exhibiting the raw materials deterioration and their oxidation to  $\text{CO}_2$

251 and H<sub>2</sub>O (Table 2). Indeed, the OM was decomposed and transformed by microorganisms to  
252 stable humic substances; the change was highlighted by a mass loss (Singh and Kalamdhad,  
253 2015). According to Kulikowska et al. (2015), the OM mineralization amount and rate depend  
254 on the raw materials chemical composition, and especially the fibers content like lignin. The  
255 date palm waste and goat manure co-composting showed an OM loss of 36.58% although in  
256 another study El Ouaquoudi et al. (2015) observed an OM loss of 29% and 62% respectively  
257 for date palm waste and date palm/couch-grass clipping wastes mixture subjected to  
258 composting during 14 months. These results proved the important effects of date palm waste  
259 soaking step and goat manure use investigated in this study.

260 Simultaneously, the OM oxidation led to carbon loss and CO<sub>2</sub> release, increasing the total  
261 nitrogen content in the medium. In fact, palm waste and goat manure co-composting exhibited  
262 a C/N ratio decrease by 3 folds at the process end confirming its maturity (Table 2) (Tian et  
263 al., 2012).

264

### 265 3.2. *Biological parameters evolution*

266

#### 267 3.2.1. *Respiration*

268

269 Respiration was usually used in compost stability and quality assessment as it refers to the  
270 microorganisms activity and OM degradation (Varma and Kalamdhad, 2014; Nikaeen et al.,  
271 2015). In this experiment, the initial respiration value was low, expressing microorganism  
272 adaptation to the environment. After 6 days, the highest respiration value was observed  
273 corresponding to thermophilic phase (Fig. 2) with intense microorganisms activity. According  
274 to Nikaeen et al. (2015), this activity would be attributed to labile OM availability which  
275 stimulates microbial activity. After this stage, the respiration decreased then a second pic was

276observed at day 33 just after the windrow return that stimulated the microbial activity. From  
277the day 34 and until the day 190, the respiration decreased slowly to reach 5.53 mg/50 g  
278reflecting compost stability (Gómez et al., 2006).

279

## 280 2.2. Germination index (GI)

281

282 During the composting process, the compost quality of the different collected samples was  
283assessed by phytotoxicity control expressed as GI (Fig. 3). For the first 45 days, the GI  
284showed fluctuations related to the OM degradation, the organic acids and the ammonia  
285releases at relatively high concentrations considered as phytotoxic (Zhang et al., 2014). Also,  
286phenols, alkaloids and ketones as well as others flavonic compounds are considered as seed  
287germination inhibitory agents (Hachicha et al., 2009a; El Fels et al., 2016). From the 60<sup>th</sup> day,  
288the germination index increased gradually, indicating the toxic compounds reduction and the  
289presence of stable humic substances, rich in organic matter synthesized and accumulated  
290during the cooling phase (El Fels et al., 2014; El Fels et al., 2016). According to Sellami et al.  
291(2008) and Zhang et al. (2014), the final GI value about 89% may reflect compost maturity  
292and phytotoxicity lack. According to Gavilanes-Terán et al. (2015) the obtained compost  
293characterized with a GI exceeding 50%, could be used in agriculture without any phytotoxic  
294effect. The GI followed a linear model with a relatively correlation with time during the  
295composting process ( $R^2 = 0.89$ ).

296

## 297 3.3. Spectral analysis

298

### 299 3.1. UV-visible spectroscopy

300

301 The UV-visible spectroscopy was used to characterize the DOM, reflecting the organic  
302 matter humification during the co-composting of date palm waste and goat manure, as well as  
303 the final compost quality. The UV-visible spectra of the initial and final composted materials  
304 showed an intensity decrease from the wavelength of 267 to 295 nm (Fig 4). According to  
305 Lim et al. (2015), this decrease is related to lignin biodegradation, since the absorbance region  
306 around 280 nm is assigned to lignin and aromatic compounds.

307 Different specific ultraviolet absorbance  $SUVA_{254}$ ,  $SUVA_{269}$  and  $SUVA_{280}$  were calculated  
308 (Table 3) and showed respectively an important increase by 2 times, while comparing the  
309 initial state (raw materials) to the mature compost. Both of the  $SUVA_{254}$  and the  $SUVA_{280}$  were  
310 relative to the aromatic constituent contents in the DOM (Wang et al., 2013), while the  
311  $SUVA_{269}$  was related to lignin derived aromatic carbon (He et al., 2014). The SUVA values  
312 increase, confirmed the organic matter degradation and lignin oxidation into aromatic  
313 compounds during composting process. In this study, the absorbance ratio  $E_2/E_3$ ,  $E_2/E_4$  and  $E_4/$   
314  $E_6$ , used to control the organic matter biodegradation and humic substances synthesis, varied  
315 during the composting process (Table 3); this may be attributed to the organic matter rich in  
316 lignin, difficult to be biodegraded. However, in the first 6 composting days,  $E_4/E_6$  decreased  
317 because of the mesophilic microorganism activity and increased after the windrow returns to  
318 reach 7.40 by the day 21, then decreased successively as a result of raw organic materials  
319 humification and humic substances synthesis (Zbytniewski and Buszewski, 2005). By the day  
320 84 of composting, the ratio  $E_4/E_6$  increased again after windrow return and then decreased at  
321 the end of the process. Wang et al. (2013) found a reduction in  $E_4/E_6$  during the active phase  
322 that rose slowly thereafter. According to Zbytniewski and Buszewski (2005), the final  $E_4/E_6$   
323 value 5.7 is an index of mature humic acids. More recently, Haddad et al. (2015) had also  
324 found an  $E_4/E_6$  ratio value of 6 at the end process of composting olive pomace and cattle  
325 manure. They had admitted this ratio as an indicator of humification intensity. Thus, when the

326ratio  $E_4/E_6$  becomes low the compost is mature and humus particles are complexes. Sellami et  
327al. (2008) associated the large values of  $E_4/E_6$  ratio to the presence of small size organic  
328molecules or more aliphatic structures with high functional groups content. The  $E_2/E_4$  index  
329progress presented the same trend as that of the  $E_4/E_6$  ratio during the composting process.  
330This ratio intensity would inform on the lignin and other materials at the beginning of  
331humification (Zbytniewski and Buszewski, 2005). Furthermore, the observed decreases in  $E_2/$   
332 $E_4$  index reflected an intense depolymerisation and microbial monomeric compounds  
333degradation (Sellami et al., 2008; Lim and Wu, 2015).

334

### 335 3.2. FTIR spectroscopy

336

337 The infrared spectroscopy was usually used as a qualitative analysis to identify and monitor  
338the chemical groups and bands present in the composted materials, describing the organic  
339matter degradation. Figure 5 represents the materials FTIR spectra at various composting  
340stages. The major compost FTIR bands are listed in Table 4, where the bands between 4,000  
341and 2,000  $\text{cm}^{-1}$  were attributed to the humic substances extracted from compost (El Fels et al.,  
3422014). At 3,290  $\text{cm}^{-1}$ , the pic was more important at the initial composting process than at the  
343end, and was assigned to phenol O-H group vibration; this group was degraded during the  
344composting process until the compost maturity (Masmoudi et al., 2013). This result is in  
345accordance with the phytotoxicity test results. The bands at 2,920 and 2,850  $\text{cm}^{-1}$ ,  
346characteristic of the aliphatic C-H groups, were more intense at the initial composting  
347samples. Indeed, during the composting process, the organic matter was biodegraded and the  
348carbon content decreased (El Fels et al., 2014). Also, the bands at 1,690  $\text{cm}^{-1}$  attributed to  
349C=O group in the organic matter, decreased during composting process by the microbial  
350activity. The bands between 1,600 and 900  $\text{cm}^{-1}$  were attributed to cellulose and early

351decomposition product (Grube et al., 2006). El Ouaquoudi et al. (2015) had assigned the peak  
352at 1,525 cm<sup>-1</sup> to lignin considering the date palm waste lignocellulosic material.

353In the region between 1,300 and 1,400 cm<sup>-1</sup>, the bands were more intense in the final compost  
354sample. According to Grube et al. (2006), this region is characteristic of nitrate bands  
355appearance, usually detected at the latter composting stage, when raw materials were well  
356composted. However, the peak at 1,170 cm<sup>-1</sup> was more intense at the initial mixture and was  
357attributed to C-O-C groups of cellulose and hemicellulose as reported by Xu et al. (2013) at  
3581,160 cm<sup>-1</sup>. At 1,560 cm<sup>-1</sup>, the band decreased with the composting time, revealing peptide  
359degradation in manure (Hachicha et al., 2009b).

360 The changes in the organic matter content and in aromaticity degree were also assessed by  
361the calculation of the 3 absorption ratios 1,650/2,845, 1,525/2,925 and 2,920/1,640 (Table 5).  
362During the composting process, the 1,650/2,845 and 1,525/2,925 ratios showed an increase  
363while the specific ratio of aliphatic C/aromatic C (2,920/1,640) decreased. These results could  
364be explained by an increase in aromaticity and the biosynthesis of aromatic structures, such as  
365humic-like and fulvic-like substances, with the decomposition/transformation of aliphatic  
366components, polysaccharides, and alcohols (Wu et al., 2011; El Ouaquoudi et al., 2015).

367 These infrared spectroscopy findings were in accordance with those of the physico-chemical  
368parameters, both of the data confirmed the date palm waste and goat manure co-composting  
369product maturity and quality.

370

### 371 3.3. Fluorescence spectroscopy

372

373 The three dimensional EEM fluorescence spectra of the DOM composted material progress  
374are presented in Figure 6 (A). These spectra were characterized by the presence of a  
375fluorophore pipeak having different intensities depending on the composting progress and it

376highest intensity was registered in the final compost spectra after 190 days of composting.  
377The variable fluorophore pi peak had an excitation/emission wavelength pairs between 350-  
378400 (mean value = 375)/400-500 (mean value = 470) nm, characteristic of humic-like  
379substances and high molecular weight (Albretch et al., 2015; Song et al., 2015; Mouloubou et  
380al., 2016). According to He et al. (2015), the fluorophores with emission wavelengths  
381exceeding 380 nm are ascribed to humic-like species, while those less than 380 nm represent  
382protein-like species, such as tyrosine and tryptophan-like species. Based on these data, no  
383proteins like fluorophore were detected during the process. Consequently, the total  
384luminescence would be issued from the humic-like compounds. However, the humic like  
385signal could be generated by a mixture of fluorescent compounds. The ratio of the red zone to  
386the total EEM fluorescent area had increased by 76% at the end process revealing the  
387formation of humic substances (Figure 6 B). The ratio fluctuation during the first 60 days of  
388composting was due to the microbial activity and the degradation of organic matter.

389 As shown in Figure 7, the PARAFAC analysis decomposed the EEM fluorescent spectra  
390into 4 fluorophore components (ConcordiaCORCONDIA > 66%). The first component is  
391located at 350/450 nm (S1) and it is representative of the humic like compounds. The second  
392(S2) is located at 400/500 nm and is less conjugated than the third (S3) one located at 450/530  
393nm. These entire three components represent the humic-like group. The fourth (S4) represent  
394the fulvic like component with an excitation at 325/400 nm. From S4 to S1 to S2 to S3 we can  
395observe a conjugate trend inducing a red shift evolution for both excitation and emission  
396wavelengths. Observing the ratio of S3 over S2 contribution during the composting process,  
397we may note that the S1 contribution increase more than other components. This is a  
398confirmation of the FTIR trend were C=C is increasing during the same time. Moreover,  
399considering the increase of the SUVAs, the fluorescence intensity and the GI while COD and

400microbial respiration decreases, we may suggest that at the end of the process (over 120 d),  
401both of conjugated carbon and aromaticity increased.

402

#### 403 3.4. *Compost specification*

404

405 The compost prepared with date palm waste and goat manure co-composting was  
406characterized by a neutral pH and relatively high EC. It also exhibited a low C/N ratio and a  
407high GI value (88%) reflecting its stability (Table 6). Considering its total nitrogen content  
408inferior to 3% and organic matter inferior to 55%, the produced compost could be classified  
409as vegetal compost (French standards NF U 44-051, 1981).

410After six months of palm waste and goat manure co-composting while comparing to El Fels  
411et al. (2014) compost who also studied the palm waste co-composting with sludge, the  
412produced compost present higher carbon content than that previously described but a lower  
413nitrogen content.

414

#### 415 3.5. *Statistical analysis*

416

417 The correlation matrix between the physico-chemical and the biological parameters  
418progress during the composting process (Table 7) and the PCA plan representation (Fig 8)  
419showed a significant positive correlation between pH, C/N ratio and OM content. However,  
420during the composting process, the organic matter biodegradation decreased and as a result,  
421the carbon content was reduced as well as C/N ratio. In addition, temperature and respiration  
422were highly correlated revealing that microorganism activity depends on the temperature and  
423specificity for each composting stage (mesophilic and thermophilic). Moreover, a negative  
424correlation was observed between the germination index and the EC. These are opposed to the

425previous group including OM, pH and C/N. Indeed, the organic acids and the other phytotoxic  
426components released during composting process affected the seed germination.

427

#### 428. **Conclusions**

429

430 Date palm waste and goat manure co-composting is an efficient solution providing organic  
431matter for a sustainable agriculture. During the composting process, physico-chemical and  
432biological parameters were in correlation, proving the organic matter biodegradation and the  
433compost produced quality. The use of spectroscopic techniques approved the lignin  
434degradation and the humic substance components formation. The obtained compost was  
435without phytotoxicity and ready for agriculture application.

436

#### 437**Acknowledgements**

438

439 The authors would like to express their gratitude to the staff at the Oasis Cheninni  
440Safeguard Association for their help in providing the material necessary for this study.

441

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565 manure amended with maize straw. *Environ. Sci. Pollut. Res.* 23, 12128-12139.

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570**Table 1.** The main physico-chemicals characteristics of the initial raw materials (dry weight  
571basis).

Parameters	Palm waste	Goat manure
pH	6.10 ± 0.18	8.02 ± 0.25
CE (µS cm <sup>-1</sup> )	2.01 ± 0.22	9700.00 ± 5.00
Dry matter (%)	18.30 ± 0.27	92.20 ± 0.53
Organic matter (%)	91.90 ± 0.55	64.65 ± 0.82
Organic carbon (%)	53.30 ± 0.55	37.50 ± 0.82
Total Kjeldahl nitrogen (%)	0.50	0.90
C/N	106.60	41.66
Phosphor (mg/g DM)	1.20	11.85
Mineral matter (%)	8.10 ± 0.55	35.34 ± 0.79
K <sup>+</sup> (%)	1.33	2.40
Ca <sup>2+</sup> (%)	1.11	0.18
Mg <sup>2+</sup> (%)	0.36	0.01
Na <sup>+</sup> (%)	0.15	0.00
Fe <sup>2+</sup> (mg/kg DM)	259	89.46
Zn <sup>2+</sup> (mg/kg DM)	6	0.35
Cu <sup>2+</sup> (mg/kg DM)	<5	0.12
Mn <sup>2+</sup> (mg/kg DM)	23	2.07

572 CE: Electrical conductivity, C/N: Carbon/Nitrogen; DM: dry matter.

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576**Table 2.** The main palm waste and goat manure physico-chemical characteristics during co-  
577composting.

Compostin							
g		MM					
duration	pH	CE	(%)	OM	C	N	C/N
(Day)		(mS cm <sup>-1</sup> )		(%)	(%)	(%)	

0	7.51 ± 0.06	2.71 ± 0.13	19.65 ± 0.49	80.35 ± 0.49	46.60	0.77	60.52
6	7.45 ± 0.05	2.70 ± 0.09	20.70 ± 0.85	79.30 ± 0.85	45.99	0.91	50.54
14	7.46 ± 0.08	2.86 ± 0.11	26.70 ± 0.85	73.30 ± 0.85	42.51	0.83	51.22
21	7.37 ± 0.13	2.78 ± 0.07	22.25 ± 0.49	77.75 ± 0.49	45.09	0.87	51.83
28	7.36 ± 0.10	2.55 ± 0.14	24.30 ± 0.99	75.70 ± 0.99	43.90	1.08	40.65
33	7.42 ± 0.03	2.83 ± 0.06	23.10 ± 0.57	76.90 ± 0.57	44.60	0.84	53.10
42	7.41 ± 0.08	2.93 ± 0.06	23.65 ± 0.64	76.65 ± 0.64	44.46	0.85	52.30
62	7.33 ± 0.06	2.93 ± 0.10	24.90 ± 1.56	75.10 ± 1.56	43.56	0.93	46.84
84	7.08 ± 0.12	3.03 ± 0.16	26.55 ± 1.77	73.45 ± 1.77	42.60	0.90	47.33
119	7.16 ± 0.05	3.32 ± 0.06	29.50 ± 0.13	70.50 ± 0.13	40.89	1.01	40.48
133	7.25 ± 0.10	3.59 ± 0.04	28.90 ± 0.28	71.10 ± 0.28	41.24	1.10	37.49
190	7.12 ± 0.17	4.33 ± 0.06	49.05 ± 2.19	50.95 ± 2.19	29.55	1.42	20.81

578CE: Electrical conductivity; OM: Organic matter; MM: Mineral matter.

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584**Table 3.** The DOM characteristics progress during the composting process.

Composting duration (Day)	DOC (mg C/l)	SUVA (x10 <sup>-3</sup> )					
		254	269	280	E <sub>2</sub> /E <sub>3</sub>	E <sub>2</sub> /E <sub>4</sub>	E <sub>4</sub> /E <sub>6</sub>
0	845.6	1.64	1.50	1.49	4.43	17.54	4.50
6	760.0	1.63	1.47	1.35	4.49	16.55	4.13
14	658.6	1.55	1.38	1.23	4.67	18.07	5.63
21	518.7	1.81	1.60	1.43	4.75	20.08	7.40
33	588.2	2.17	1.89	1.69	4.63	19.47	6.38
42	431.5	2.65	2.32	2.06	4.65	19.71	5.63
84	748.8	2.13	1.83	1.63	4.93	22.22	9.17
190	397.9	3.48	3.04	2.69	4.63	18.79	5.70

585 DOM: dissolved organic matter; DOC: dissolved organic carbon; SUVA: specific ultraviolet

586absorbance.

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598**Table 4.** The mean infrared spectral bands related to the compost.

Wavenumber (cm <sup>-1</sup> )	Assignment
3200-3600	Hydroxyl stretching vibration of phenol, alcohol and organic acid and N-H vibrations from amines and amides
2850-2964	Aliphatic C-H group
1750-1600	C=O amount in the organic matter
1716	C=C stretching vibration of acids and ketones carboxyl groups
1620-1650	Unsaturated aromatic C=C or ketone acids
1540-1580	Amide II bands and carboxylic group, N-H deformation
1525	Lignin pic
1420-1450	Aliphatic C-H deformation
1380-1400	O-H deformation of phenolic structure, C-H deformation in cellulose and hemicellulose
1220-1270	C-OH stretching of aromatic groups
1003-1200	C-O stretching of polysaccharides
1112	C-OH stretching of aliphatic OH
720-590	Alcohol, OH out of plane bend

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607 **Table 5. Evolution of the humification ratios during the composting process.**

Compostin g progress (Day)	Absorbance ratios et définit wavenumber		
	1_650/2_845	1_525/2_925	2_920/1_640
Initial	1.43	0.37	1.60
6	1.51	0.49	1.31
14	1.94	0.79	1.00
21	1.67	0.61	1.22
28	1.65	0.60	1.12
33	1.68	0.71	1.07
42	1.80	0.66	1.02
62	1.50	0.50	1.32
84	1.64	0.80	0.96
119	1.70	0.67	1.01
134	1.70	0.69	0.98
190	1.95	1.13	0.71

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618**Table 6.** The palm waste and goat manure compost characteristics.

619	Parameters	Compost	
620	CE: Electrical	pH	7.12 ± 0.17
		EC (mS cm <sup>-1</sup> )	4.33 ± 0.06
621	dry matter.	Moisture (%)	25.86 ± 1.11
		Dry matter (%)	74.14 ± 1.11
622		Organic matter (%)	50.95± 2.19
		Total organic carbon (%)	29.55 ± 2.19
623		TKN (%)	1.42± 0.00
		C/N	20.81± 2.19
624		Mineral matter (%)	49.05 ± 2.19
625	<b>Table 7.</b>	Phosphor (g kg <sup>-1</sup> DM)	10.24
		K (g kg <sup>-1</sup> DM)	88
626	between physico-	Mg (mg kg <sup>-1</sup> DM)	227
627	biological	Na (mg kg <sup>-1</sup> DM)	127
		Fe (mg kg <sup>-1</sup> DM)	57
628	materials.	Mn (mg kg <sup>-1</sup> DM)	1.85
		Zn (mg kg <sup>-1</sup> DM)	0.60
	pH	Cu (mg kg <sup>-1</sup> DM)	0.78
		Germination index (%)	88.48 ± 0.52

conductivity; DM:

Correlation matrix

chemical and  
composted

Respiratio GI

pH	1						
CE	-0,704	1					
Température	-0,066	-0,012	1				
OM	0,688*	-0,930	-0,067	1			
C/N	0,735**	-0,848	-0,110	0,909**	1		
Respiration	0,476	-0,488	0,734**	0,399	0,296	1	
GI	-0,837	0,875**	-0,122	-0,753	-0,745	-0,524	1

629\*: Correlation is significant at the 0,05 level (2-tailed).

630\*\*: Correlation is significant at the 0,01 level (2-tailed).

631CE: Electrical conductivity; OM: Organic matter; GI: Germination index.

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### Figure captions

644**Figure 1.** Windrow and ambient temperatures evolution during date palm waste (2/3) and  
645goat manure (1/3) co- composting.

646**Figure 2.** Respiration profile reflecting microorganism activity during composting process.

647**Figure 3.** The germination index progress during date palm waste and goat manure co-  
648composting.

649**Figure 4.** The UV-visible spectra of the initial and final compost samples.

650**Figure 5.** Infrared spectra of various composting stage.

651T0: initial compost sample; T6: compost sample at 6 d ; T14: compost sample at 14 d ; T21:  
652compost sample at 21 d ; T28: compost sample at 28 d ; T33: compost sample at 33 d ; T42:  
653compost sample at 42 d ; T62: compost sample at 62 d ; T84: compost sample at 84 d ; T119:  
654compost sample at 119 d ; T134: compost sample at 134 d ; T190: final compost sample.

655**Figure 6. (A)** Excitation-emission matrix (EEM) fluorescent spectra of the different DOM  
656extracted from compost materials during co composting process expressed in days (d); **(B)**  
657The ratio evolution of the red zone specific of humic substances to the total fluorescent area.

658 **Figure 7.** Excitation-emission matrix spectra of principal fluorescent components identified  
659 by the PARAFAC analysis. (S1) component 1; (S2) component 2; (S3) component 3; (S4)  
660 component 4.

661 **Figure 8.** Principal component analysis of the physico-chemical and biological parameters.

662 **CE:** electrical conductivity, **OM:** organic matter, **GI:** germination index.

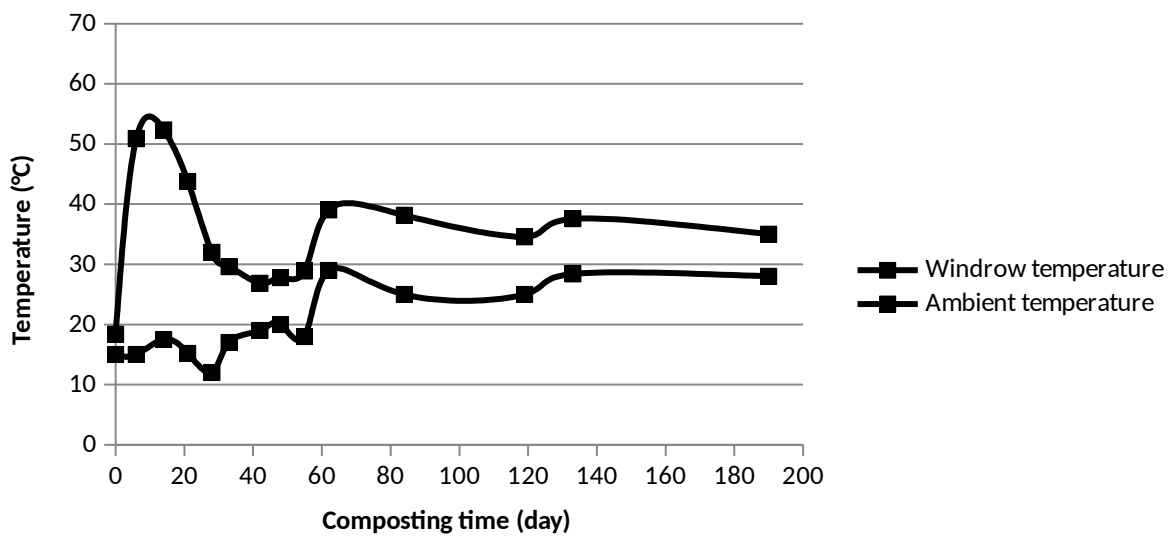
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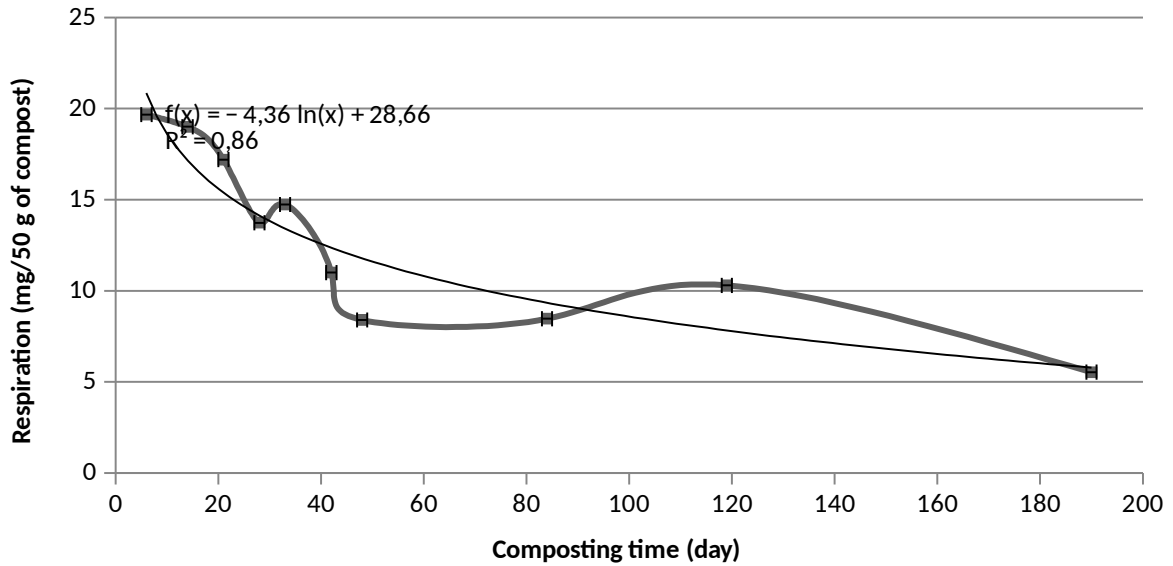


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**Figure 1**

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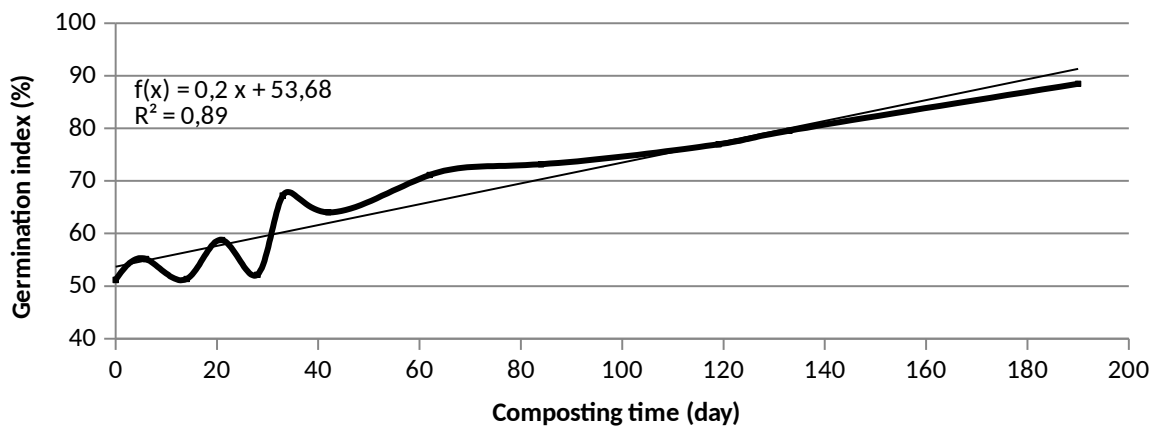
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672 Ne pas mettre la ligne grise, laisser les points expérimentaux non relié. Préciser dans le  
 673 texte que la corrélation n'est pas linéaire entre la respiration et le temps.

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**Figure 2**

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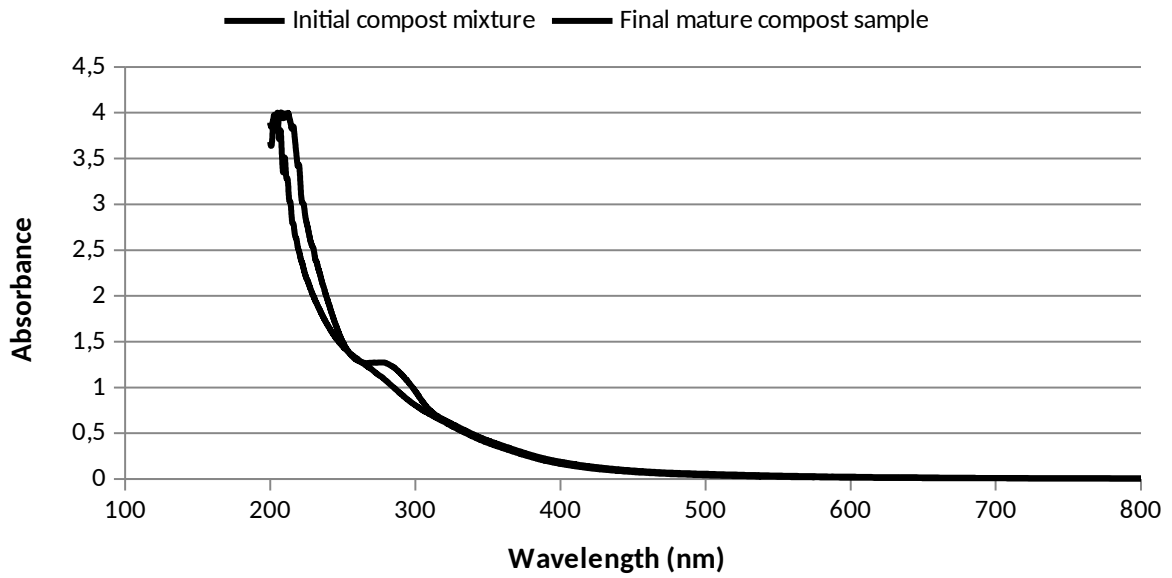


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677 Figure 2 : Mettre les points de mesures sans ligne (ligne noire).

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**Figure 3**



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680 Le pic un peu avant 300 nm pourrait être dû au nitrate en fait !

Figure 4

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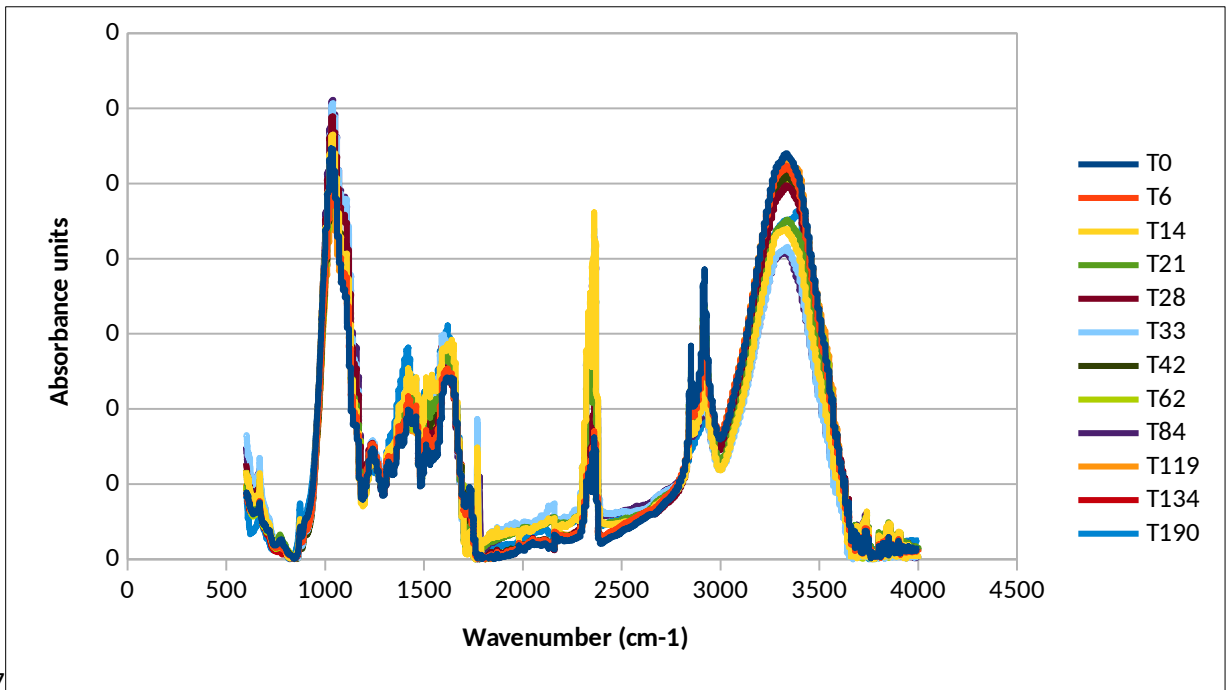
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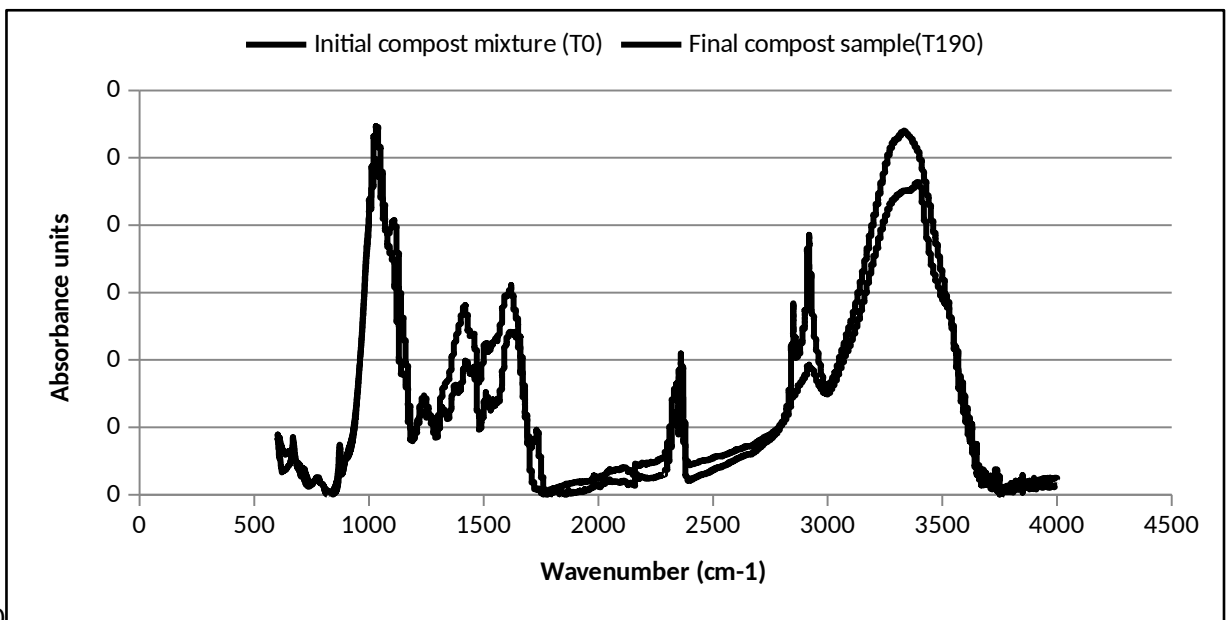
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688A la place de la table 4 (pic IR) je mettrais les formules ou les noms sur le spectre, encore une fois si

689besoin seulement, le texte suffit à lui même je pense.



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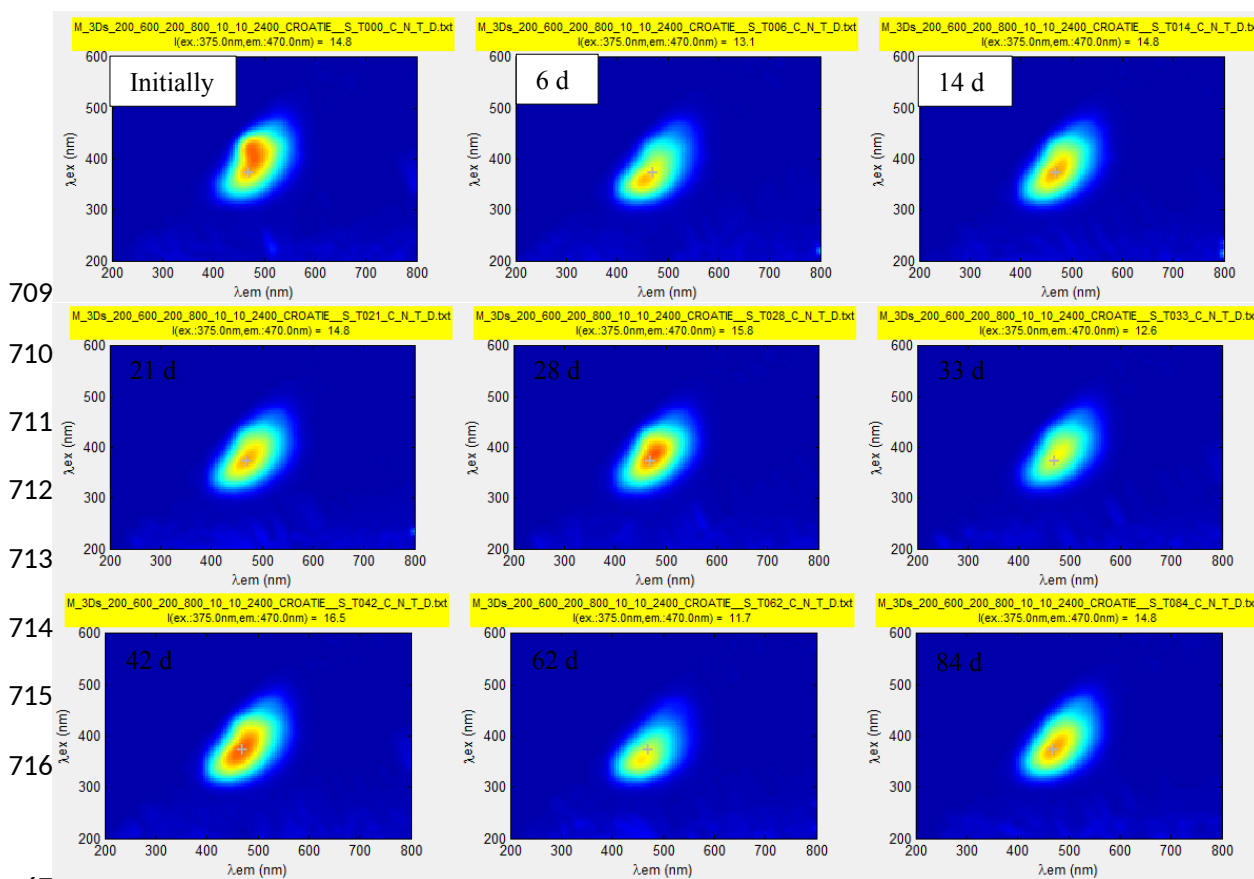
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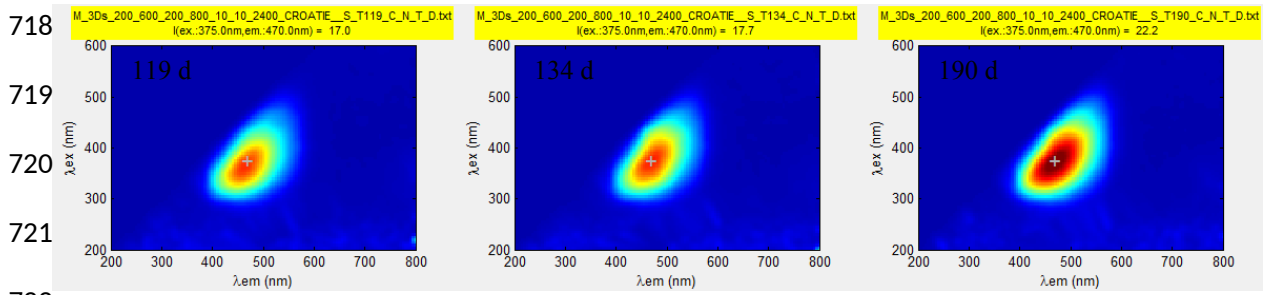
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Figure 5



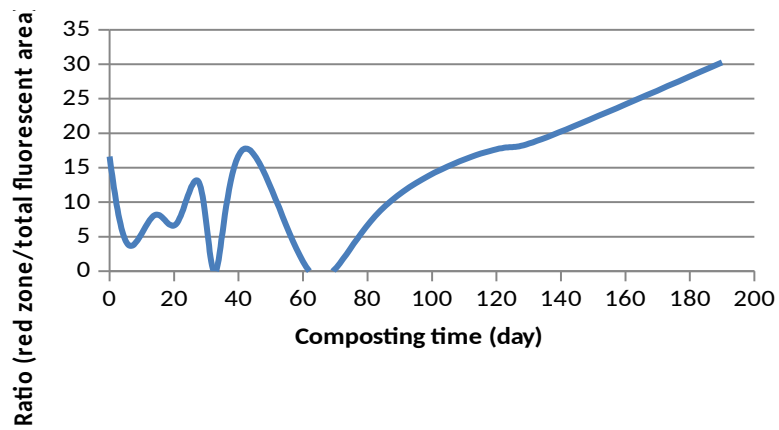
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723(B)

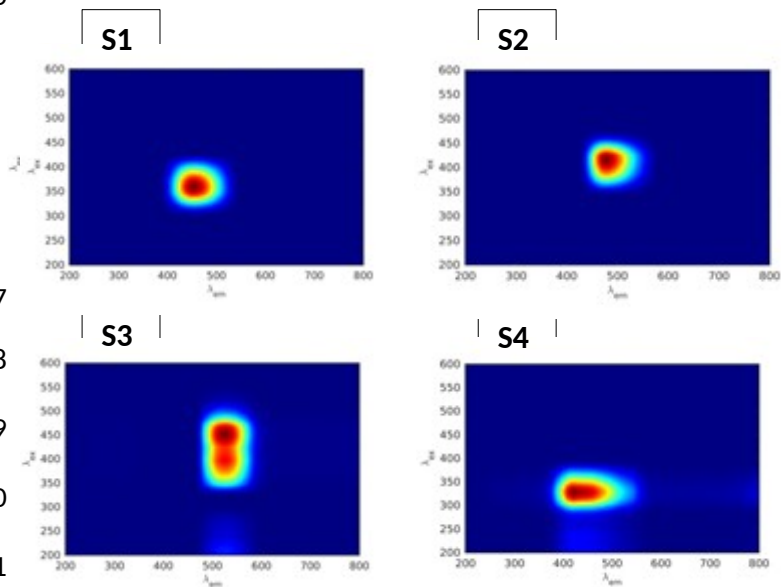


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Figure 6

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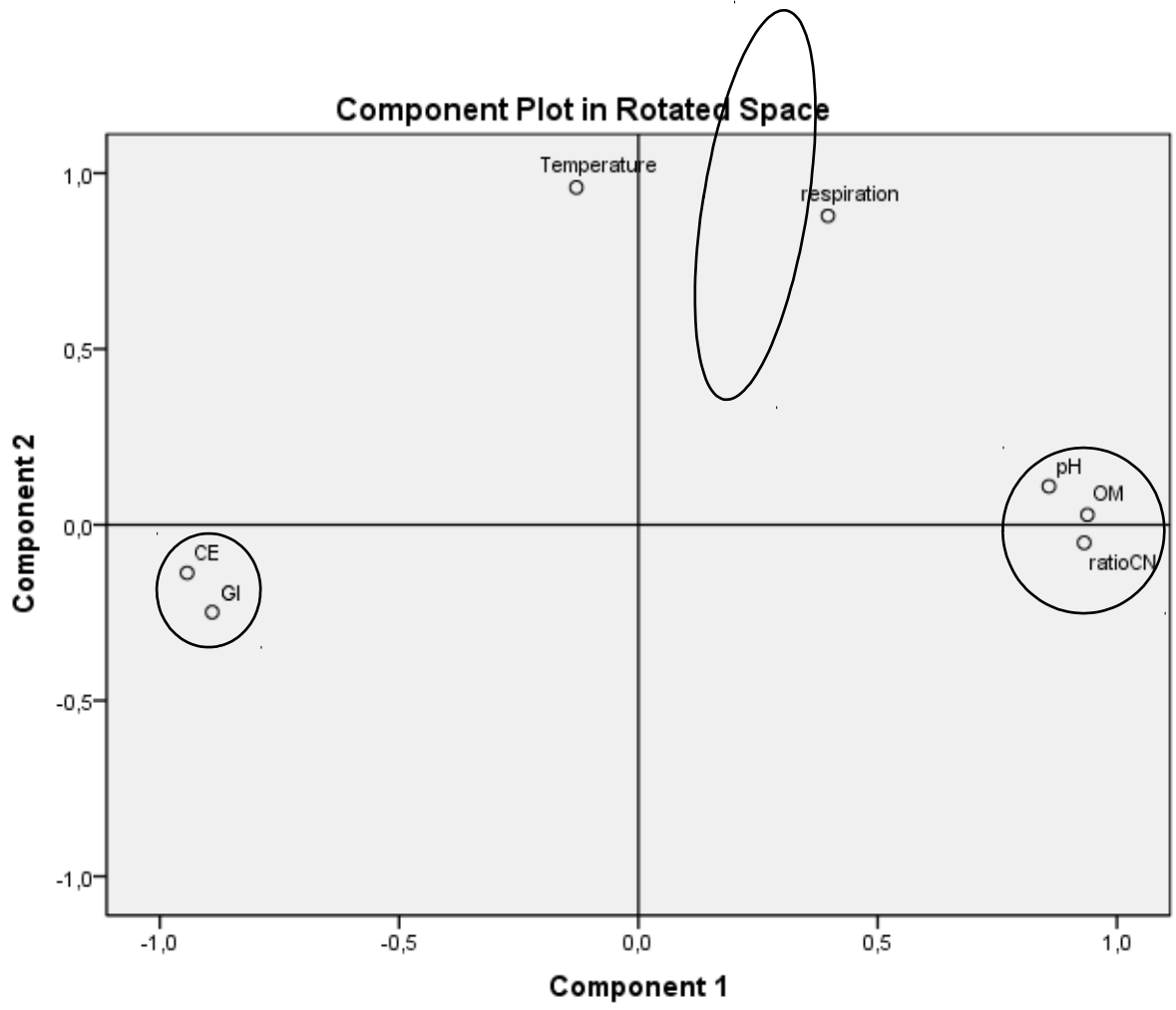
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Figure 7



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Figure 8