Olive mill wastewater and wine by-products valorization by co-composting

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Abstract
Composting is a process transforming the organic matter by an aerobic biological process, allowing the organic matter degradation and stabilization. The resulting product is the compost, a soil organic amendment. The study aims, on the one hand, the waste valorization as well as the development of the green waste and agro-industrial discharges: the olive mill wastewater and the wine by-products, by co-composting, and on the other hand, the quality assessment of the produced compost through aerated window. Furthermore, the progress of the physico-chemical parameters (pH, electrical conductivity, temperature, C/N ratio) was achieved during all the process. The biological system stability was noticed at approximately two months. Consequently, the final product was neutral and rich in fertilizing elements. Its heavy metal content was within the standard required limits and didn’t represent any phytotoxic effect.

1. Introduction
The regular growth of the population and the change of production and consumption patterns led to an important production of the solid waste. Over time, these harmful wastes are accumulated and need efficient solutions to be treated or recycled after their discharge. In Morocco, the total solid waste generation is 6.852 million tons, including the current production of household production of 5.5 million tons per year [1]. The industrial sector generates more than 1.2 million tons which 289.385 tons of hazardous waste [1-2]. Around of the two third of this waste (67%) are produced by the food-processing industry [2].

The green wastes as well as the food-processing industries discharged wastes are generally organic waste, their composition is suitable for microbial biodegradation, that may lead to humic substances.

The present work intends to valorize the green waste and the agro industrial discharged wastes: olive mill wastewater, and wine by-products, by process aerobic biodegradation: Co-composting method, to produce stable compost which is rich in its fertilizing elements.

A number of parameters are monitored throughout the progress of the co-composting process to determine the quality of our compost obtained: (i) The physico-chemical parameters for the control of the process (pH, Electrical Conductivity, Organic Matter, C /N ratio) (ii) Compost quality monitoring parameters (Phytotoxicity Test, Fertilizing Elements, Heavy Metals).

2. Materials And Methods
2.1. Composting procedure
The olive mill wastewater, the grape marc and green waste were mixed. The olive mill wastewater was collected from the storage tank of a modern oil mill located at Meknassa Ben Ali (8km from Taza-Morocco) and the the
grape marc by-product come from the company of Celliers Meknes company (Meknes-Morocco). The green waste was issued from Taza’s markets, consisting mainly from fruit and vegetable residues. The mixture (600 kg) was arranged in a windrow (0.6 m high by 1.2 m diameter base and 2 m length). During the composting process the windrow were watered with the olive mill wastewater. This operation was carried out while turning, frequency of once every three days during the beginning of the composting, then once every 7 days and finally once a 15 days. The compost was developed aerobically.

2.2. Physico-chemical characterization

The characterization of the substrates was determined by measuring pH, electrical conductivity (EC), moisture (M), organic matter (OM), mineral matter (MM), total nitrogen Kjeldahl (TNK) and C/N ratio. Electrical conductivity and pH were measured in a 1:5 (w/v) water soluble extraction [3], using the multiparameter consort C535. The moisture content was assessed by drying at 105°C for 24 h and organic matter (OM) by determining the loss-on ignition at 550°C for 4 h. The organic carbon (OC) was calculated using the following equation:

\[ \text{CO} (\%) = \frac{\% \text{MO}}{1.724} \]

The total nitrogen Kjeldhal (TNK) by the Kjeldhal method. The determination of heavy metals and oligo-elements was performed to assess the quality of the compost using ICP-AES method (Inductively coupled plasma atomic emission spectroscopy).

2.3. Test phytotoxicity

The compost phytotoxicity was determined by evaluating its aqueous extract on seed germination. The germination tests were carried out on 10 seeds of tomato and cress experimented at different dilutions in petri dishes including filter paper soaked by the compost extract. The tests were conducted in the dark at room temperature (25 °C) during 72 hours [4-5]. Three repetitions were performed.

The germination index (GI) was determined considering to the number of sprouts and root growth using equation (1) [5]:

\[ \text{GI} (\%) = \frac{\text{GB}}{\text{GT}} \times \frac{\text{LB}}{\text{LT}} \times 100 \] (1)

Where:
GB, the number of germinated seeds in the case of aqueous extract; GT, the number of germinated seeds in the case of the control where the distilled water was used; LB: Root length on compost extract; LT: Root length control.

Inhibition’s percentage of germination (IG) was calculated according to equation (2) [6]:

\[ \text{IG} (\%) = \frac{(\% \text{Gc} - \% \text{Gtr})}{\% \text{Gc}} \times 100 \] (2)

Where: %Gc, the germination percentage of the control, %Gtr, the germination percentage of the lot treated with the compost extract.

The vigor index (VGI) was giving by the equation (3):

\[ \text{VGI} = \frac{\% \text{G} \times (\text{Average } \text{RL} + \text{Average } \text{HL})}{100} \] (3)

Where: %G: the germination percentage, RL: the root length (cm), HL: the hypocotyls length (cm).

3. Results And Discussion

3.1. Characterization of the substrates used in co-composting

The results of the substrates physicochemical characterizations are presented in Table 1. The grape marc is a solid residues generated during wine making process, it has a neutral pH, rich in organic matter and have a C/N ratio of 42.06. The Olive mill wastewater is an acidic effluent, characterized by high mineral content and low nitrogen rate. The green waste, characterized by high nitrogen rate and low C/N ratio compared to grape marc and olive mill wastewater.

3.2. Physico-chemical parameters’ evolution during the composting process

3.2.1. Temperature and pH’s evolution

Figure 1, representing the evolution of the temperature and the pH during the co-composting process shows that the curve is composed of three phases: a mesophilic phase, a thermophilic phase and maturation phase. The Firt phase is characterized by heat raise temperature to 47.5 ° C observed in the first days; showing a rapid colonization of mesophilic microbial populations. This phase is accompanied by a slight acidification of windrow due to the release and accumulation of molecules of organic acids produced by the first colonizers in anaerobic conditions established at the beginning of the composting process [7]. The second phase shows a
temperature rise to 68 °C and pH to 8. This is the result of a strong microbial activity generated by the presence of readily biodegradable organic matter [8].

Tableau 1: Physico-chemical characterization of the substrates.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Grape marc</th>
<th>Green waste</th>
<th>Olive mill wastewater</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>7.41±0.02</td>
<td>5.88±0.02</td>
<td>5.42±0.05</td>
</tr>
<tr>
<td>EC (mS.Cm⁻¹)</td>
<td>4.81±0.03</td>
<td>1.63±0.02</td>
<td>8.02±0.02</td>
</tr>
<tr>
<td>Moisture %</td>
<td>72.42±0.9</td>
<td>36.98±1.2</td>
<td>22.66±1.35</td>
</tr>
<tr>
<td>OM %</td>
<td>69.80±1.75</td>
<td>64.84±1.2</td>
<td>74.74±1.05</td>
</tr>
<tr>
<td>MM %</td>
<td>30.19±1.25</td>
<td>35.15±1.12</td>
<td>25.25±0.98</td>
</tr>
<tr>
<td>OC %</td>
<td>40.49±0.96</td>
<td>37.61±1.02</td>
<td>43.36±0.98</td>
</tr>
<tr>
<td>TNK %</td>
<td>0.96±0.09</td>
<td>1.52±0.06</td>
<td>0.86±0.12</td>
</tr>
<tr>
<td>C/N ratio</td>
<td>42.18±2.36</td>
<td>24.74±1.19</td>
<td>50.42±1.7</td>
</tr>
</tbody>
</table>

These high temperatures may result in the reduction of pathogens and weed seeds [9]. As for the pH increase, it is explained by a process of ammoniation and ammonia production from the degradation of the amines (proteins, nitrogenous bases ...) [10]; the concentration of Ca²⁺ can also be the cause of this alkalizing [7].

The final phase, after the sixth week, the temperature gradually decrease in windrows with a pH stabilization until neutralization revealing power humus buffer during the maturation phase [11]. This phenomenon is the result of a slowdown in the activity of microorganisms due to the depletion of easily degradable organic matter [12]. This results is in line with findings related to bioconversion of wastes from the olive oil [13] and Compost of poultry manure and olive mill wastes [14].

Figure 1: Evolution of the temperature and the pH according to time during the process of the co-composting.

3.2.2. Electrical conductivity and C/N ratio’s evolution

Figure 2, represents the variation of electrical conductivity and the C/N ratio according to time during the co-composting process; it show an increase in the electrical conductivity during the first two weeks explained by the mineralization of the organic material during the co-composting process. A progressive decrease in the electrical conductivity is then observed showing the result of a loss of salts by leaching and decreased extractability salts due to their greater binding to the stabilized organic matter [15-16]. This result is proved by the decrease in the C/N ratio according to time up to 12 indicating stability of mature compost which shows the biodegradation of organic matter resulting in the lowering of total carbon levels due to mineralization of the organic material accompanied by a production of CO₂ associated with the increase of the nitrogen concentration [7]. This results correlates with the experiments about composting [17-18].

3.3. Phytotoxicity test

3.3.1. The germination index

Figure 3 illustrates the effect of the extract of the compost on the cress germination and tomato. The results observed showed that the germination index, for a dose of 25% compost extract was 88% for cress, and 115 for tomato. 87.87 and 63.47 germination indexes are respectively observed at a dose of 50 to 75% among cress and 106 % and 103% for the same doses in tomato. In contrast, a low index of germination is obtained for 100% compost; 41% for tomatoes and cress to 55%. However, the highest germination indices were observed for a
dose of 25, 50 and 75%. These results indicate that, first, the germination rate varied with the dose of compost made, and second with the type of culture.

![Graph 1](image1.png)

**Figure 2:** Evolution of the electrical conductivity and the C/N ratio according to time during the co-composting process.

![Graph 2](image2.png)

**Figure 3:** Effect of the extract of the compost on germination of tomato and cress at different dilutions.

3.3.2. Germination inhibition rate

Figure 4 demonstrates the effect of inhibition of compost juice on the cress germination and tomato. The results obtained indicate that at a dose of 25% the compost juice has no effect on the inhibition of germination for both cultures.

![Graph 3](image3.png)

**Figure 4:** Germination inhibition rate of tomato and cress at different dilutions of the extract of compost.
Dilutions of 75 and 100% of compost extract have an inhibitory effect on the germination percentages with respectively 11.6 and 38.4% for the tomato. In the cress the inhibitory effect is observed for doses 50, 75 and 100% with respectively percentages of 8.5, 41.5 and 36.7%. This could be the result of the high conductivity of the extract of the compost [19].

3.3.3. Vigor index
Referring to Figure 5 showing the vigor index of tomato and cress, we notice that this parameter decreases with increasing concentration of compost juice. This decrease is related to the germination and the length of roots and hypocotyls. Indeed, the higher ratios are observed for the dilution of 25% compost extract in two crops: 11.3 and 5.6 respectively. However the vigor index of tomato is almost double the cress.

![Figure 5: Vigor Index of tomato and cress at different dilutions of the extract of compost.](image)

The overall results show that germination indexes above 50 for the tomato to the dilutions 25, 50, 75% and cress to dilutions of 25, 50, 75 and 100% compost juice. Therefore, the compost in question has no phytotoxic effect, these results indicate that the depressive effect of compost is not only related to the immaturity of compost but also provided doses of extract of compost and also with the type of Culture. This is consistent with the results found by Abad Berjon and Camparé et al. [8-20].

3.4. Characterization of mature compost
The compost produced characterization is mentioned in Table 2.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Values</th>
<th>Normes NFU44-051 (limits)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture %</td>
<td>30±1.2</td>
<td>ND</td>
</tr>
<tr>
<td>pH</td>
<td>7.02±0.01</td>
<td>ND</td>
</tr>
<tr>
<td>Electric Conductivity (mS.cm-1)</td>
<td>1.75±0.01</td>
<td>ND</td>
</tr>
<tr>
<td>C/N ratio</td>
<td>12±1.23</td>
<td>&gt;8</td>
</tr>
<tr>
<td>Minerals and fertilizing elements (mg/kg)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>856</td>
<td>ND</td>
</tr>
<tr>
<td>K</td>
<td>2763.56</td>
<td>ND</td>
</tr>
<tr>
<td>Mg</td>
<td>897.68</td>
<td>ND</td>
</tr>
<tr>
<td>Ca</td>
<td>10153.95</td>
<td>ND</td>
</tr>
<tr>
<td>Fe</td>
<td>5415.84</td>
<td>ND</td>
</tr>
<tr>
<td>Na</td>
<td>225.67</td>
<td>ND</td>
</tr>
<tr>
<td>Mn</td>
<td>849.65</td>
<td>ND</td>
</tr>
<tr>
<td>Heavy metals (mg/kg)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>As</td>
<td>&lt;0.01</td>
<td>18</td>
</tr>
<tr>
<td>Cd</td>
<td>1.5</td>
<td>3</td>
</tr>
<tr>
<td>Ni</td>
<td>&lt;0.01</td>
<td>2</td>
</tr>
<tr>
<td>Cr</td>
<td>9.35</td>
<td>12</td>
</tr>
<tr>
<td>Cu</td>
<td>22.26</td>
<td>300</td>
</tr>
<tr>
<td>Zn</td>
<td>57.83</td>
<td>600</td>
</tr>
<tr>
<td>Se</td>
<td>&lt;0.01</td>
<td>12</td>
</tr>
</tbody>
</table>
The compost in question is neutral, rich in fertilizing and mineral elements (Ca, Mg, Fe, Mn and Na). These results are lower than those mentioned by Compaoré et al. [8] and Temgoua et al. [21]. This difference is probably due to the nature of composted waste.

On the other hand, compared to the standard NFU44-051 [22], compost has low contents of heavy metals. Although they are below the limit values, there is reason to fear a risk of accumulation of these trace elements following a regular spreading of the compost in the fields [8].

Conclusions
This study was conducted in order to contribute to the valorization of olive mill wastewater and under–wine products in the composting stream. Compost was made from three substrates: olive mill wastewater, grape marc and green waste.

The evaluation of the quality of the compost prepared was carried out by monitoring physical and chemical parameters according to time and its maturation. The stability of the biological system was found at the end of approximately two months. The final product complies with an organic amendment (NFU44-051). Composting can be an effective solution to reduce the harmful effects of tested waste.

This work strongly contributes to the reduction of environmental pollution by solid and liquid waste as well as protection of natural resources and human health.

References

(2017) ; http://www.jmaterenvironsci.com /