Algerian Journal of Environmental Science and Technology
Avriledition. Vol.2. N°2. (2016)
ISSN : 2437-1114
www.aljest.webs.com

Valorization of by-products from the olive oil extraction: evolution of chemical and spectroscopic characteristics during composting

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ARTICLE INFO

Article History:

Received **: 30/01/2016**
Accepted **:29/06/2016**

Key Words:

olive-products,
composting,
valorization.

ABSTRACT/RESUME

Abstract:*Compost plays a central role in organic soil fertility. Composting is considered a viable solution for olive mill waste. The objective of this work was to study the composting of olive pomace in order to obtain reusable compost in organic farming. Four different windrows were prepared by mixing olive pomace with different agro-industrial by-products, which were used either as building agents or as N sources. The main process parameters (temperature, pH, organic carbon) were monitored over four months to ascertain the maturity of the compost. Fonctionnel groups of compost were investigated with FT-IR spectroscopy. All windrows reached temperatures of 60°C indicating good sanitation composts The initial acidic pH of 5.53 to 6.13 reached adequate pH values of 6.6 to 8 after 120 days. The decrease in carbon content is lower than expected because high content of lignin in initial compounds. IF spectra windrows presented strong similarities. The massif around 3440 cm $^{-1}$ and pic of 2924 cm^{-1}disappears after 120 days. It would be a degradation of holocellulose.*

I. Introduction

Algeria is one of the main Mediterranean countries where the climate is more conducive to the cultivation of olive trees. It is positioned after Spain, Italy, Greece, Tunisia and Morocco, which are the largest olive oil producers [1].Olive cultivation has changed dramatically in recent decades. This led to; firstly,getting virgin olive oils of high organoleptic quality.Secondly, obtaining co-products of good quality(olive mill wastewater (OMW), olive pomace,leaves, prunings), this allow to direct research towards biotechnology valuation of these raw materials [2].The rejection of the effluents of industries producing olive oil such as dry pomace(oil mill in three phases), the wet pomace (oil mill in two phases) and OMW pose an environmental major problem, particularly in the countries of Mediterranean Basin. These highly polluted discharges cause serious damage to the environment. The OMW from these mills do not undergo any treatment and are often discharged into waste water sewers, stored in evaporation ponds and/or applied directly on the soil without control.This results in a negative impact on the environment that translates to the clogging of soil, pollution of surface water and groundwater and release of noxious odors [3]. The agricultural use of these by-products is one way to restore to the soil nutrients exported annually by the olive harvest[4,5 and 6].However the return to field directly out of these raw residues of olive crushing chains cannot be considered because of their wealth in non-stabilized organic matter and their content of phytotoxic and antimicrobial substances (phenols, fatty acids and organic acids) [7]. The composting of these co-products of the olive industry is a double profit solution. On the one hand discharges of these pollutants in the environment are reduced. On the other hand, these composts can be used as organic fertilizers that help improve agricultural soils deficient in organic matter and nutrients. The soils in Algeria are often deficient in organic matter. Composts from olive pomace can prove to be a deposit in carbon, humic acids and interesting nutritional element for the rehabilitation of degraded lands. The objective of this work is to study the composting of olive pomace in order to obtain reusable compost in organic farming.

I1.Materials and Methods

II.2.Composting and Sampling Method

The chemical characteristics of windrows components and the composting devices are summarized in Tables 1I and 2.

The composting process used was a windrow composting. Each windrow was trapezoidal form with an initial height of 1 m, 1.5 m long and 1.5 m wide. The volume of each windrow was 2.25 m^3. The windrows were willing to air, on a well-prepared plot: terraced, groomed and covered with a black tarp to prevent pollution of soil and groundwater by leachate. The windrows were made from by-products of olive growing in mixture (tab. II). After the implementation of all windrows, they have been covered with a black tarp to protect from rain. They watered with municipal water whenever their moisture was between 40 and 60%.Only windrow 3 has been sprayed with OMW (210 l) and then watering has been continued with water. We conducted one rollover per week during the thermophilic phase to limit the increase of temperature of the windrows. Indeed, the thermophilic phase coincided with the increase in atmospheric temperatures during the cooling phase, the windrows were submitted to a reversal every two weeks. Sampling is carried out by taking both East and West cardinal points, three heights; 0 - 0.3m, 0.6m 0.3, 0.6-1m and this had 4 stages of composting. The samples were taken at the initial stage to 40 days, 80 days and 120 days. The temperature of each windrow was measured during the whole composting process by a probe thermometer.

Table 1. *Some chemical characteristics of the components of the 4 windrows.*

	Pomace II phases	Pomace III phases	Wood	Grass	Cattle manure
pH	5,1	6,1	/	/	8,2
C%	48,6	48,6	49,8	46,6	24,5
N%	1,21	1,21	0,71	1,55	1,22
OM%	97,1	97,2	99,5	93,1	49,0
C/N	40	40	70	30	20

Table 2. *Experimental composting design.*

	Windrows			
	Windrow 1	**Windrow 2**	**Windrow 3**	**Windrow 4**
Olive pomace II phases (%)	75	50	25	0
Olive pomace IIIphases (%)	0	0	0	25
Grass (%)	0	0	75	75
Wood (%)	25	25	0	0
Cattle manure (%)	0	25	0	0
Primary humidification (L)	Water	Water	Water +OMW	Water
Number of watering	12	12	14	14
Volume of water used (L)	360	360	180	420
Volume of OMW used (L)	0	0	210	
Number of reversals	9	10	10	9

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II.2.Analysis Methods

The windrow samples at different composting stages were submitted to measurement with ratio compost/water of 1: 10. Measurements of pH were performed before each reversal during the composting operation. The rate of carbon content was calculated by loss on ignition of dry matter after drying at 105 ° C.A carbon proportion factor corresponding to the ratio M.O / C of2 was applied. Infrared spectrometry with Fourier transform (FTIR) was performed using an infrared spectrophotometer Fourier transform model SHIMADZU FTIR-8400 controlled by a computer provided with a processor with a resolution of 4 cm^{-1}.Pellets of KBr and sample mixture with a proportion of 100 mg and 1 mg respectively, were analyzed.

III.Results and Discussion

III.1. Composting Process

III.1.1.Temperature

Temperatures within the swath were measured along with the composting, the temperature profiles are shown in Figure 1.

These findings identify three different phases for four windrows succeeding over time, called mesophilic, thermophilic and cooling stage. The observed mesophilic phase was of variable duration for 4 windrows. It was 4 to 5 weeks for the windrow 1 and 2 to 10 weeks for windrows 3 and 4. The reversal of eachcompost after 3 weeks improved the increase in temperature (Figure 1). All the windrows have reached their thermophilic phase at temperatures above 60 ° C (Figure 1). Comparable maximum temperature values (50–60 _C) were found in composting asolid fraction of OMW with olive leaves [8] and different organic matter inputs (cotton gin waste and sewage sludge) [9, 10] reported that a temperature above 55 ° C allows the compost hygienisation by destroying pathogens. According to these authors, a temperature between 45 and 55 ° C promotes the biodegradation and between 35 and 40 ° C, it improves the diversity of microorganisms. It is interesting to note that the windrow 1, 2 have reached temperatures of about 65 ° C after 4 weeks, which indicates that the activity in these windrows is more intense compared to windrows 3 and 4.

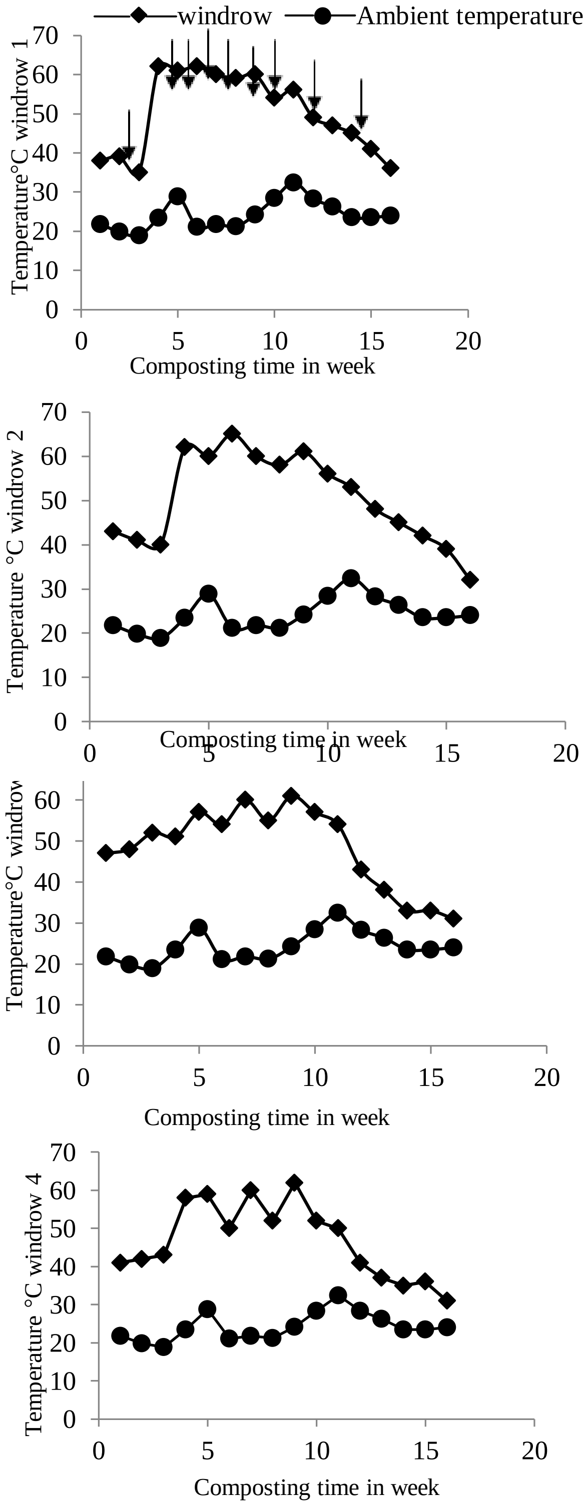

Figure 1.*Temperature profiles during composting (arrows indicates windrows reversals).*

III.1.2. Biodegradation of organic matter

The initial concentration of organic matter (OM) was approximately (tab.III). For all windrows an organic material loss is observed after 120 days. It varies from 24.9 to 32.9%. This reduction in the carbon content was due to the degradation and mineralization of organic matter by microorganisms. This decomposition rate remains low compared to the results obtained by [11]. In fact, these authors obtained after 120 days of composting, a loss of OM in the range of 32-72%, but in their case the structuring is not the olive pomace rich in lignin. In addition, the lipids contained in the pomace and OMW slow down or delay the decomposition of organic matter by making access more difficult for decomposers [9]. These small reductions of OM rates,combined with a long cooling period was mainly due to the lignin concentration of the pomace and comminuted olive branches. The carbon content decreased more rapidly in windrow rich of grass (windrows 3 and 4) compared to those richer in lignin (windrows 1 and 2). In the case of these windrows, composting time may be longer in order to obtain a better decomposition of the initial organic material.

Table 3. *Parameters of the Composting Process.*

Windrows	**Compostingdays**	**pH**	**OM %**
	0	5,07	97,75
	40	5,53	87,03
	80	6,67	92,84
1	120	7,4	81,3
	0	5,83	85,05
	40	6,13	74,05
	80	7,41	84,66
2	120	7,17	73,05
	0	5,07	94,2
	40	5,59	92,76
	80	7,53	84
3	120	8,05	77,66
	0	6,15	94,11
	40	5,51	96,3
	80	7,21	87
4	120	6,54	81,23

III.1.3. Variation of pH

Measurements of pH performed while composting were recorded at the table III.The pH was a good indicator of the state of progression of composting [12]. The pH of the windrow 4 remained acidic after 40 days of composting.Indeed, as the degradation of organic matter, bacteria and fungi release organic acids that can accumulate during the mesophilic phase of composting [11]. The pH of the windrows 2, 3 and 4 reached neutrality after 80 days. The pH of the windrow 4 after reaching neutrality (7.21) at 80 days decreased to the value of 6.54 at 120 days. The increase of the pH of windrow 1 was progressive and reached neutrality after 120 days.The increase in pH at the stage 80 days for all the windrows would be due to the release of NH_3 during the decomposition of the grass for windrows 3, 4, to the addition of urea for the windrow 1, to decomposing manure for windrow 2. In addition, the degradation of acid residue in compost induced an increase in pH. This is consistent with the findings of [12]. The pH stability in stage 80 days in the windrow 2 was assigned to the buffering effect of manure on the acidity of the compost. It was interesting to note that watering windrow 3 with the OMW, acid effluent (pH=5) did not cause a decrease in pH of the windrow. Right from the stage 80days, the pH of the windrow is in the vicinity of neutrality;

Algerian Journal of Environmental Science and Technology
Avriledition. Vol.2. N°2. (2016)
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this latter is close to the compost pH considered ripe for upgrading. Right from the stage 120 days, all of the windrows have pH between 6 and 8 corresponding to mature windrows. This increase in the pH was observed by [13,14 and 15] during the composting of waste of the industry of olive. [13,16] made the assumption that the ions ammonium NH4+ released during the process also would contribute to the increase in the pH.It would be necessary to follow the pH of the window 4, since a decrease in pH of up to an acid pH was observed from the stade 120 days (tab.III). After 120 days of composting the pH of all windrows have increased and were between 6.19 and 8.13 which corresponds to pH good quality compost.

III.2. Infrared-Spectrometry

III.2.1. Composition of composts

Infrared spectra recorded between 3500 and 500 cm^{-1}samples are shown in Figure 2. The spectra obtained were very similar with regard to the general shape and positions of the main peaks. The spectra consist of a broad band around 3440 cm^{-1}. It would be hydroxyl (OH) of the hemicellulose, cellulose, lignin. The band located to 2924 cm^{-1} can be attributed to -CH, - and CH_2 - CH_3 of holocellulose and lignin. The area between (1650 - 1600 cm^{-1}) corresponds to C = C bond of the carbonyl. Bands between 1000-600 cm^{-1} can be assigned to -C-C of aromatic.

III.2.2.Evolution during the composting

The massif located in the vicinity of 3440 $cm^{-1}$and the band at 2924cm^{-1}disappeared after 120 days. It would be holocellulose degradation which may indicate a degradation of the original organic matter during composting. The carbonyl band (1650-1600cm^{-1}) decreases transmission intensity during composting, which informs that the concentration has increased. The same reasoning may be used for aromatics.

IV.Conclusion

Characterizing the evolution by the composting of the olive industry by-products in mixture with other types of organic material enabled us to emphasize (i) the existence of temperatures around 65 ° C which correspond to those of good hygienisation composts (ii) a cooling phase of 40 days was not yet complete.

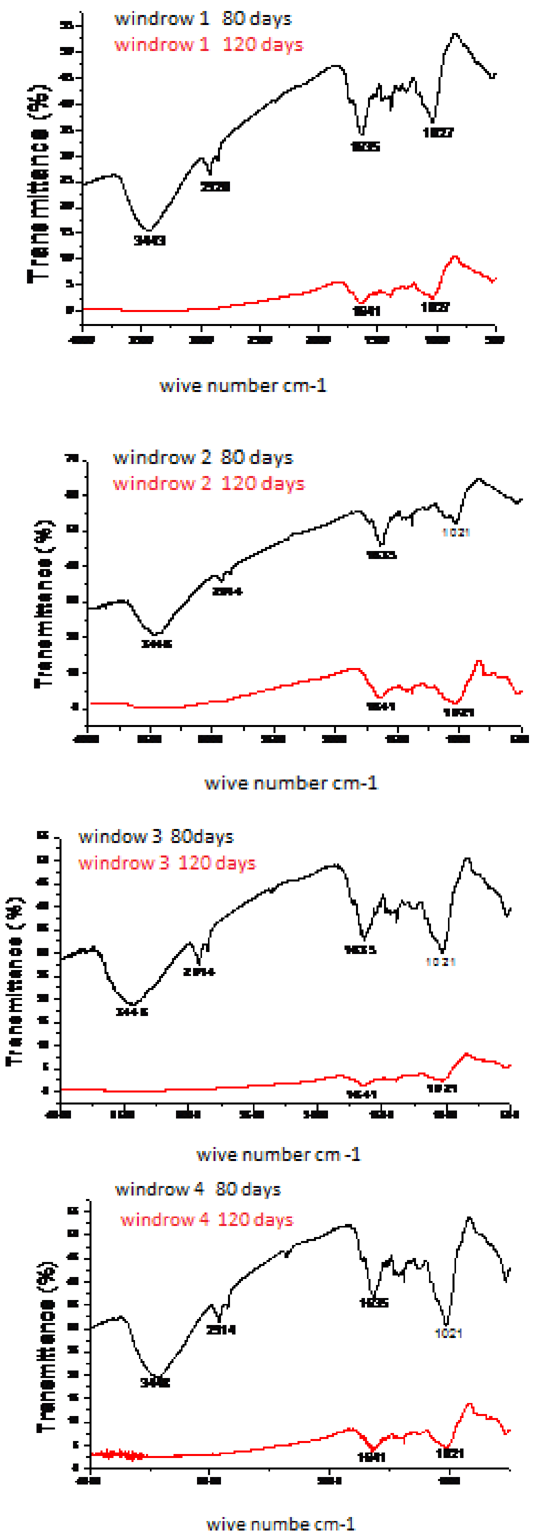

Figure 2.*FTIR spectra of windrows after 80 and 120 days of composting.*

The results of our experiments showed that the pH was acid for 4 windrows during the initial phase, after 120 days of composting the pH of all windrows have increased to the set values between 6.54 and 8.05 which corresponds to pH of good quality compost. The uses of Fourier transform infrared spectroscopy, to determine a degradation of the initial organic material. It would represent the holocellulose degradation in 120 days. These preliminary results showed that composting in olive products is sustainable. Indeed, these composts are valued in agriculture and can overcome the scarcity of organic amendments in Algeria in a context of generally deficient soils in organic carbon.

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Acknowledgement

We thank the CFPA Mechtras (Tizi Ouzou) for having provided us with a composting area. We thank M.M. Rahmoune for his help in interpreting the infrared spectra and Bellal A. for revision of text in English

Please cite this Article as:
Mouas Bourbia S.,Kana S., Mahmoudi K., Amokrane K., Louni D., Lardjane N., BoudiafNait Kaci M., Issaoun D., Yahiaoui Tibiche G., *Valorization of by-products from the olive oil extraction: evolution of chemical and spectroscopic characteristics during composting*. ***Algerian J. Env. Sc. Technology**, **2**:**2** **(2016) 20-25**