4 Evaluation of the stability and maturity of composts based on date palm waste[2] (Boutalbi *et al.*, 2025)

This part of the report largely corresponds to the paper in preparation of Boutalbi *et al* (2025). Only figures and some little part of the text have been modified.

Abstract.

This work aims to assess the stability and maturity of compost and to develop a simple and effective method to improve the compost process produced on-site, as well as to evaluate the degree of maturity of four types of compost based on date palm leaves (PD) prepared from different mixtures: PD + sheep manure (SM), PD + poultry manure (PM), PD + sewage sludge (S) and PD + leachate (L).The results indicate that all composts produced were of good quality and rich in essential nutrients such as nitrogen, potassium, and phosphorus. The analysis of variance for organic matter and organic carbon contents, and C:N ratio showed highly significant differences between the treatments. Among the treatments, SM emerged as the most effective, exhibiting a reduced C_{org}/N ratio (15.56), balanced nutrient levels (K: 70.4 ppm, N: 2.41%, P: 1.08 ppm), and a high germination rate (70–90%). PM had the highest potassium content (98.4 ppm) but displayed lower germination rates, indicating potential phytotoxicity. S, while highly mature (C_{org}/N: 9.83), was limited by a lower potassium content (45.1 ppm). L showed slower organic matter degradation and lower stability. In conclusion, SM is the most suitable treatment for producing a stable, nutrient-balanced compost that meets the requirements of sustainable agriculture.

4.1 Introduction

In arid regions, intensive cultivation leads to a decline in soil fertility, which is characterized by a loss of stable organic matter and an increased vulnerability of plants to nutritional imbalances and diseases. To address this issue, the continuous application of manure is often necessary. However, manure in these areas is not only scarce but also of inconsistent quality (Haddad, 2007). In contrast, oasis environments produce significant quantities of organic by-products, which, when recycled through composting, offer the potential for producing high-quality compost suitable for horticultural use. Composting is an eco- friendly and cost-effective method for treating organic waste, converting it into a stable, mature, and sanitized product. This process eliminates pathogens and weed seeds while producing material rich in humic substances that is easy to store and can be marketed as an organic fertilizer or soil amendment (Chakroune *et al*., 2005; Beran *et al*., 2009).

This study aims to assess the quality of compost produced from date palm residues in combination with various organic additives, such as sheep manure, poultry manure, sewage sludge, and leachate. By examining the physico-chemical properties and biological characteristics of the compost, we seek to develop an optimized composting process that can

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[2] This study is in preparation to be submitted: Boutalbi H, Guimeur K, Sbih M, Merad M, Rikki M. Evaluation of the stability and maturity of composts based on date palm waste.

be easily applied on-site. This research also evaluates the degree of maturity of the different compost treatments to ensure their suitability for sustainable agricultural use.

4.2 Materials and methods

The research was conducted at the Department of Agricultural Science at the University of Biskra, Algeria.

4.2.1 Substrates used

The organic substrates used were derived from plant-based waste (date palm leaves) and animal-based waste (poultry manure, sheep manure), as well as sewage sludge and landfill leachate. Prior to use, these substrates were subjected to grinding to facilitate the biodegradation process during composting.

4.2.2 Conditions for the Experiment

For this study, the chosen composting method was composting in closed barrels. The experiment was conducted in barrels equipped with a forced aeration system. These barrels were placed near a water source and sheltered from direct sunlight and wind. A total of 12 barrels were used, divided into four treatments.

4.2.3 Composition of the mixtures

To maintain the appropriate C_{org}/N ratio in each barrel, the compost was prepared using a blend of raw materials as follows (Table 1):

- SM: 70% date palm leaves(DP) + 30% sheep manure (SM)
- PM: 70% DP + 30% poultry manure(PM)
- S: 70% DP + 30% sewage sludge (S)
- L: 90% DP +10% leachate (L)

Each mixture had a total weight of 4 kg.

4.2.4 Monitoring of the Composting Process

For this 3-months experiment, a total of 20 turnings were performed. Aeration was carried out concurrently with the turnings, while maintaining a moisture content close to 60% for all mixtures. Temperature, pH, and moisture measurements were performed daily until the end of the composting process.

Analysis of the physicochemical parameters such as pH and electrical conductivity (EC) was performed using a conductometer at 25°C, with a matter-to-water ratio of 1:5. EC of the water used was 4.05 mS/cm, which may have contributed to the overall salt concentration in the composting mixture.

The calcination method was employed to determine the organic matter (OM) content, based on the following procedure: Four 1 g samples of the treatment grindings were weighed and placed in porcelain crucibles. The samples were first dried in an oven at 105°C for 24 hours, then transferred to a muffle furnace set to 550°C. The heating process began with moderate heat for two hours, followed by a gradual increase to red heat over a four-hours period.

The organic carbon (C_{org}) content can be deduced from the OM content using the following relationship: $OM\ \% = C_{org}\ \% \times 1.72$.

Total nitrogen was determined by the Kjeldahl method. Phosphorus (P) content was determined by dry-ashing 1 g of compost in a muffle furnace at 550°C for 6 hours. The ash was then dissolved in 25 mL of 1N HCl, filtered, and the phosphorus content was measured colorimetrically. For potassium, 1 g of each substrate was calcined in a muffle furnace (2 hours at 220°C and 6 hours at 550°C), then treated with 2 mL of concentrated HCl. After evaporation, 5 mL of 0.1N HCl was added, and the solution was filtered into a 100 mL volumetric flask. The samples were then analyzed using a flame photometer, after calibrating with the appropriate solutions.

4.2.5 Phytotoxicity test

Phytotoxicity is one of the final indicators used to distinguish between mature and immature compost. To assess the maturity of the composts produced, we examined the effect of the compost's aqueous extract on barley seed germination (Zucconi and Bertoldi, 1987). Compost maturity is determined based on the percentage of seed germination.

4.2.6 Statistical analyses

The data were submitted to variance analysis using the locally licensed version 27 of IBM SPSS software. Data analysis was performed using ANOVA, followed by Tukey's post hoc test at a significance level of 5%.

4.3 Results and discussion

4.3.1 pH and EC

The pH is an important parameter in the composting process, as it helps determine the acidity or alkalinity of the substrate. The pH did not evolve much during composting. It is close to 7 in all treatments and ranged between 7 and 8, the value of mature compost (Larbi, 2006; Moubareck *et al*., 2023).

Figure 8 shows that EC at the beginning of composting was 5.7, 6.8, 6.4, and 6.0 mS/cm for SM, PM, S and L, respectively. It then increased over time, reaching 8.9, 10.6, 9.9, and 8.9 mS/cm for SM, PM, S and L, respectively. The electrical conductivity values of the composts are very high and exceed the threshold value of 5 $mS.cm^{-1}$ suggested by Gondek et al. (2020). The increase in EC values during composting is due to the mineralization of the organic matter in the materials used, which leads to an increase in salt concentration and, consequently, a rise in EC (Larbi, 2006). The high EC of the water used in composting process also lead to increase the EC of the compost.

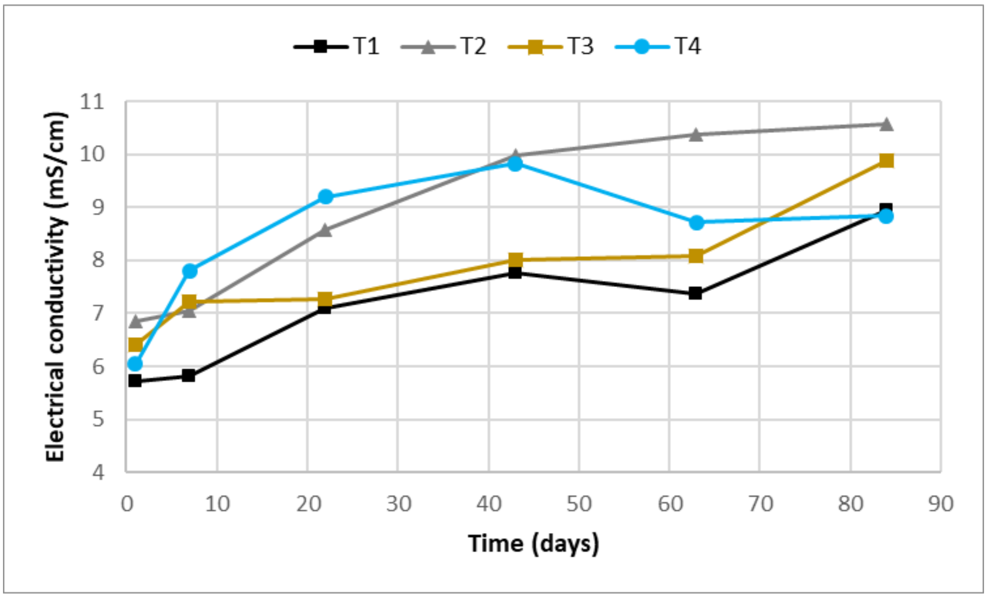

Figure 8 : Evolution of electrical conductivity during composting

4.3.2 Evolution of total nitrogen, phosphorus, and potassium contents between the beginning and the end of composting

The analysis of variance for total nitrogen, phosphorus, and potassium content showed highly significant differences between the treatments (Table 3).

Table 3 :Evolution of the three major element contents during composting process (N, P, K)

	N(%)			P (mg/g)			K (mg/g)		
	Beginning of composting	End of composting	(G)	Beginning of composting	End of composting	(G)	Beginning of composting	End of composting	(G)
T1	0.82 ± 0.03	2.41 ± 0.05	a**	0.01 ± 0.02	0.30 ± 0.02	a**	1.85 ± 4.01	7.04 ± 0.82	a**
T2	1.68 ± 0.16	2.86 ± 0.24	b**	0.02 ± 0.09	0.28 ± 0.01	a**	4.64 ± 2.06	9.84 ± 1.41	b**
T3	1.36 ± 0.06	4.1 ± 0.26	b**	0.03 ± 0.48	0.57 ± 0.21	b**	3.72 ± 1.42	4.51 ± 0.56	a**
T4	0.47 ± 0.05	2.32 ± 0.09	a**	0.02 ± 0.17	0.01± 0.06	a**	2.95 ± 9.07	7.27 ± 0.56	b**

(G)Group. ** highly significant

The initial total nitrogen contents in SM, PM, S and L mixtures were 0.82%, 1.68%, 1.36%, and 0.47%, respectively. After 84 days of composting, these values increased, reaching their highest levels at the end of the process at 2.41%, 2.86%, 4.1%, and 2.32%, respectively. These results fall within the standard range for mature compost (0.5–2.5%) (Wang *et al.*, 2019). The S treatment exhibited the highest total nitrogen content among all treatments.

Phosphorus and potassium levels also increased during the composting process across all treatments. This observation aligns with findings from Wang *et al.* (2019), which reported that composted organic matter lead to increase of nutrient contents.

4.3.3 Organic matter degradation and C_{org}/N evolution

The analysis of variance for organic matter (OM), organic carbon (C_{Org}), and C_{org}/N content showed highly significant differences between the different mixtures (Table 4).

Table 4 : Evolution of the Organic matter, Organic carbon and C_{org}/N during composting process

	OM (%)			C_{org} (%)			C/N		
	Beginning Of Comp	End Of Comp	(G)	Beginning Of Comp	End Of Comp	(G)	Beginning of Comp	End of Comp	(G)
T1	74.9 ± 6.5	64.5 ± 2.35	b**	43.55 ± 3.78	37.50 ± 1.37	b**	53.11 ± 4.01	15.56 ± 0.82	a**
T2	71.4 ± 1.49	52.1 ±3.29	a**	41.51 ± 0.87	30.29 ± 1.92	a**	24.71 ± 2.06	10.59 ± 1.41	a**
T3	80.6 ± 0.62	69.3 ± 0.82	b**	46.86 ± 0.37	40.29 ± 0.48	b**	34.46 ± 1.42	9.83 ± 0.56	a**
T4	85 ± 1.1	79.4 ± 3.99	c**	49.42 ± 0.65	46.16 ± 2.32	c**	105.15 ± 9.07	19.90 ± 0.56	b**

(G) Group. **highly significant

During composting, the decrease of organic matter (OM) content and of C_{org}/N ratio are crucial indicators of the biodegradation and transformation of raw materials. By the end of the process, the OM loss was recorded at 10.4%, 19.3%, 11.3%, and 5.6% for SM, PM, S and L, respectively, demonstrating the bioconversion of raw materials and their oxidation to CO_2. Microorganisms played a key role in decomposing and transforming the OM into stable humic substances, a process evidenced by the observed mass loss (Singh and Kalamdhad, 2015).

In other studies, to assess compost maturity, ratio between final and initial C_{org}/N ratio is calculated. The compost was considered mature when this ratio was below 0.70 (Zucconi *et al.*, 1981). In the present study, the ratio values of the composted substrates were 0.29, 0.42, 0.23, and 0.19 for SM, PM, S and L, respectively, at the end of composting, indicating the initial OM degradation and compost maturity.

4.3.4 Phytotoxicity tests

The results of the variance analysis showed highly significant differences between the four treatments (Figure 9). According to the results, barley germination rates ranged from 75% to 97% for treatments SM, S and L. Lower germination rates were observed for PM, with a 49% germination rate. SM, S and L exhibited the highest germination rates, suggesting low phytotoxicity and good compost maturity. In contrast, PM showed lower germination rates (49%), indicating higher phytotoxicity, likely due to ammonia emissions from poultry manure Tang *et al.*, 2006).

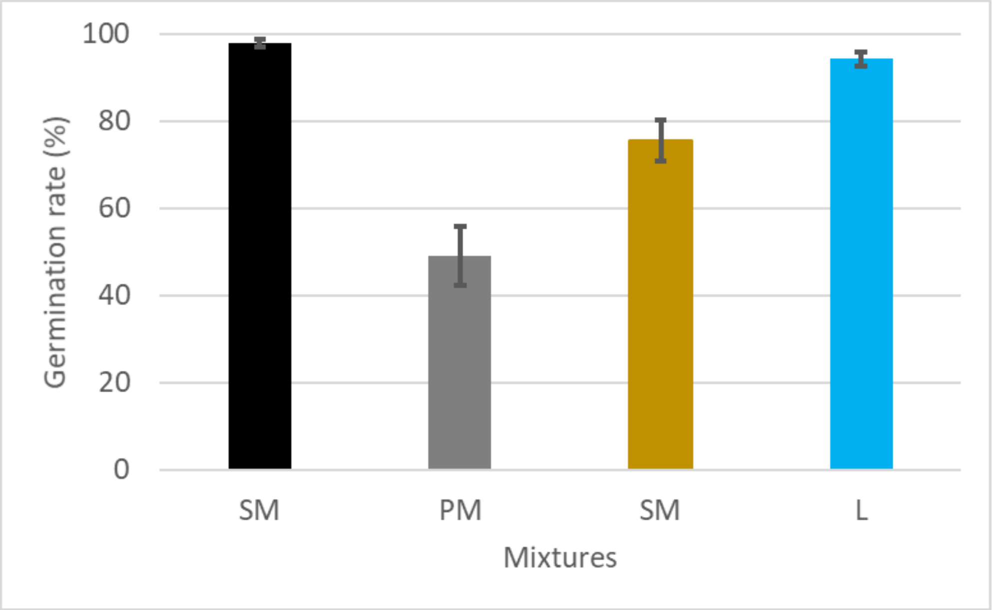

Figure 9 : Germination rate for the different composts produced

4.4 Conclusions

The results obtained after 90 days of composting show that the composts produced from the different mixtures of date palm residues exhibit good quality, characterized by stability, maturity, and richness in essential mineral elements such as nitrogen, phosphorus, and potassium. The physico-chemical analyses and compost maturity tests reveal characteristics favorable for its use as a soil fertilizer. In particular, the mixture with sheep manure (SM) stood out as the most effective, with a reduced C_{Org}/N ratio (15.56), balanced nutrient levels (K: 7.04mg/g, N: 2.41%, P: 0.30 mg/g), and a high germination rate (97%), indicating good stability and maturity of the compost.

The mixture with poultry manure (PM) showed high levels of potassium (9.84 mg/g) and nitrogen (2.86%), but had a lower germination rate (49%), suggesting potential phytotoxicity at this stage. The mixture with sewage sludge, although mature with a low C_{Org}/N ratio (9.83), showed lower potassium content (4.51 mg/g). Finally, the mixture with leachates exhibited slower organic matter degradation and lower stability, limiting its agronomic effectiveness.

In conclusion, the SM compost represents the optimal choice for producing balanced and stable compost suitable for sustainable agriculture. The PM compost, while it may offer advantages in nutrient release, requires optimized management to minimize the risks of phytotoxicity.

4.5 Upcoming research work

According to the results of this first experiment, the treatment with only landfill leachates, less biodegradable, was eliminated. The modalities with the addition of these leachates were selected for the second experiment as it is a source of bacteria. The 4 combinations of organic products selected to improve and accelerate the composting process are therefore: date palm residues and i) sheep manure (30%) and landfill leachates (7%), ii) poultry manure (30%) and landfill leachates (7%), iii) sewage sludge and landfill leachates (7%) and iv) sheep manure (15%), poultry manure (15%) and landfill leachates (7%). For this 2nd experiment, a bioreactor was designed at the University Mohamed khider of Biskra in collaboration with the University of Batna Hadj Lakhder to accelerate the composting process (Figure 10). This bioreactor has been the subject of a patent application filed with the Algerian National Institute of Intellectual Property (INAPI, Appendix 2). The mixture of date palm residues and organic products is stirred with a rotating axis inside the bioreactor. Parameters necessary for biological processes, such as aeration and humidity level, are automatically controlled to optimize the composting process. The analysis of the results of this experiment are currently in progress.

Figure 10 : bioreactor designed for Houda Boutalbi's PhD

5 Characterization of organic amendments and discussion [3]

This part of the report largely corresponds to a part of the doctoral thesis of Le Guyader (2024). Only a few minor changes in form have been made compared to the original manuscript.

5.1 Selected organic amendments

Based on the first studies, only some organic amendments have been deeply characterized.

Three composts from the mixture of date palm residues and sheep manure (2 from Tunisia and 1 from Algeria) were characterized. These are the composts used in field trials on the experimental sites and for pot trials. The Tunisian composts were produced by the Association pour la Sauvegarde de l'Oasis de Chenini (ASOC), Algerian compost was produced by our partner Palm Compost.

Biochar from date palm residues collected in Murcia region was obtained by slow pyrolysis under constant nitrogen flow (without oxygen) at a temperature of 450 °C ± 5 °C at LERMAB (Laboratory for Studies and Research on Wood Materials) in Épinal (France). Pyrolysis time was two hours at 450 °C after pre-heating to 150 °C, with the temperature rising at a rate of 4.9 °C·min-1 up to 450 °C.

The Table 5 shows the abbreviations for each of the OAs.

Table 5 : Abbreviation for the different organic amendments

Organic amendment	**Abbreviation**
Tunisian compost number1	ASOC 1
Tunisian compost number2	ASOC 2
Algerian compost	Palm compost
Biochar	BC

5.2 Analytical techniques used to characterize organic amendments

All the analysis methods used to characterize composts and biochar are summarized in the Table 6.

Table 6 : Summary of measured parameters and analysis methods used to determine the properties of organic amendments

Parameter	**Method**	**Reference**	**Sample analysed**
Mineral matter	Loss on ignition	NF EN 15935	All
C, H, N total contents	Total dried combustion, elemental analysis	NF EN ISO 21663	ASOC 1, BC
Total N content	Kjeldahl	NF ISO 11261	ASOC 2, Palm compost
Organic carbon	Rock-Eval ®	-	ASOC 2, Palm compost, BC

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[3] This part of the report is already published in Le Guyader, 2024. Influence des amendements organiques issus de résidus de palmier dattier sur les propriétés et la fertilité de sols de régions arides et semi-arides. Doctoral thesis, University of Reims Champagne-Ardenne. 269p.

Inorganic carbon	Rock-Eval ®	-	ASOC 2, Palm compost, BC
Total P content	Aqua regia mineralization then ICP dosage	-	ASOC 1, ASOC 2, Palm Compost
Total Ca, K, Mg, Na, Fe, Mn, Zn, Cu contents	Loss on ignition then HNO_3 mineralization and ICP dosage	Adapted from Singh et al. (2017)	ASOC 2, Palm Compost, BC
Total S, Cl contents	X-ray fluorescence (Mining mode)	-	ASOC 2, Palm compost
pH, electrical conductivity	pH meter, conductivity meter	Solid ratio: 1:5 solution for compost and 1:10 for biochar (Singh et al., 2017)	All
Organic Matter Stability Index (OMSI)	Organic fractionation, carbon mineralization at 3 days	FD U44-162 and FD U44-163	ASOC 2, Palm compost
Analysis of surface functional groups	Fourier Transform Infrared Spectroscopy (FTIR)	-	ASOC 2, Palm Compost, BC
Specific area	N_2 Sorption/desorption	Brunauer et al. (1938)	BC
Porosity	Mercury intrusion porosimetry	Brewer et al. (2014)	BC

5.2.1 Physicochemical parameters

The mineral contents of the organic amendments were measured by loss on ignition at 550°C in a muffle furnace for 6 hours (NF EN 15935). The silica sand content was measured in the composts from 5 g dry samples that were calcined. The residue was decarbonated with 10% HCl on a filtration column and the soluble elements were removed by repeated rinsing with milli-Q water. Dry sieving allowed the fraction greater than 50 µm to be collected and quantified.

The total carbon (C), hydrogen (H) and nitrogen (N) contents of biochar and ASOC 1 were measured at the SOCOR company (France, Dechy) by dry combustion using a CHN microanalyzer (NF EN ISO 21663). The total nitrogen contents of ASOC 2 and Palm compost were measured using the Kjeldahl method (NF ISO 11261). The organic and inorganic carbon contents of ASOC 1, ASOC 2, Palm compost and biochar were measured by Rock-Eval 6 (Vinci Technologies, University of Lausanne). The total phosphorus content was measured after mineralization with aqua regia in all composts.

For biochar, ASOC 2 and Palm compost, the concentrations of major elements (K, Mg, Ca, Na), trace elements (Fe, Mn, Zn, Cu) and trace metal elements (Pb, As, Cd, Cr and Ni) were determined by inductively coupled plasma optical emission spectroscopy (ICP-OES). After calcination, 0.25 g of residue was mineralized with aqua regia (3.5 ml HCl 35% + 1.2 ml HNO_3 70% + 7 ml HNO_3 10%), followed by filtration using cellulose acetate filters with a porosity of 0.45 µm. The compost calcination residues were analyzed by X-ray fluorescence (portable Thermo Scientific Niton XL3t 980 Geometrically Optimized Large area Drift Detector GOLDD+ energy-dispersive XRF) to determine their sulfur (S) and chlorine (Cl) concentrations. The element concentrations in the calcination residues were summed with the elements measured in the soluble mineral fraction of the composts.

The pH and electrical conductivity (EC) of the composts were measured in ultrapure water at a solid:solution ratio of 1:5 (ISO 10390:2005). The pH and EC of the biochar were measured in ultrapure water at a solid:solution ratio of 1:10 (Singh et al., 2017). The potential cation exchange capacity (CEC) (pH=7) of the biochar was calculated by adding the extractable cations to 1 mol.L^{-1} ammonium acetate after a 0.05 mol.L^{-1} HCl rinsing phase (Munera-Echeverri et al., 2018).

The anion and cation contents were determined in the soluble mineral fraction of ASOC 2 and Palm compost. The raw products were dried at 40°C and a subsample was used to measure the residual moisture (NF EN 13040). An extraction was made with 5 g of compost in 100 ml of ultrapure water preheated to 100°C. Moderate boiling was maintained for 30 min. The tubes were placed at room temperature, then the supernatant was taken and filtered after cooling. The filtrate for the analysis of SO_4^{2-} and Cl^- anion contents was obtained with cellulose acetate filters with a porosity of 0.45 µm and analyzed immediately by ion chromatography (Dionex ICS1000 model). Samples for cation analysis were filtered in the same manner, acidified with 20 µL 70% HNO3 and stored at 4°C before analysis. Major element concentrations (K, Mg, Ca, Na) in the filtrates were determined by ICP-OES.

Water release measurements of the elements Na, Ca, Mg and K were carried out with the biochar with the same solid:solution ratio (1:20) as for the composts. For this, 10 g of dry biochar were mixed with 200 ml of ultrapure water in 250 ml polypropylene bottles. The tubes were shaken for 4 h and then centrifuged for 30 min at 3500 rpm. A total of five successive rinses were carried out until there was no longer a significant difference in electrical conductivity between two rinses. The cations were measured in the filtrates by ICP-OES as previously indicated.

5.2.2 Characterization of organic matter in organic amendments

5.2.2.1 *Organic matter stability index of composts*

The C_{org}/N ratio is frequently used to predict the humic potential of organic waste products. It is accepted that the higher the C_{org}/N ratio of a product, the slower the degradation of organic matter in the soil and the more stable the humus formed (Allison, 1955; Fog, 1988). However, several studies have shown the limits of the systematic use of this parameter by highlighting rapid decomposition rates for products with high C_{org}/N or slow decomposition for products with a low C_{org}/N ratio (Linères & Djakovitch, 1993; Mary et al., 1993). The estimation of the biodegradability of organic products has therefore been completed by organic fractionation methods.

The Van Soest and Wine (1967) method allows organic matter to be fractionated with reagents of increasing acidity and indirectly quantifies the soluble (SOL), hemicellulose (HEM), cellulose (CEL) and lignin equivalent (LIG) fractions (Linères and Djakovitch, 1993). The lignin fraction is considered to be the fraction most resistant to biodegradability, while the hemicellulose fraction is the most degradable (Baddi et al., 2004). This is not precisely a method for assessing the maturity of composts but it allows us to know their amending value, i.e. their capacity to maintain the stock of organic matter in soils (Sharma, 2006).

Lashermes et al. (2009) conducted a laboratory study on the degradability of 83 organic materials of various types (composts, sludge, manure, etc.). The authors defined the Organic Matter Stability Index (I_{ROC}), calculated based on the biochemical composition of the organic materials (FD U44-162 2016) and the proportion of carbon mineralized after 3 days of incubation (FD U44-163 2018), using the following formula:

$$I_{ROC} = 44{,}5 + 0{,}5\ \mathrm{SOL} - 0{,}2\ \mathrm{CEL} + 0{,}7\ \mathrm{LIG} - 2{,}3\ \mathrm{C}_{3d}$$

with I_{ROC} the proportion of exogeneous organic carbon contributing to soil organic C storage (expressed as g C kg^{-1}) the concentrations of soluble fractions (SOL), cellulose-equivalent (CEL), and lignin-equivalent (LIG) expressed in g OM·kg^{-1}. C_{3d} represents the proportion of organic carbon mineralized after 3 days (as a percentage of the product's carbon).

I_{ROC} index represents the proportion of exogenous organic matter (EOM) contributing to soil organic matter storage. The analyses were conducted on ASOC 2 and Palm compost, in duplicate. Since the biochar had undergone pyrolysis, it was not subjected to the organic fractionation method.

The high presence of soluble inorganic elements in the compost samples required adapting the first stage of organic fractionation of the FD U44-162 standard to obtain the water-soluble organic fraction. The raw products were dried at 40°C and a subsample was used to measure the residual moisture (NF EN 13040). An extraction was carried out in triplicate with 5 g of compost in 100 ml of ultrapure water preheated to 100°C. Moderate boiling was maintained for 30 min. The tubes were placed at room temperature, then the supernatant was collected after cooling. The dissolved organic carbon (DOC) content in the soluble organic fraction was determined after filtration of the supernatant with 0.7 µm porosity glass fiber filters. The equipment used was an autoanalyzer (Aurora Model 1030, O.I Analytical, College Station, TX, USA). The solid residue was calcined by loss on ignition at 480 °C (FDU 44-162) for the measurement of total organic matter of the compost samples in the same test portion.

5.2.2.2 *Thermochemical characterization of organic matter*

Rock-Eval thermal analyses were conducted in triplicate at the University of Lausanne (Rock-Eval 6, Vinci Technologies). This method was used to quantify organic and inorganic carbon in biochar samples and composts. The finely ground samples (<200 µm) were previously desalted with ultrapure water to preserve the operation of the device's thermocouples and ensure the quality of the analyses. One of the advantages of Rock-Eval analyses is the measurement of organic and inorganic carbon in the same test sample since the sample undergoes a pyrolysis phase followed by an oxidation phase (Figure 11). Inorganic carbon is obtained mainly during the oxidation phase with a characteristic peak close to 850°C. The biochar studied is compatible with these analyses since it was produced at a temperature below 650°C, the maximum temperature reached during the Rock-Eval pyrolysis phase.

During the pyrolysis phase, the effluents emitted are measured every second by a flame ionization detector for hydrocarbon compounds (HC) and an infrared detector for CO and CO2. During the oxidation phase, only CO and CO2 are measured with version 6 of Rock-Eval. A total of five thermograms are obtained per sample (Figure 11). The thermograms provide information on the thermochemical stability of the organic compounds. Acquisition parameters were defined by Behar et al. (2001) from the peaks obtained as a function of the pyrolysis and oxidation temperatures, and applied for the first time by Disnar et al. (2003) to characterize the

MOS. The correspondence between the acquisition parameters obtained and the nature of the compounds detected is detailed in Behar et al. (2001).

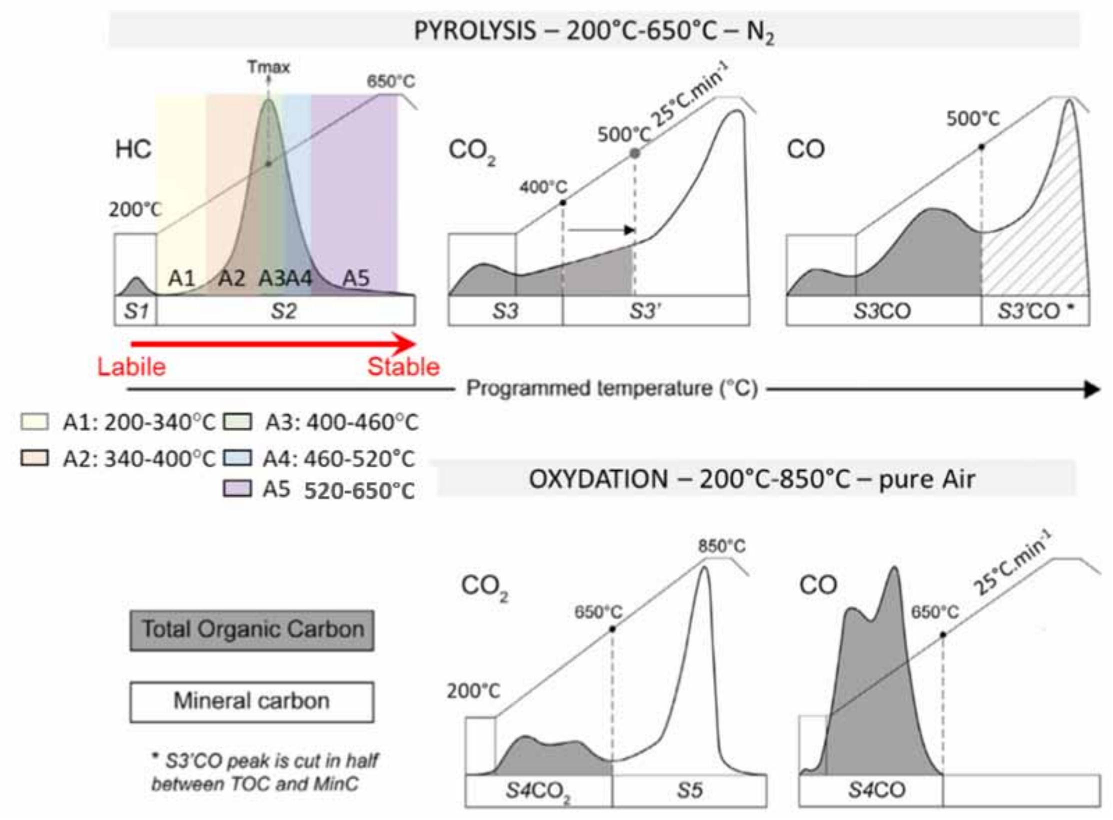

Figure 11 : Principle of Rock-Eval 6 analysis (Le Guyader, 2024, modified from Ducasse et al., 2023).

The R400 index was calculated for organic amendments. It represents the proportion of the S2 peak area integrated below 400°C (Disnar et al., 2003). The area under the curve of hydrocarbon compounds (S2 peak) measured during the pyrolysis phase was decomposed into five pools at the temperature limits defined by Malou et al. (2020): 200−340 °C for A1, 340−400 °C for A2, 400−460 °C for A3, 460−520 °C for A4 and 520−650 °C for A5 (Figure 11). The degree of thermochemical stability of organic matter depends on the cracking temperature of the molecules, with compounds being classified from the most labile (pool A1) to the most refractory (pool A5, Sebag et al., 2016).

From the quantification of the different pools, it is possible to calculate the following indices, defined by Sebag et al. (2016):

$$R = [A3+A4+A5]/100$$

$$I = \log10[(A1+A2)/A3]$$

The R index (refractory index) is linked to the stabilization of organic matter, while the I index (immature index) is linked to its decomposition. The first varies between 0 and 1 and increases with the thermal stability of the OM. The I index varies from -1.5 to 1.5 and a value close to 1.5 indicates the presence of labile, little evolved OM.

5.2.2.3 Characterization of surface functional groups

Biochar and composts were characterized by Fourier Transform Infrared spectroscopy (FTIR spectrometer, Perkin Elmer) in attenuated total reflectance mode in order to characterize the chemical groups present on the surface of the samples. This method does not always allow a quantification of the chemical groups present but it allows their identification. The finely ground samples (<200 µm) were analyzed and the infrared spectra obtained are an average of 16 scans per sample.

5.2.3 Porosity measurement

Porosimetry intrusion method has already been described in the section 2.2.3.

The specific surface area of biochar sample was measured in duplicate by nitrogen (N_2) sorption at 77 K using a Micromeritics ASAP2020. Samples were degassed for 12 h at 350 °C before analysis. The specific surface area was calculated using the Brunauer-Emmett-Teller (BET) method supplemented by Rouquerol correction. This technique characterizes the pore volume of samples in a range of pores from 0.4 to 50 nm in diameter.

5.3 Characteristics of organic amendments

5.3.1 Characteristics of biochar

5.3.1.1 Physicochemical properties

The average mineral content of date palm biochar produced at 450 °C is 15.2 ± 0.6%. The C/N ratio is 114. The H/C ratio is 0.04. For pyrolysis temperatures ≤450 °C, Xiao et al. (2016) reported H/C ratios consistently above 0.4 with biochars from various biomasses. The C/N ratio obtained in our study is therefore low and indicates a high degree of OM condensation for date palm. The biochar is rich in Ca, Mg, K and Na elements (Table 7). The measured values are of the same order as those measured by Usman et al. (2015) with biochars from date palm residues. Biochar does not contain lead (Pb) in significant concentrations and is low in trace elements Fe, Mn and Zn.

Table 7 : Total element content in biochar

Elemental composition	Biochar	replicates
Total carbon (%)	62,5	1
Mineral matter (%)	15,2 ± 0,6	3
Nitrogen (%)	0,55	1
Hydrogen (%)	2,32	1
Calcium (g kg^{-1})	34,2 ±0,9	3
Magnesium (g kg^{-1})	17,4 ±0,8	3
Potassium (g kg^{-1})	39,3 ±0,2	3
Sodium (g kg^{-1})	2,61 ± 0,03	3
Fe (mg kg^{-1})	0,19 ± 0,01	3

Mn (mg kg^{-1})	0,08 ± 0,00	3
Zn (mg kg^{-1})	0,02 ± 0,00	3
Cu (mg kg^{-1})	0,01 ± 0,00	3
Pb (mg kg^{-1})	0,001 ± 0,00	3

Based on cation release measurements in ultrapure water, the soluble fractions of K and Na are approximately 91% and 82%, respectively. The soluble fraction of Ca is lower, on average 28%. These results illustrate the highly soluble behavior of monovalent cations in this biochar.

The biochar has a high pH and electrical conductivity (EC), in agreement with the results of Usman et al. (2015) with biochars from the same biomass. The potential CEC of the biochar is 126 ± 5 cmol.kg-1 (Table 8). This was calculated by summation of the cations extracted with ammonium acetate after pre-treatment under acidifying conditions. Indeed, we observed the ineffectiveness of isopropanol as a rinsing agent for ammonium acetate after the saturation phase of the sorption sites, in agreement with the study of Munera-Echeverri et al. (2018). The authors indicate that the CEC calculated during the last phase which consists of exchanging the NH_4^+ ions with a counterion (K^+) overestimates the CEC by a factor of up to 4. The CEC of date palm biochar is high compared to the literature where potential CEC values up to 201 cmol.kg^{-1} have been reported for biochars (Cheng et al., 2008).

Table 8 : Physico-chemical properties of the biochar (n=3)

Paramètre	**Biochar**
pH (eau)	9,7 ± 0,1
CE (mS.cm^{-1})	7,6 ± 0,3
CEC potentielle (cmol.kg^{-1})	126,4 ± 5,3

5.3.1.2 properties of organic matter

According to the thermogram obtained in the pyrolysis phase with the Rock-Eval method, the area of the S2 peak for biochar is 12.4 ± 1.2 mgHC.g^{-1}. The part of carbon that this represents in relation to the total carbon content of the biochar is low. Indeed, the carbon in biochar is mainly emitted during the oxidation phase at temperatures between 410 and 610°C (Aubertin et al., 2024).

The R400 index, which represents the proportion of the area of the S2 peak integrated below 400°C, is 0.11 ± 0.01 for biochar. The temperature peak (T_{peak}) is on average 539 ± 7°C. This high value is characteristic of coals whose T_{peak} is between 500 and 600°C. These two calculated parameters demonstrate the particularly refractory nature of the organic matter that makes up the biochar studied. It can be noted that the S2 peak of the biochar is truncated because the hydrocarbon compounds are still emitted at 650°C (Figure 12).

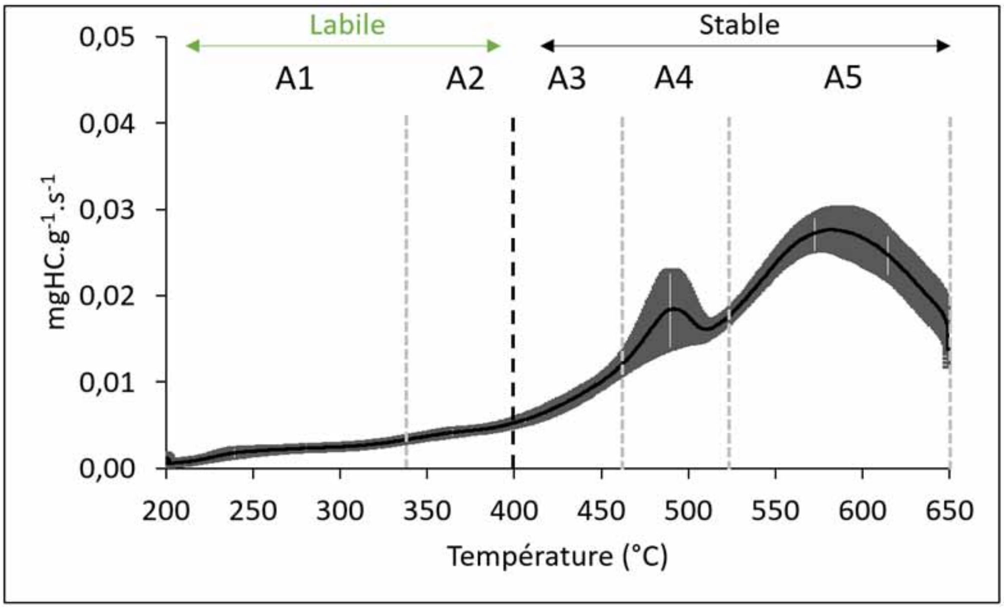

Figure 12 : Thermogram S2 for date palm biochar with temperature limits of pools A1 to A5 defined by Malou et al. (2020). (Le Guyader et al., 2024)

5.3.1.3 Surface functional groups

The obtained IRTF spectrum showed the presence of peaks at wavelengths ranging from 874 to 3320 cm^{-1} (Figure 13). The peak detected at 874 cm^{-1} is linked to the presence of C-H bonds characteristic of aromatic hydrocarbons (C. Zhang et al., 2020). The peaks detected at 1120, 1420 and 1590 cm^{-1}, of high intensities, reflect the high degree of aromaticity of the biochar. The peaks at 1420 and 1590 cm^{-1} also indicate the presence of carboxylic compounds whose origin is probably hemicellulose. The presence of aliphatic groups is visible at the wavelength 2975 cm^{-1}. A high intensity peak was observed around 3320 cm^{-1}, corresponding to the hydroxyl groups (OH bonds). This peak indicates the presence of volatile organic compounds and bound water. The presence of this peak provides information on the hygroscopic behavior of this biochar. Tests involving the application of water droplets to the surface of the biochar confirmed its hydrophilic nature.

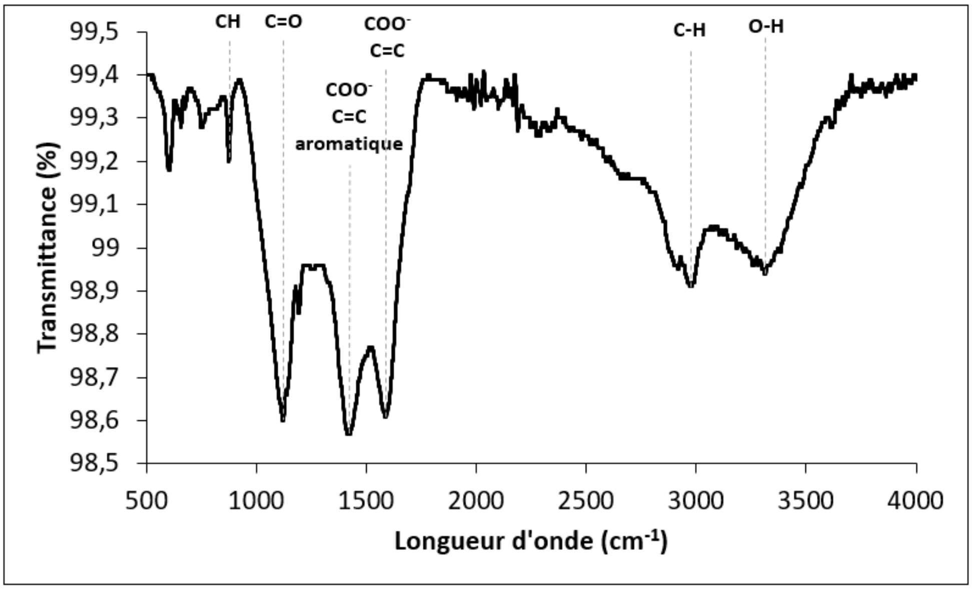

Figure 13 : Fourier Transform Infrared (FTIR) Spectrum of Biochar (Le Guyader et al., 2024)

5.3.1.4 Porosity

The use of mercury porosimetry showed a wide range of pore diameters for the unground biochar, with diameters ranging from 376 µm to 6 nm (Figure 14). The total porosity of the unground biochar was 72.7 ± 3.4%, an increase of approximately 19% compared to the same biomass unpyrolyzed (54.0 ± 2.8%). Pyrolysis resulted in a decrease in the bulk density of the material, initially 0.63 ± 0.04 g.cm^{-3}. The biochar from date palm had a bulk density of 0.37 ± 0.04 g.cm^{-3}. Pyrolysis at 450°C changes the pore size distribution of the material: the volume of pores between 0.2 and 1 µm in diameter increases the most (Figure 15).

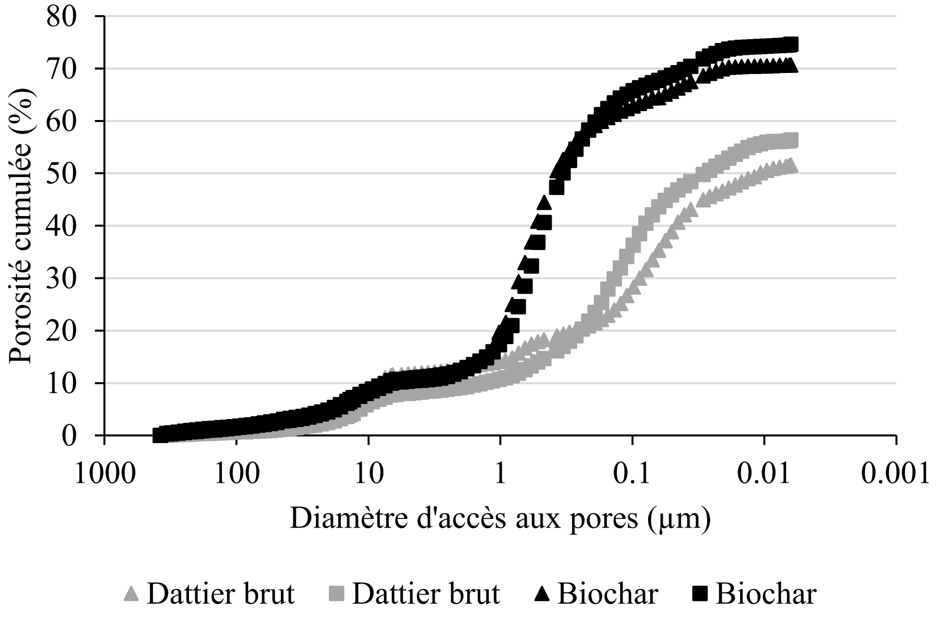

Figure 14 : Cumulative porosity of raw date palm rachis and biochar as a function of pore size determined by mercury intrusion porosimetry. (Le Guyader et al., 2024)

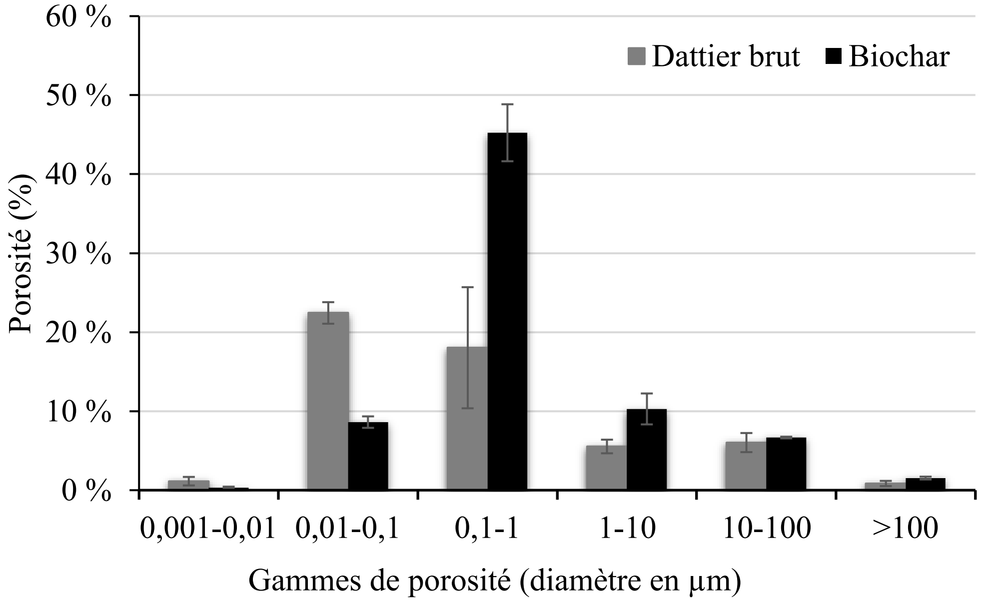

Figure 15 : Distribution of pore size of date palm rachis before and after pyrolysis (Le Guyader et al., 2024)

The results of the dinitrogen sorption measurements showed a very low pore volume of the biochar (0.02 $cm^3.g^{-1}$) in the range of 0.4 to 50 nm and a low specific surface area of 13.5 $m^2.g^{-1}$. These results indicate a very low microporosity of the biochar. This is due to the relatively low pyrolysis temperature of the biochar.

5.3.2 Characteristics of composts

5.3.2.1 Physicochemical properties

ASOC 2 compost is richer in organic carbon than Palm compost (Table 9). ASOC 1 has a low total carbon content. The compost produced by Palm compost is about twice as rich in nitrogen as that of ASOC. The total phosphorus content is about twice as high in ASOC batches. The mineral fraction is the majority in all compost batches. The highest mineral content is for ASOC 1 with a value of 72.7%. ASOC 2 and Palm compost contain about 22 and 13% non-calcareous sands (data not shown), and 12.0 ± 0.5% and 8.5 ± 0.1% soluble elements of mineral origin, respectively (Table 10). They also have high calcareous contents for this type of product. Limestones represent approximately 60% by mass of sediments >50 µm for Palm compost and 24% for ASOC 2 (data not shown). Compared with the results of published work on composts produced from date palm residues, the composts studied are particularly rich in mineral matter. Salt analysis showed very high concentrations of chlorides and sulfates, especially in the ASOC samples. The concentration of water-extractable sodium is approximately three times higher in the Palm compost batch than in the ASOC 2 batch (Table 10). The Palm Compost compost also releases many more divalent cations (Ca, Mg).

The highest concentrations of trace elements (Fe, Mn, Zn) are those of the Palm compost sample (Table 9). The composts have ETM concentrations lower than the limits set by the French standard for composts (NF U44-051).

The pH of the composts is neutral to slightly alkaline depending on the batches. The highest pH is that of Palm compost, which contains very high limestone contents for this type of product (Table 11). The electrical conductivity values of the composts are very high and exceed the threshold value of 5 $mS.cm^{-1}$ suggested by Gondek et al. (2020).

Table 9: Elementary composition of the three composts

Elemental composition	**ASOC 1)**	**ASOC 2)**	**Palm Compost**	**Replicates**
Total carbon (%)	13,3	ND	ND	1
Organic carbon (%)	ND	21,3 ± 3,1	15,6 ± 1,8	3
Total nitrogen (%)	0,97	1,15 ± 0,25	2,44	1 à 4
Hydrogen (%)	1,24	ND	ND	1
Organic matter (%)	26,3	37,3 ± 1,7	33,0 ± 0,3	3
Mineral matter (%)	73,7 ± 0,2	62,7± 1,7	67,0 ± 0,3	3
Total limestone (%)┼	ND	12,9 ± 1,4	28,3 ± 1,6	3

Phosphorus (%)	1,79	2,23	0,97	1
Calcium (g. kg^{-1})	ND	70,4 ± 7,0	164,1 ± 26,3	3
Magnesium (g. kg^{-1})	ND	7,8 ± 1,0	15,4 ± 0,5	3
Potassium (g. kg^{-1})	ND	21,7 ± 1,1	23,4 ± 0,5	3
Sodium (g. kg^{-1})	ND	13,1 ± 1,0	18,8 ± 0,3	3
Chlorine (g. kg^{-1})	ND	57,2 ± 2,7	20,3 ± 0,6	3
Sulfur (g. kg^{-1})	ND	67,9 ± 1,2	21,9 ± 0,6	3
Fe (mg. kg^{-1})	ND	3957 ± 471	11343 ± 1337	3
Mn (mg. kg^{-1})	ND	71 ± 9	311 ± 21	3
Zn (mg. kg^{-1})	ND	63 ± 10	149 ± 11	3
Cu (mg. kg^{-1})	ND	34 ± 4	28 ± 2	3
As (mg. kg^{-1})	ND	1 ± 0	7 ± 7	3
Cd (mg. kg^{-1})	ND	0,1 ± 0,0	0,5 ± 0,1	3
Cr (mg. kg^{-1})	ND	7 ± 1	15 ± 1	3
Ni (mg. kg^{-1})	ND	4 ± 0	9 ± 2	3
Pb (mg. kg^{-1})	ND	5 ± 0	9 ± 2	3

$^{+}$mesured by Rock-Eval analyses
ND : not determined

Table 10: Concentrations of water-extractable elements (n=3).

Samples	**Concentrations (mg. kg^{-1} DM)**						**Soluble mineral fraction (% DM)**
	Ca	**Mg**	**K**	**Na**	**Cl$^-$**	**S**	
ASOC 2	1406 ± 15	331 ± 5	15413 ± 58	5434 ± 12	47854 ± 2630	16478 ± 696	12,0 ± 0,5
Palm compost	9907 ± 89	3719 ± 32	14694 ± 253	16845 ± 335	18162 ± 539	7190 ± 271	8,5 ± 0,1

Table 11: pH and electrical conductivity (EC) of composts

Parameter	**ASOC 1**	**ASOC 2**	**Palm Compost**
pH (water)	7,0 ±0,1	7,4 ±0,1	7,6 ±0,1
EC (mS.cm^{-1})	9,2 ±0,3	20,2 ±1,3	17,6 ±0,3

5.3.2.2 organic matter stability index

The soluble organic fraction (SOL) is high in Palm compost and ASOC 2 composts, and represents the dominant organic fraction for ASOC 2. According to part 5.2.2.1 on the limits of application of the organic fractionation method of the FD U 44-168 standard, the calculation of the I_{ROC} cannot be calculated correctly in our compost samples due to the presence of carbonates and salts in high concentrations. The results were therefore not interpreted here because the I_{ROC} values obtained are high due to the overestimation of the soluble fraction. This concerns less than 1% of organic products according to the FD U 44-168 standard.

The results of carbon mineralization according to the FD U44-163 standard are presented in order to estimate the biological stability of the OM of the composts. The share of mineralized organic carbon after 3 days of incubation is higher for ASOC 2 compost with 1.9 ± 0.0% on average compared to 1.2 ± 0.1% for Palm compost. This indicates a higher degradability of ASOC 2 compost.

The hot water soluble organic fraction is presented for both composts in Table 12. The percentage of dissolved organic matter (DOM) relative to total OM is higher on average for Palm compost than ASOC 2. This result confirms the observation of the much browner color of the filtrates for Palm compost (Figure 16). The soluble fraction includes a wide variety of organic compounds: nitrogen molecules, polysaccharides, and lipids (Peltre et al., 2010). It is generally considered to be composed of labile compounds and should therefore decrease during composting (Thuriès et al., 2002).

Table 12. Soluble organic fraction of composts measured after extraction with hot water (n=3).

Soluble organic fraction	**ASOC 2**	**Palm compost**
gC. kg^{-1} DM (dry matter)	6,3 ± 0,2	16,9 ± 0,5
*DOM / Total OM (%)	3,4 ±0,2	10,2 ± 0,4

*DOM = DOC × 2

Figure 16. Filtrates after extraction of composts with hot water. From left to right: CT=ASOC 2; CA=Palm compost. (Le Guyader et al., 2024)

5.3.2.3 Thermochemical characteristics of composts

According to the Figure 17, the hydrocarbon compounds of ASOC 2 compost are mainly emitted at temperatures below 400°C. This provides information on the particularly labile nature of the OM that composes it, if we refer to the R400 index which amounts to 0.73 ± 0.02 for the ASOC batch. The batch produced by Palm compost has an R400 index of 0.58 ± 0.01. Thus, the relative proportion of thermochemically stable OM is higher in the Palm compost batch.

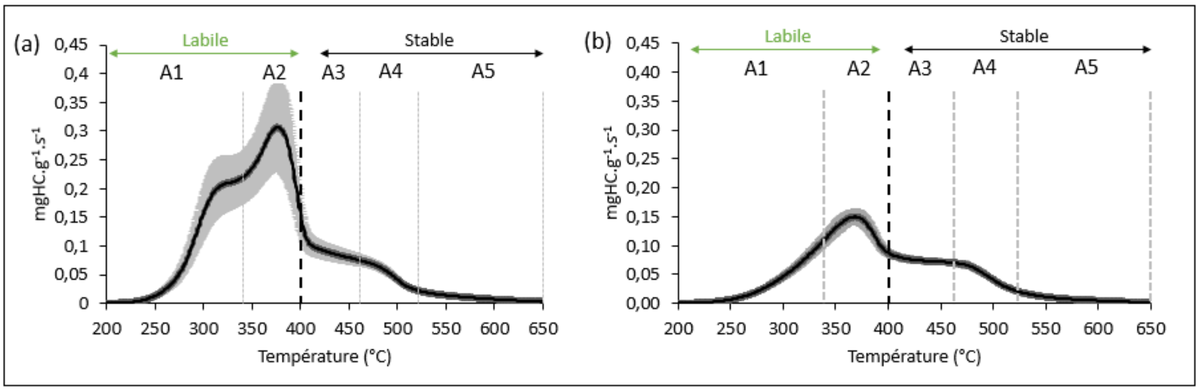

Figure 17 : S2 thermograms for composts produced by (a) ASOC 2 and (b) Palm compost with the temperature limits of pools A1 to A5 defined by Malou et al. (2020) (Le Guyader et al., 2024)

Figure 17 plots the compost samples in a graph representing the I index, related to the decomposition of organic matter, as a function of the R index, related to its stabilization. Palm compost is consistent with existing literature, with values very similar to those of compost obtained from food and wood waste (Ducasse et al., 2023). In contrast, ASOC compost has a thermal signature characteristic of fresh or poorly decomposed organic matter. The R indices of ASOC 2 samples are lower than that of a mixture of sewage sludge, green waste and pine bark after four days of composting (Albrecht et al., 2015).

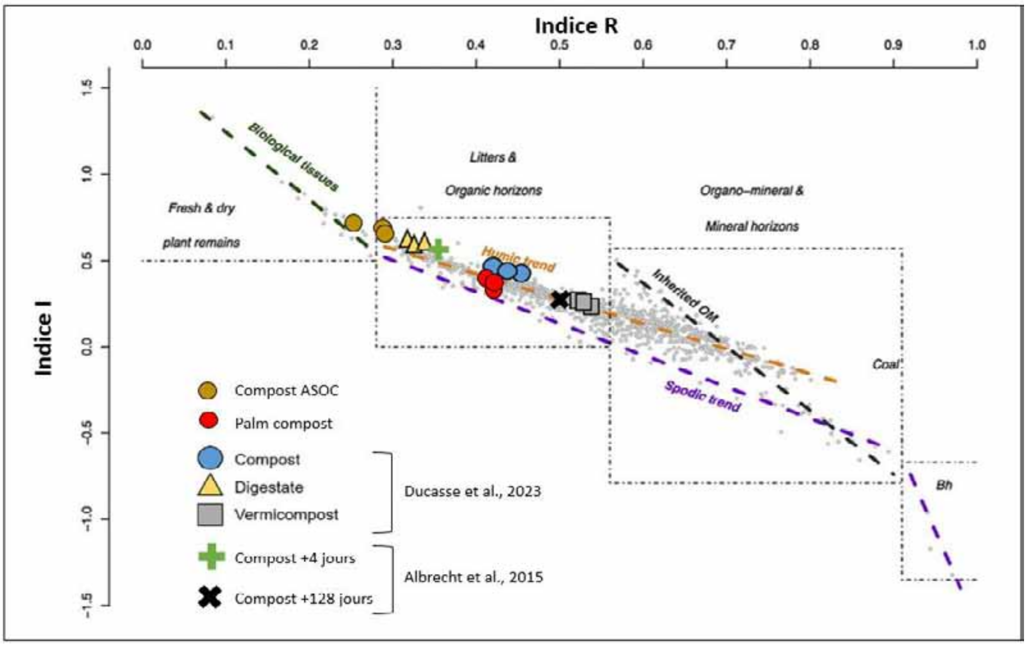

Figure 18 : I and R indices diagram for comparison of thermal signature of date palm compost with organic products from literature (adapted from Sebag et al, 2016) (Le Guyader et al., 2024)

5.3.2.4 Surface functional groups

The spectrum obtained showed the presence of peaks at wavelengths ranging from 797 to 3400 cm^{-1}. Several peaks at wavelengths are similar between the composts but are of variable intensities. A peak at 797 cm^{-1}, characteristic of aromatic compounds, is observed only for Palm compost. The intensity of the three peaks identified between 1084 and 1637 cm^{-1} is higher in the Palm compost sample. This reflects a higher degree of aromaticity compared to the ASOC compost. The peak observed around 1418-1459 cm^{-1} also indicates the presence of carboxylic compounds, the origin of which is probably hemicellulose. Carbonates, whose signal is obtained around 1350–1550 cm^{-1} (Grunenwald et al., 2014), could also have contributed to the signal obtained in this wavelength range since they were measured in significant concentrations in the two composts studied.

5.4 Conclusion

The composition of organic amendments is summarized in Figure 19. The amount of OM in the composts is higher in ASOC 2 compared to Palm compost and ASOC 1. From the results of carbon mineralization after three days of incubation and the characterization of the thermal signature of the OM, the stability of the OM is higher in the batch produced by Palm compost. Limestone and sand represent important fractions in the composts. In alkaline soils, these mineral fractions should not have negative effects on soil fertility but they can limit the effect of composts that are relatively low in OM.

Biochar contains about 85% OM. The mineral fraction consists of ash including a water-soluble fraction that represents about 5% of the dry matter (DM) of the biochar (Figure 19). The results of date palm biochar porosity measurement indicated high total porosity in uncrushed samples, while microporosity and macroporosity of this material were low.

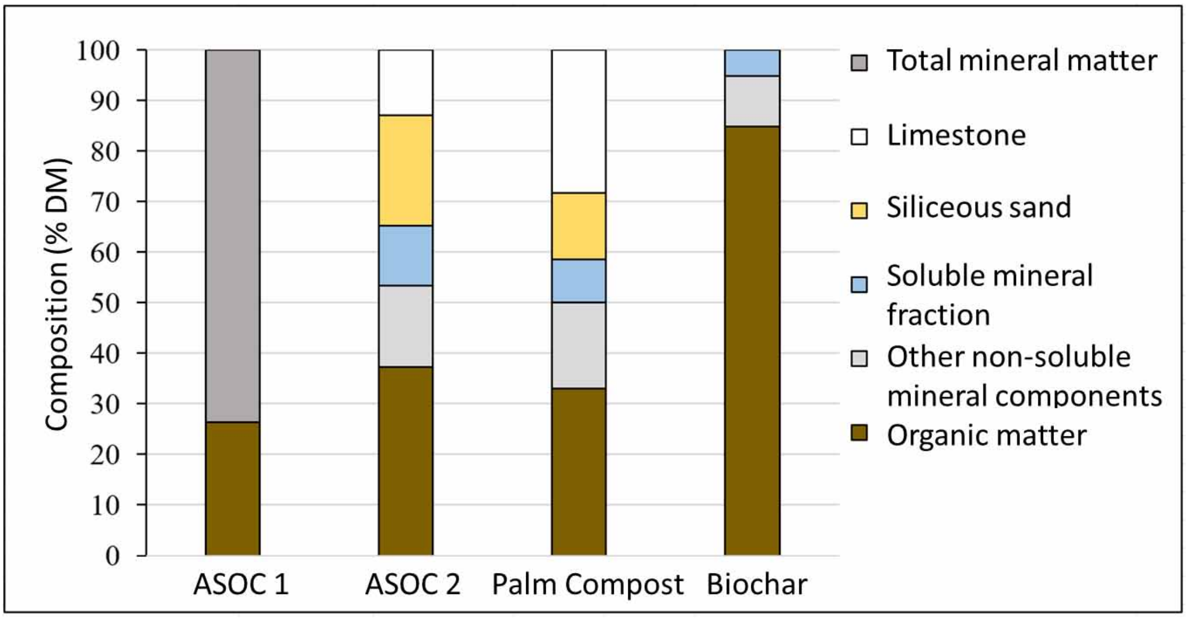

Figure 19 : Organic and mineral composition of organic amendments (Le Guyader et al., 2024)