Sludge stabilization using ozonation: a pre-treatment method for composting waste activated sludge

Zahra Asadgol\textsuperscript{a,b}, Mohammad Mahdi Emamjomeh\textsuperscript{c}, Ali Esrafily\textsuperscript{a,b}, Mitra Gholami\textsuperscript{a,b}, Mahdi Farzadkia\textsuperscript{a,b,*}

\textsuperscript{a}Research Center for Environmental Health Technology, Iran University of Medical Sciences, Tehran, Iran, Tel. +989122588677; +982188607945; email: farzadkia.m@iums.ac.ir (M. Farzadkia), Tel. +989127468322; email: Asadgol.z@iums.ac.ir (Z. Asadgol), Tel. +989124976672; email: a.esrafili@iums.ac.ir (A. Esrafily), Tel. +989123906308; email: gholamim@iums.ac.ir (M. Gholami)

\textsuperscript{b} Department of Environmental Health Engineering, School of Public Health, Iran University of Medical Sciences, Tehran, Iran

\textsuperscript{c} Social Determinants of Health Research Center, Research Institute for Prevention of Non-Communicable Diseases, Qazvin University of Medical Sciences, Qazvin, Iran, Tel. +989126216141; email: m_emamjomeh@yahoo.com

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Abstract

The effects of ozonation have been studied as a pre-treatment of waste activated sludge (WAS) on duration of the composting process. This study was aimed to evaluate the comparison of two pre-treatment processes on degradation of the WAS and particularly on the duration of pre-treated sludge composting. The ozone was injected by two different doses to make semi-stabilized sludge (0.006 g O\textsubscript{3}/gTSS) and completely stabilized sludge (0.041 g O\textsubscript{3}/gTSS). Three mixtures were composted in 150 L three reactors using the raw (CRS), semi-stabilized (CSS) completely stabilized sludge (CCS). The organic matter evolution was assessed by characteristic of the total organic carbon, total Kjeldahl nitrogen, total phosphorous, volatile solid and total coliforms during composting. The results show that CCS reactor reached a considerable enhancement in quality of compost which could be a consequence of higher organic matter mineralization during the process of composting compared with the other two reactors. The results show that ozonation as an oxidative pre-treatment could solubilize the organic matters and could increase the biodegradability of WAS which led to rapid mineralization rate of organic matter during composting.

Keywords: Sludge stabilization; Ozonation; Composting; Waste activated sludge

1. Introduction

Increasing population, urbanization and anthropogenic activities led to an extreme increase in wastewater production and consequently biological excess sludge in the recent decades. Treatment and disposal of enormous amounts of excess sludge are one of the most critical environmental issues in worldwide [1,2] such that, up to 60\%, of the operating cost of wastewater treatment plants (WWTPs) was dedicated to associated processing of sludge. Therefore, the interest of applying methods with effective sludge reduction and minimization of the treatment loads with the lowest expenses are going to increase [3,4].

Composting is an aerobic process used for sludge treatment which augment the microbiological activity and leads to biodegradation of decomposable organic matters, recycling of nutrients, formation of relatively inert humic substances and pathogen-free product that is often used as a bio-fertilizer for land application. During the composting, two phases can be distinct: active phase, where insoluble...
organic matter decomposed into soluble small molecular substances by extracellular enzyme of microorganisms. This phase may progress in high temperature which, therefore, constitutes the thermophilic stage of composting. The next step known as curing or maturation phase, which includes the reduction of the temperature (to mesophilic range) and also degradation of remaining organic compounds, occurred at a slower rate [5]. Selection of primary mixture of sewage sludge compost is an important factor that effect on properties of the sludge, static condition, porosity, moisture content, C:N ratio and the ability to produce more enough energy to drive the process [6,7]. The duration of the first phase strongly depends on the controlling parameters management such as aeration and watering and also characteristics of the waste such as amount of easily degradable matter [5]. Within adequate range of aeration, maximum temperatures are achieved to ensure pathogen damage and fast stabilization. Appropriate rate of aeration is necessary because high aeration can lead to extreme heat loss and inadequate stabilization [8]. As mentioned above, the degradation potential of material is an important factor in duration of active phase. Increase in biodegradability of organic matter lead to decrease in duration of active phase, odor-producing and cost of composting. So, different pre-treatment processes have been proposed to enhance the biodegradability and hydrolysis of activated sludge [9,10].

Many sludge pre-treatment processes are currently used including biological, thermal hydrolysis, mechanical (such as ultrasound, high pressure) and chemical with oxidation (mainly ozonation) [11]. Ozonation is a most widely used chemical pretreatment and because of the complexity of sludge composition, ozone decomposes itself into radicals and attacks the extra cellular polymeric substances and oxidize the complex macromolecules that lead to enhanced sludge treatability and biogas production and also convert refractory organic matter into biodegradable form [12,13]. Ozonation was known as a process with microbial aggregates' properties in wastewater treatment system which can destroy the cell structure and release intracellular materials to the supernatant [14]. The increase in the soluble nitrogen and phosphorus concentrations after ozonation affirms the release of cell contents. Released organic N was oxidized to NH3 and finally nitrite and nitrate, which lead to increase of soluble TN (included the soluble nitrate and nitrite) [15]. The purpose of these methods is reduced excess sludge, improvement of the sludge dewatering, acceleration of the hydrolysis step, solubilization of the particulate organic matters that would allow to reduce the stabilization time for further microbial action [16,17]. Therefore, the aim of this study was to determine the effects of ozonation of WAS on duration of the composting process. Also, we followed some of the physico-chemical and microbiological characteristics of composting of semi-stabilized and completely stabilized sewage sludge with ozonation during the process.

2. Materials and methods

2.1. Waste activated sludge collection

The raw biological sludge was taken from an extended aeration activated sludge system in the local WWTP. This sludge was obtained from the return sludge line of the settling tank which represents medium strength domestic sewage and then transferred to laboratory and kept in the refrigerator (4°C) until use. The samples were analyzed to determine the initial characteristics of WAS, which was carried out on a dry weight basis and the standard deviations are based on triplicate analysis (Table 1).

2.2. Analytical methods

The total solid (TS), volatile solid (VS), total suspended solids (TSS), volatile suspended solids (VSS) were measured as described in Standard Methods [18]. Total coliform (TC) and fecal coliform (FC) for compost were measured by methods as mentioned in USDA and USCC [19]. pH was determined using a double-distilled water suspension of compost in a 1:5 ratio (w/v). Moisture and ash contents were determined by heating at 105°C and for 2 h at 550°C, respectively. Decomposition degree was calculated from the initial (A0) and final (Af) ash according to the following formula [20,21]:

Table 1

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Unit</th>
<th>Raw WAS</th>
<th>Semi-stabilized sludge (0.006 g O3/g TSS)</th>
<th>Completely stabilized sludge (0.041 g O3/g TSS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>–</td>
<td>7.58 ± 0.11</td>
<td>7.12 ± 0.15</td>
<td>7.07 ± 0.18</td>
</tr>
<tr>
<td>TS</td>
<td>g/L</td>
<td>18.35 ± 2.33</td>
<td>18.10 ± 3.01</td>
<td>17.07 ± 2.94</td>
</tr>
<tr>
<td>VS</td>
<td>g/L</td>
<td>14.51 ± 1.44</td>
<td>12.08 ± 1.73</td>
<td>7.34 ± 0.98</td>
</tr>
<tr>
<td>VS (%TS)</td>
<td>%</td>
<td>79 ± 3</td>
<td>67 ± 4</td>
<td>42 ± 4</td>
</tr>
<tr>
<td>TSS</td>
<td>g/L</td>
<td>14.18 ± 2.1</td>
<td>12.9 ± 1.5</td>
<td>12.01 ± 1.7</td>
</tr>
<tr>
<td>VSS</td>
<td>g/L</td>
<td>12.08 ± 1.5</td>
<td>10.5 ± 1.2</td>
<td>8.90 ± 1.1</td>
</tr>
<tr>
<td>VSS (%TSS)</td>
<td>%</td>
<td>85 ± 2</td>
<td>81 ± 3</td>
<td>74 ± 2</td>
</tr>
<tr>
<td>TOC</td>
<td>%</td>
<td>28.97 ± 3.21</td>
<td>27.93 ± 3.53</td>
<td>27.05 ± 2.89</td>
</tr>
<tr>
<td>TKN</td>
<td>%</td>
<td>1.7 ± 0.25</td>
<td>1.93 ± 0.16</td>
<td>2.05 ± 0.32</td>
</tr>
<tr>
<td>TP</td>
<td>%</td>
<td>5.39 ± 1.01</td>
<td>16.87 ± 1.92</td>
<td>18.44 ± 3.42</td>
</tr>
<tr>
<td>Ash</td>
<td>%</td>
<td>31.64 ± 3.44</td>
<td>31.72 ± 3.26</td>
<td>32.23 ± 2.18</td>
</tr>
</tbody>
</table>
The concentration of mineral N in the supernatant was analyzed by colorimetry using the Greiss and Ilosvay method [22]. The total organic carbon (TOC) was measured by a TOC analyzer system (multi N/C® 3100, Germany). All chemical analyses were performed with two to three replicates per sample, and the mean results per sample were used for statistical data treatment. Statistical calculations were done using the computer software package IBM SPSS statistics 20.0 (standard version).

2.3. Sludge ozonation

For ozonation, 1.2 L of sludge was placed in a 1.5 L bubble column with a diameter of 10 cm when is operated in batch scale and at room temperature. Ozone was produced by passing pure gaseous oxygen through ozone generator (Danali, Iran) by a maximum ozone production capacity of 6 g/h. Oxygen flow rates were changed from 0.2 to 1.5 L/min when different inlet ozone concentrations were placed. After each run, to determine an optimal ozone dose for production of semi-stabilized and completely stabilized sludge (CCS), samples were withdrawn for analysis.

To produce fine ozone bubbles and improve ozone mass transfer, a stone diffuser was installed and ozone was bubbled from the bottom of the reactor. The yield of ozonation at different O₃-injecting flux was measured by iodometric method. Also, the amount of ozone, leaving in the exhaust gases, was also measured by iodometric method [26]. Ozone consumption was calculated as the amount of ozone entering to the reactor minus the amount of ozone leaving the reactor:

\[
\text{Consumed ozone}(O₃) = \frac{(O_{\text{in}} - O_{\text{out}})}{S}
\]

where \(O_{\text{in}}\) = (ozone yield × ozone bubbling time), \(O_{\text{out}}\) = amount of ozone leaving the reactor, and \(S\) = dry weight of sludge. The applied ozone was quantified in terms of ozone dosage (g O₃/g TS) [27].

2.4. Composting

Two plastic containers (70 cm diameter and 30 cm high) were prepared for composting of semi-stabilized (CSS) and completely stabilized (CCS) sludge. One plastic container also was provided for composting of raw WAS (CRS) as a control treatment under controlled environmental conditions. The bottom of the reactor was filled by a layer of large woodchips (5 × 1 cm; depth 10 cm) to recirculate the air which separated with grid lace from the material. The dewatered sludge (dewatering on filter press at 51% (w/w)) was mixed with sawdust at 6.5% (w/w) to obtain a C/N ratio of 15:1. Matured vermi-compost was added as an amendment material at 32% (w/w) to the pile. The dewatered sludge does not have adequate porosity for sufficient aeration so bulking agents such as woodchips provide porous structure and maintain air spaces with the compost pile [28]. Therefore, woodchips were homogeneous mixed at 10% (w/w) with the composting system. The heaps were manually turned and mixed once in a week to provide aeration for the pile and average moisture content was maintained between 50% and 60% throughout the aerobic composting phase by periodic sprinkling of adequate quantity of tap (potable) water. The reactor was kept at room temperature (25°C ± 2°C) and heap temperatures were monitored daily. Heap temperature was measured at two-third depth from heap surface. During the composting process, samples were collected after every 7 d for the future analyses and all samples were analyzed for various physico-chemical parameters (pH, MC, TS, VS, TOC, TN, TP and microbiological parameters). For sampling, smaller amounts of mixture were collected from various locations of the reactor to obtain 1 kg samples. Then it was completely mixed, and a 250-g homogenized sample was taken and analyzed. The remaining sample was returned to the reactor. All experiments were performed in triplicate and data reported as average of triplicates.

3. Results and discussion

3.1. Ozonation

3.1.1. Ozone dose optimization

To determine the sludge stabilization, the sludge characteristics are tested using different ozone doses. By installing of stone diffuser in the bottom of the column, there was the high solubility of ozone in water when the ozone concentration at the contactor outlet was very low. Solids concentration was used for the quantification of ozone’s effect on organic and mineral matter. Ozonation released the soluble cell compounds and hydrolysis solid organics content of sludge which lead to increase in inorganic compounds such as phosphate, nitrate and sulfate and also decrease in the TS and VS content [27,29]. TS concentration was almost constant at a mean value of 18.35 g/L and the organic content (VS/TS ratio) was detected to be 79% for WAS. To determine the semi and completely stabilized sludge, the impact of the variation of ozone dose on the ratio of VS/TS was analyzed. By increasing in ozone dose, a significant decrease in the organic content (VS/TS ratio) was observed. Accordingly, the maximal VS/TS ratio was 67% and the minimal VS/TS ratio was 39% for ozone doses of 0.006 and 0.0782 g O₃/g TSS, respectively. Therefore, inlet ozone concentrations to produce the semi-stabilized and completely stabilized sludge were determined to be 0.006 g O₃/g TSS (VS/TS ratio of 67%) and 0.041 g O₃/g TSS (VS/TS ratio of 42%) in temperature of 25°C ± 2°C, respectively (Fig. 1).

3.1.2. Ozonated sludge characteristics

Many studies have shown that pre-treatment of WAS by ozonation improved the characteristics of sludge for biodegradability [27,30]. As seen in Table 1, the pH value
decreased obviously from 7.58 for WAS to 7.12 and 7.07 for semi-stabilized and completely stabilized sludge. The decrease in pH during ozonation was due to the formation of acidic compounds such as carboxylic acids and volatile fatty acids from the degradation of lipid compounds [13,31]. By increasing the ozone dose, TS reduction was increased by 1.36% to 6.97% and the VS reduction was increased by 16.74% to 49.41% for semi-stabilized and completely stabilized sludge, respectively. The VSS was 85% of TSS in WAS before the injection of ozone. But this parameter was reduced for ozonized WAS, which could attribute to primary effect of ozonation on sludge disintegrations [32].

After ozonation, TOC concentration was decreased from 28.97% to 27.05% for semi-stabilized and completely stabilized sludge which represents an average removal rate of 3.6% and 6.62%, respectively. The decrease in total TOC is because of a complete oxidation of the sludge to CO₂ [33]. However, Pei et al. [34] demonstrated that ozonation generates oxidizing and non-selective radicals which lead to release of more soluble organic matter and meaningful increase in TOC concentration. The TN and TP concentration during ozonation showed significant rises in two indexes. Minimum amount of TN and TP was found for WAS to be 1.7% and 5.39%. After ozonation at 0.006 (semi-stabilized) and 0.041 (completely stabilized) g O₃/g TSS doses, TN represented an increase by 12% and 17%, and TP with mainly increasing became 68% and 70%, respectively. One possible explanation is that penetrated ozone rises the cell membrane osmosis of the microorganisms which causes interaction with the cell membrane and release the cell contents into supernatant [27,35]. The increase in the soluble TKN and TP concentrations in the WAS after ozonation confirm the release of cell contents. After ozonation of WAS and releasing of intracellular materials, organic N was oxidized to NH₃ and finally nitrite and nitrate which lead to increasing of soluble TN (included the soluble nitrate and nitrite). Furthermore, reduction of VS and increasing dissolved contents were emphasised on more effective solubilization of WAS via ozonation. On the other word, raising in volatile dissolved solids and total dissolved solids go along with decreasing VSS and TSS of WAS after ozonation strongly indicated solubilization [15].

3.2. Composting

3.2.1. Physico-chemical changes during composting

3.2.1.1. Temperature

The change of temperature recorded during the composting is presented in Fig. 2. The mean temperature of the composting in the first day was in the range of 25°C–27°C for three reactors. In initial phase of composting, a fast increase of temperature was observed in both CSS and CCS as the temperature reached 55°C and 58°C within 5 d, respectively. Although the temperature changes for CRS were obviously slow, the two reactors of CSS and CCS were consisted of easily biodegradable matters which lead to fast breakdown of organic compounds by intense microbial activities [36,37]. After almost 20 d and development of mesophilic communities, temperature dropped to maturing phase and reached the ambient 25°C.

3.2.1.2. pH

The physical and chemical properties of compost changed significantly after processing. There was a significant change in the pH value as shown in Fig. 3a. The initial pH values were 7–7.6 in all the trials. The difference in pH values could be attributed to the formation of acidic compounds in ozonized sludge. The pH values in all treatments increased until 14th d to 8.1, 8 and 7.58 for CRS, CSS and CCS, respectively. It seems possible that these results are due to intense microbial activity, mineralization of organic matter and ammonia volatilization during first few days [38]. Although after 14th d, values of pH rapidly decreased in all treatments to reach levels of about 7–7.5, the reduction trend in CRS and CSS was faster than CCS. Due to degradation and stabilization of organic matter in the ozonation stage the lowest pH is reported in CCS.

3.2.1.3. Volatile solid

The concentration of TS and VS in the three reactors was monitored during the experiments. Significant reduction in VS was known as one of the important parameters for substrate mineralization, decomposition and compost maturity. The rate of reduction in CRS was higher when
compared with CSS and CCS under the same experimental conditions (Fig. 3b). The final VS was found to be 45.8%, 42% and 37% for CRS, CSS and CCS, respectively. However, the initial VS in reactor CCS was lower than the two others. The highest mortality during compost was reported to be 28% in CRS, while these casualties were reported by CCS 26% and 17% in VS. The slowly rate of decreasing for CCS can be attributed to mainly mineralization of organic matter during ozonation stage and presence of a considerable amount of non-biodegradable matter in the completely stabilized sludge.

3.2.1.4. Ash and moisture content

An important parameter for mineralization and decomposition of the substrate is found to be ash content. The content of ash was increased during 35 d of process in all treatments. Fig. 4a shows the ash content profile during each experiment. The average value of initial ash content in the CRS, CSS and CCS was found to be 48.2%, 48.5% and 49.2%, respectively. It can also be seen from Fig. 4a that the rate of increasing in ash content in CCS (17%) was significantly faster than CRS (9%) and CSS (13%) that indicated the higher rate of volatilization, which is a good measure of degradation of the organic waste. On the other hand, decomposition degree of organic matter in the end of the composting process for CRS, CSS and CCS was estimated to be 17.8%, 25.5% and 33.42%, respectively (Fig. 4b). Jouraiphy et al. [20] reported that after 135 d of composting of sewage sludge and green plant waste, degree of decomposition reached 60.8% [20]. Increase in ash content and decomposition degree during composting reflected microbial degradation of organic matter and stabilization during composting and the weight loss of dry matter [21].

Fig. 3. Variation of pH (a) and VS (b) during composting of raw and ozonized WAS.

Fig. 4. Variation of ash content (a) and degree of decomposition (b) during composting of raw and ozonized WAS.
The moisture content throughout the experiment in reactor CRS, CSS and CCS in all of the 35 d was adjusted to reach optimal moisture content between 60% and 65%. The moisture content for composting is necessary to achieve adequate water content for microorganisms to move and transport nutrients, and adequate oxygen flow to maintain aerobic conditions [21].

3.2.1.5. TOC

It is apparent from Fig. 5a that there is a significant decrease in TOC in all treatments which may be due to mineralization of net organic matter by microorganisms [4]. During the process, concentration of TOC in CCS was significantly lower as compared with control (CRS). The maximum and minimum TOC losses in final mixtures were observed to be 26.5% and 14.5%, respectively.

This could be due to the increase of biodegradability of organic matter by ozonation in the last stage and enhanced metabolic activity of microorganisms on the ozonized WAS in CCS that led to increases in carbon consumption and CO₂ release. Variation of pH, VS, ash content and TOC in CRS, CSS and CCS during the composting is shown in Table 2.

3.2.1.6. TKN and ammonium nitrogen (N–NH₄⁺)

The total nitrogen consists of the inorganic forms of nitrogen (N–NH₄⁺ and N–NO₃⁻) and organic nitrogen (N_{org}). In Fig. 5b there is a clear trend of increase in TN content in all the treatments. The initial TN content of three reactors was approximately same. But no differences in total nitrogen were observed between reactors of CRS and CSS during 35 d of composting. The content of TN varied from 0.61%–0.84%, 0.63%–0.90% and 0.67%–1.05% for CRS, CSS and CCS, respectively. The maximum and minimum increase was observed in composting of completely stabilized sludge in CCS and composting of WAS in CRS with 36% and 27%, respectively. The increasing trend in TN content during composting in this study corroborates these earlier findings [39,40]. Substrate utilization by microbes and their metabolic activities led to the net reduction in dry mass (organic carbon in terms of CO₂), also, evaporation during mineralization of organic matter caused reduction in water and might led to relative increase in TN [21]. Although, these results differ from some published studies, they illustrated a decrease in amount of total nitrogen during the composting of sewage sludge [9,41].

As can be seen in Table 3, inorganic nitrogen (N–NH₄⁺ and N–NO₃⁻) was significantly changed during the composting.
for all treatments. It is apparent that a decrease in N–NH$_4^+$ and an increase in N–NO$_3^-$ were occurred. The highest release of N–NH$_4^+$ was demonstrated in CCS which it may be due to enhancement of degradation in the pretreatment by ozonation. In accordance with the present results, previous studies have demonstrated that ammonia reduction is closely related to organic matter degradation [42]. After generation of ammonia during the biological decomposition, it was transformed to nitrate by nitrification bacteria and led to increase in amount of nitrate by decrease in ammonia [43]. According to Garcia et al. [44], the increase in nitrate, related to nitrification, resulted in NH$_4^+/NO_3^-$ ratio < 1 at the end of the process, suggesting that the final compost had reached maturity.

### 3.2.1.7. C/N

In all treatment process, the C/N values decreased considerably during the process, with initial values of 30.4, 27.3 and 27.1 falling to 19, 16 and 12 in 35th d for CRS, CSS and CCS, respectively (Table 4). The reduction in C/N ratio during composting was 37%, 40% and 53% for CRS, CSS and CCS, respectively. The decreasing trend of C/N ratio during the process corroborated these earlier findings [20,38]. Decrease in the C/N ratio is expected as a consequence of organic waste mineralization and stabilization rate during the process of composting.

As seen in Table 4, CCS had the highest reduction of C/N ratio over time compared with CRS and CSS and also, CCS could reach C/N ratio to less than 16 after 21th day. Among these reactors, the difference in the reduction of the C/N ratio indicates that the role of organic matter degradation at the cylinder stage is much faster and shows that the amount of organic matter mineralization has increased over compost. Reducing organic carbon to CO$_2$ due to microbial respiration increases nitrogen levels and, as a result, decreases C/N ratio. According to Senesi [45], an advanced degree of stabilization and acceptable maturity is the C/N ratio < 20, while a ratio of 15 or less being suitable for agronomic usage of composts, since plants assimilate nitrogen just in the ratio of 20 or less. So, in this study, an advance degree of organic matter stabilization was achieved in three treatments, however, the C/N ratio for CCS was considered satisfactory for agricultural use.

### 3.2.2. Microbiological changes during composting

The microbiological parameters of TC and FC contents in the composted material are presented in Fig. 6. There was a reduction in the number of coliforms in all the reactors. It was observed that the amount of TC decreased in CRS, CSS and CCS in 97%, 98% and 99.13%, respectively. The TC and FC in CCS reached to standard values after 28th d. The difference in the initial TC and FC in the reactor of CRS, CSS and CCS can be attributed to the ozonation process caused significant reduction in all the TC and FC. The decrease of pathogen during composting can be attributed to reduction of microbial activity due to elimination of organic matter and also the competition between pathogens and microbes for the limited resources during the process [46].

### 4. Conclusions

This work has demonstrated that ozonation of WAS had an affirmative effect on composting materials. The results clearly suggest that this pre-treatment process could enhance

<table>
<thead>
<tr>
<th>Parameters</th>
<th>C/N (%)</th>
<th>C/P</th>
<th>TC (MPN/g dw)</th>
<th>FC (MPN/g dw)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CRS</td>
<td>CSS</td>
<td>CCS</td>
<td>CRS</td>
<td>CSS</td>
</tr>
<tr>
<td>0 D</td>
<td>30.4</td>
<td>27.3</td>
<td>27.2</td>
<td>3.67</td>
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<td>7 D</td>
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</table>
some important plant nutrients, for example, TN, available P content and decrease in C:N ratio up to its acceptable limit (1:20) in the shorter time. In addition, this study introduced the ozonation as a sustainable pre-treatment technology for chemical–biological treatment. Increase in biodegradability of organic matter could lead to decrease in duration of active phase in composting process and convert the noxious wastes into organic fertilizer to apply in agricultural lands. Also, it can be concluded from the present study that the combined ozonation–composting process can gain complete sanitization under desirable environmental conditions.

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