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The Use Of Respirometric Techniques For The Monitoring Of The Composting Process: Comparison Of Assays At Fixed And Process Temperatures.

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Abstract

A static respirometer was built to determine the Respirometric Index (RI) of composting samples at different temperatures. Composted materials were dewatered sludge (DS) and dewatered anaerobically digested sludge (ADS) obtained from a wastewater treatment plant. Respirometric indexes were obtained at a fixed value of 37 ºC (RI_{37}) and at the *in situ* temperature of the reactor at the moment of sampling (RI_T). Results indicated that both indexes correlated well with temperature evolution in the composter. However, RI_T were more representative of the metabolic activity in the reactor and were also more sensitive to temperature and composition variations of the composting material. Nevertheless, when process temperature felt below optimum mesophilic conditions, RI_T were useful for the monitoring but not for determining the actual stability of the material. Moreover, the importance of the units used to express RI was also analysed during the composting of sludge from the paper industry, a highly compostable residue. RI expressed on dry matter basis indicated that this was a stable material even before composting (values below 1 mg $O_2 \cdot g\ DM^{-1} \cdot h^{-1}$) whereas RI expressed on organic matter basis corresponded to an unstable material (values around 2.5 mg $O_2 \cdot g\ OM^{-1} \cdot h^{-1}$).

Key words: composting, sludge, respirometric index, monitoring, stability.

Introduction

Composting has received much attention as a potential technology for treating solid wastes such as municipal solid wastes, sewage sludge, yard trimmings, and agricultural wastes. During the last decades research has focused on the qualitative evaluation of the composting process. Several indicator variables have been proposed for monitoring the process and evaluating the stability of the final product. Biological and biochemical indexes are particularly useful since they relate composting processes to metabolic activity; methods such as total ATP, enzyme activity and microbial biomass measurements have been employed (García *et al.*, 1992; Tiquia & Tam, 1997; García & Polo, 1999; Benítez *et al.*, 1999; Tiquia *et al.*, 2002). However, the most widely used biological methods are the respirometric studies which determine the O_2 or CO_2 evolution during the microbial metabolism (Kuter *et al.*, 1985; Atkinson *et al.*, 1996; Pérez *et al.,* 1999, Larsen & McCartney, 2000; Lasaridi *et al*., 2000). The basis of these methods is that immature compost has a strong demand for O_2 and high CO_2 production rates due to intense development of microorganisms as a consequence of the easily biodegradable compounds in the raw material. Conversely, at late composting stages both processes decline as the amount of degradable organic mater decreases. However, it has been argued (Haug, 1986; Adani *et al.,* 2002) that O_2 consumption is a more reliable method since oxygen is directly responsible for the oxidation of the organic matter. Still, O_2 monitoring requires more control and therefore more sophisticated equipment and is more time consuming than the measurement of CO_2 production.

Methods based on O_2 consumption have been classified into dynamic and static protocols (Adani & Genevini, 2001). Dynamic methods are characterised by a continuous oxygen supply whereas static methods can be used with solid or liquid samples. A wide range of devices has been developed for the measurement of respiration from solid matrices. One of the most widely reported uses a dissolved oxygen (D.O.) meter with a polarographic probe measuring the decline in oxygen concentration over a compost sample in a flask. Such tests generally provide near-optimum conditions for microbial respiration (*e.g.* moisture, nutrients, and oxygen supply). In almost all respirometric experiments, the measurement of respiration activity is carried out at standard temperatures of about 30 - 37 ºC (USDA, 2001; Ianotti *et al.* 1993; Lasaridi & Stentiford, 1998). It is considered that respirometric activities measured at these fixed temperatures are good indicators of the mean metabolic potential of the compost. Nevertheless, composting is a complex process where the rate of degradation is a result of the metabolic activity from a mixed microbial population that includes microorganisms with different optimum growth temperature. It can be considered that, although respirometric experiments performed at fixed temperatures are a useful indicator of the compost stability, they do not show the actual metabolic stage of the process and therefore, cannot be used to follow its evolution. On this basis, this paper describes the experimental setup of a respirometer developed to determine the Respirometric Index of composting samples at different temperatures. The final aim of this work will be to have a reliable method to monitor the composting process.

2. Materials and Methods

2.1 Substrates characteristics

Homogenised source-separated organic fraction of municipal solid waste (OFMSW) was obtained from the full-scale municipal solid waste composting plant in Granollers (Barcelona). Samples were taken directly from the composting tunnels.

Dewatered sludge (DS), consisting of primary and activated sludge, was obtained from the municipal wastewater plant in La Garriga (Barcelona). Dewatered anaerobically digested sludge (ADS) was obtained from the municipal wastewater treatment plant in Granollers (Barcelona). Physico-chemical sludge (PCS) was obtained from the de-inking process of a recycled paper manufacturing industry.

Wood chips, consisting of a variable mixture of pine and beech tree wood, were obtained from a local carpentry and used as bulking agent. Sludge and wood chips were mixed by hand after the screening of the bulking agent. A semi-industrial sieve (Filtra Vibración, FT-400) was used for the wood chips screening. Three different screens (20, 10 and 5 mm) mesh were available. A 1:1 sludge:wood chips volumetric ratio was used in all experiments since it had been previously optimised by Gea *et al.* (2003).

2.2 Composter

A 100 L old fridge was conditioned to be used as a static composter and was filled either with OFMSW, the sludge:wood chips mixture or PCS. No conditioning was needed for the OFMSW since it had been directly taken from the piles in the composting plant and it already included the bulking agent.

The recipient was kept horizontally with a slight inclination to allow its opening from the top and to make the collection of possible leachates easier. A plastic mesh was fitted at the bottom of the recipient to support the material and collect the leachates. Several holes were perforated through the walls of the vessel to permit the entry and exit of air, the discard of leachates and the insertion of different probes. Air inlet was at one end of the vessel whereas its outlet was at the other end. An air flow rate of 20 L/min was used and its entry was regulated either by temperature or O_2 control. Four Pt 100 sensors (Desin mod. SR-NOH) inserted at different points inside the vessel, were used for monitoring the temperature. Oxygen concentration in interstitial air was monitored with an oxygen sensor (Sensox, Sensotran, Spain). CO_2 concentration at the outlet of the composter was monitored with an infrared detector (Sensontran I.R., Sensotran, Spain). All sensors were connected to a home-made data acquisition system. Oxygen was controlled by means of a feedback oxygen control which automatically supplied fresh air to the reactor when oxygen concentration fell below 10%.

2.3 Chemical and physical analyses

Approximately 1 L samples were collected periodically to determine organic matter content and the respirometric index. Samples from OFMSW were sieved to remove glass, plastics and other inerts and oversized material. For respiration experiments, humidity content was adjusted to a range of 40 – 50% (w/w) if required. Analytical parameters were determined according to the standard procedures recommended by the Test Methods for the examination of Composting and Compost (TMCC) (USDA, 2001).

2.4 Respirometer

A static respirometer (Figure 1) has been built according to the original model described by Ianotti *et al.* (1993, 1994) and following the modifications and recommendations given in the TMCC (USDA, 2001). The drop of oxygen content in a flask containing a compost sample was monitored with a dissolved oxygen meter (Lutron 5510, Lutron Co. Ltd., Taiwan) connected to a data logger.

The setup included two water baths to allow carrying out experiments at two different temperatures simultaneously. Temperatures assayed were at a fixed value of 37 ºC and at the *in situ* temperature of the composter at the moment of sampling. Prior to the assays, samples for experiments at 37 ºC were incubated for 18 hours at this temperature, while samples for experiments at *in situ* temperatures where incubated for 4 hours at such temperature. During all the incubation period samples were aerated with previously humidified air at the sample temperature.

The aeration system consisted of a flask with a two-hole stopper and two glass delivery tubes. At the bottom tip, the delivery tube had an aquarium air-stone to produce small air bubbles. This sparger was immersed in water to humidify the air. The flask was always kept inside the incubation bath. The delivery tube was connected to a manifold that served to deliver air to the different flasks containing the samples. The manifold was constructed from plastic tubing connected by quick disconnect fittings. Individual valves were also fitted to each sample tubing to regulate the air flowing.

Once the incubation period was finished, the D.O. sensor was assembled into the flask without removing it from the water bath. The air pressure inside the flask was then equilibrated with the outside pressure by inserting a hypodermic needle through the stopper and leaving it in place for 5 minutes. O_2 level was then recorded every 15 seconds for 90 minutes. In all experiments three replicates were used. After the D.O. readings finished, the total volume of free air space in each sample flask was determined as described in TMECC (USDA, 2001).

2.5 Calculation of Respirometric Index (RI)

The Respirometric Index (RI) of the compost sample referred to total organic matter content was calculated from the slope in a linear segment on the chart O_2 (%) versus time by using equation 1.

$$RI = \frac{V \cdot P \cdot 32 \cdot m \cdot 60}{R \cdot T \cdot X \cdot DM \cdot OM} \qquad (1)$$

where: RI, respirometric index (mg O_2· mg OM^{-1}· h^{-1}); V, volume of air in flask (mL); P, atmospheric pressure at elevation of measurement (atm); m, slope of change in percent O_2 saturation per minute divided by 100; R, ideal gas constant (0.08206 L· atm· mol^{-1}· $^{o}K^{-1}$); T, temperature in degrees (K); X, wet weight of compost test aliquot (g); DM, fraction of total solids of a parallel sample aliquot (g DM· g X^{-1}); OM, fraction of organic matter of a parallel sample aliquot in dry basis (g OM· g DM^{-1}).

3. Results and Discussion

3.1 Respirometer validation

Amongst the modifications developed in this work it is worth mentioning that air used for the aeration of the samples before the respirometric assays was saturated with water at the same temperature as the water bath, otherwise aeration resulted in the drying of the sample. Moreover, the use of quick disconnect fittings and individual valves allowed a better control of the aeration rate.

Once the respirometer was built, next step was to evaluate the error of the respirometric assays. RI of OFMSW samples obtained from the static composter at different days were determined. Temperatures assayed were at a fixed value of 37 ºC (T_{37}) and at the *in situ* temperature of the composter (T) at the moment of sampling with 18 and 4 hours incubation time respectively. Incubation time for the fixed temperature was chosen according to the literature (Iannotti *el al.*, 1993; USDA, 2001). As no information was available regarding incubation for the experiments at the *in situ* temperature, different incubation times were assayed. Results indicated (data not shown) that RI values remained practically constant for incubation times above 4 hours. Figure 2 illustrates a typical chart O_2% versus time and RI values for three replicates of a same sample, as expected % oxygen saturation decreased with time. Table 1 shows RI values and standard deviations obtained for the two temperature conditions assayed. Standard deviations from respirometric tests at the start of the composting process are larger than those obtained at the end probably due to the homogenisation of the material along the process. It is also observed that at high temperatures (55 ºC) standard deviations for RI evaluated at T were higher than those for RI at 37ºC. This could be because in this case, the former was closer to the upper limit of the temperature compensation of the electrode (60 ºC). This could mean that, little variations in the temperature of the water bath may lead to small oscillations in the O_2 probe lecture, increasing the error associated to the measurement of the slope.

3.2 Units of RI

RI units are generally referred on the basis of total organic matter content. However, the convenience of using units based on total solids content has also been suggested (CCQC, 2001) since the organic matter content can be very variable depending the heterogeneity of the material. In consequence, this variability may introduce significant errors in the evaluation of the RI.

Therefore, the significance of the units used for the respirometric index was evaluated during the pilot-scale composting of physico-chemical sludge (PCS) resulting from the de-inking process of a recycled paper manufacturing industry. Characteristics of this material (Table 2), such as relatively high C/N ratio, low organic content and moisture had indicated a low compostability. However composting at laboratory scale with no bulking and no amendment showed good performance (data not shown). Temperature profile and respirometric indexes determined at 37ºC (RI_{37}) obtained during the composting at pilot-scale are shown in Figure 3, where it is observed that a thermophilic range was reached within two days, and was maintained for more than two weeks.

From Figure 3, it can also be seen that RI values calculated on dry matter basis were all below 1 mg $O_2 \cdot$ g $DM^{1} \cdot h^{-1}$ regardless of the composting stage. According to literature (USDA, 2001; CCQC, 2001), these values would correspond to a stable material. This is in clear contradiction to the actual activity shown by the material as confirmed by the temperature profile of the process in Figure 3. Conversely, RI based on organic matter content correlates well with the temperature profile with high values corresponding to an unstable material during the thermophilic range and falling down to 1 mg O_2 g $OM^{1} \cdot h^{-1}$ during the mesophilic phase. These results seem to indicate that when dealing with materials with high content of non-degradable material, respirometric indexes should be referred to organic matter basis since the high content of non-degradable material may lead to misleading results when expressed on dry matter basis. Therefore, although its variability, it seems more appropriate to express RI on organic matter basis specially when comparing different processes.

3.3 Monitoring of the composting process by respirometric techniques

Figure 4 shows that composting of sludge (DS) follows the typical evolution of this type of processes. It is observed that during the first two days process temperature quickly rose to 65 ºC followed next day by a fast drop to 40 ºC and a less pronounced fall during the following days to temperatures as low as 20 ºC. Temperatures then increased slowly up to values around 30 ºC. They were maintained in the mesophillic range until the process was finished.

Evolution of the composting of anaerobically digested sludge (ADS) is shown in Figure 5. In this case, it can be seen that temperature rapidly reached a thermophilic range in the first days of the process, with values up to 60 ºC, followed by a moderate drop to around 38 ºC and a subsequent quick increase up to 50 ºC. Afterwards, temperature slowly decreased to the mesophillic range.

There are two possible explanations for the temperature profiles during the first stage of the process. The most generally accepted is that once easily degradable materials are depleted metabolic activity slows down causing a temperature drop. Microorganisms able to degrade more complex materials develop then and their metabolism provokes again the generation of heat and a new temperature increase. However, it has also been argued that this behaviour maybe because high temperatures may lead to a sudden decrease in the metabolic activity of the microorganisms and consequently, a reduction in the amount of heat generated. The material then cools down and temperature drops. This cooling down reactivates the metabolic activity, heat is generated again and so temperature rises again. This last argument could explain the behaviour of the DS since the drop in temperature was quicker than for ADS and its organic matter content was higher (80.2 % dry basis) than for ADS (61.7 % dry basis). However, in practice it is very difficult to determine the actual reason for the temperature profiles.

Figure 4 and Figure 5 also show the respirometric indexes obtained at 37 ºC (RI_{37}) and at the *in situ* temperature (RI_T) along the process for fresh sludge (DS) and anaerobically digested sludge (ADS) respectively. From these Figures it can be seen that both type of indexes correlated well with temperature evolution. The values indicate a higher metabolic activity at the beginning of the process, a reduction in activity during the cooling down phase and, an increase in activity once the temperature started to increase again. Also, indexes were fairly constant when temperature changes were small.

Nevertheless, although RI_{37} and RI_T values followed the same trend, several differences are found between them. In the case of DS (Figure 4), when temperatures in the composter were high, RI_T were significantly higher than RI_{37} and as temperature quickly dropped so RI_T did. The reason for this could be that at high temperatures prevailing population in the composter was thermophillic. Therefore, this population would show higher activity for assays performed at the actual temperatures of the reactor compared to the activity shown at 37 ºC despite the incubation prior to the assay. Conversely, when composting temperatures fell to around 20 ºC, RI_T were lower than RI_{37}. This would indicate that RI_T were sensitive enough as to detect the decrease in the metabolic activity within the reactor whereas RI_{37} were less sensitive to these changes. As expected, when process temperatures were closer to 37 ºC, both indexes were similar.

In the case of ADS, Figure 5 shows that RI_T and RI_{37} followed the same trend as for DS and that RI_{37} values are quite similar for both materials. However when comparing both indexes, it can be seen that RI_T values were considerably lower for the ADS. This lower activity maybe related to the differences in the organic matter content of both sludge since DS contained 80.2 % (dry basis) of organic matter while ADS contained 61.7% (dry basis). Besides, ADS not only contained less organic matter but since they were an already digested material, their organic fraction was less biodegradable than that of DS. Therefore, activity is not only related to the organic matter content of the material but to its nature as well.

The differences in RI_T values for DS and ADS are in good agreement with the differences in the composting profiles of both sludge. Higher activity and sharper increase and higher temperatures are observed at the beginning of the process for DS (Figure 4) when compared with the composting of ADS (Figure 5). A faster drop in activity is also observed. The main reason for this difference seemed to be the different organic content and biodegradability of both materials.

Results then indicate that RI_T are more sensitive to temperature changes and composition variations of the composting material than RI_{37}. Therefore, RI_T would provide more representative information of the actual metabolic activity in the composter. However, when process temperature falls below optimum mesophillic conditions, although RI_T are able to detect the actual metabolic activity in the reactor, they are unable to indicate the potential metabolic activity of the material since their values would correspond to a stable material which in practice is not. Therefore under these conditions RI_T are useful for the monitoring of the composting process but not for determining the actual stability of the material. Moreover, from the operational point of view RI_T assays are faster than RI_{37} since incubation time is only 4 hours compared to the 18 required for the later.

While respirometry techniques have been widely used as stability indicators for compost, their use for the monitoring of the process has been limited. Results presented here demonstrate that they are actually an excellent indicator for the monitoring of the process although they are by far, more complicated than temperature measurement and monitoring. Nevertheless, relationship between temperature and respirometric indexes are currently under investigation.

Conclusions

A reliable device has been constructed for determining the RI of compost samples. Results have shown that RI measurements at the *in situ* temperature of the process are more representative of the metabolic state in the composter. Measurements at 37 ºC may lead to underestimate the microbial activity. Composting of sludge (DS) is a faster process than the composting of anaerobically digested sludge (ADS) due to its higher biodegradable material content. RI measurements correlate well with these findings.

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References

Adani, F., Genevini, P., 2001. Determination of biological stability and oxygen uptake rate on MSW and derived products. Compost Sci. Util. 9(2), 163-178.

Benítez, E., Nogales, R., Elvira, C., Masciandaro, G., Ceccanti, B., 1999. Enzyme activities as indicators of the stabilization of sewage sludges composting with *Eisenia foetida*. Bioresource Technol. 67, 297-303.

California Compost Quality Council, 2001. CCQC Compost Maturity Index. http.//www.ccqc.org/Documents/MatIndex.pdf

García, C., Hernández, T., Costa, F. , Ayuso, M., 1992. Evaluation of the maturity of municipal waste compost using simple chemical parameters. Commun. Soil Sci. Plant Anal. 23,1501-1512.

García, C., Polo, A., 1999. Estudio de parámetros bioquímicos en procesos de estabilización de residuos orgánicos urbanos. Residuos 51, 76-81.

Gea Leiva, T., Artola Casacuberta, A., Sánchez Ferrer, A., 2003. Application of experimental design techniques to the optimization of bench-scale composting conditions of municipal raw sludge. Compost Sci. & Util. 11 (4), 321-329.

Haug, R., 1986. Composting process design criteria, part 1: Feed conditioning. Biocycle 27(8), 36.

Iannotti, D.A., Pang, T., Toth, B.L., Elwell,D.L., Keener, H.M., Hoitink, H.A.J., 1993. A quantitative respirometric method for monitoring compost stability. Compost Sci. Util. 1, 52-65.

Iannotti, S.A. Grebus, M.E., Toth, B.L., Madden, V. Hoitink, H.A.J., 1994. Oxygen respirometry to assess stability and maturity of composted municipal solid waste. J. Environ. Qual. 23, 1177-1183.

Kuter, G., Hoitink, H., Rossman, L., 1985. Effects of aeration and temperature on composting of municipal sludge in a full-scale vessel system. J. Water Pollut. Control Fed. 57(4), 309-315.

Larsen, K. McCartney, D., 2000. Effect of C:N ratio on microbial activity and N retention: bench-scale study using pulp and paper biosolids. Compost Sci. Util. 8(2), 147-159.

Lasaridi, K.E., Stentiford, E.I., 1998. A simple respirometric technique for assessing compost stability. Wat. Res. 32, 3717-3723.

Pérez, C., Manzano, S., Soliva, M., 1999. Compostaje conjunto de la fracción orgánica de residuos municipales (FORM) y residuos vegetales: influencia sobre los desprendimientos de CO_2 y NH_3. Residuos 46, 44-51.

Tiquia, S., Tam, N., 1997. Effects of bacterial inoculum and moisture adjustment on composting of pig manure. Environ. Pollution 96(2), 161-171.

Tiquia , S., Wan, J., Tam, N., 2002. Microbial population dynamics and enzyme activities during composting. Compost Sci. Util. 10(2), 150-161.

U.S. Department of Agriculture & U.S. Composting Council, 2001. Test methods for the examination of composting and compost. Edaphos International, Houston, TX.

Table 1. Respirometric indexes and standard deviation of MSW samples.

Mean Process temperature (ºC)	**Time of composting (Day)**	**Temperature of the respirometric test (ºC)**	**Respirometric Index RI (mg O_2·g OM^{-1}·h^{-1})**	**Standard deviation**
55	2	55	8.75	1.17
60	4	37	3.86	0.10
55	8	37	2.46	0.20
		55	3.75	0.59
55	11	37	2.23	0.11
		55	6.95	0.63
55	17	55	3.17	0.21
50	25	37	0.96	0.45
		50	4.31	0.19

Table 2. Main characteristics of physico-chemical sludge from recycled paper manufacturing industry.

Dry matter (%)	63.3
Water content (%)	36.7
Total organic matter (% dry matter basis)	33.7
pH (water extract 1:5)	7.50
Electrical conductivity (dS/m, water extract 1:5)	1.92
Total N-Kjeldahl (%, dry matter basis)	0.43
C/N ratio	34.0
$N-NH_4$ (%, fresh matter basis)	0.08
Total P (%, dry matter basis)	<0.10
Total K (%, dry matter basis)	<0.10

LEGENDS TO FIGURES

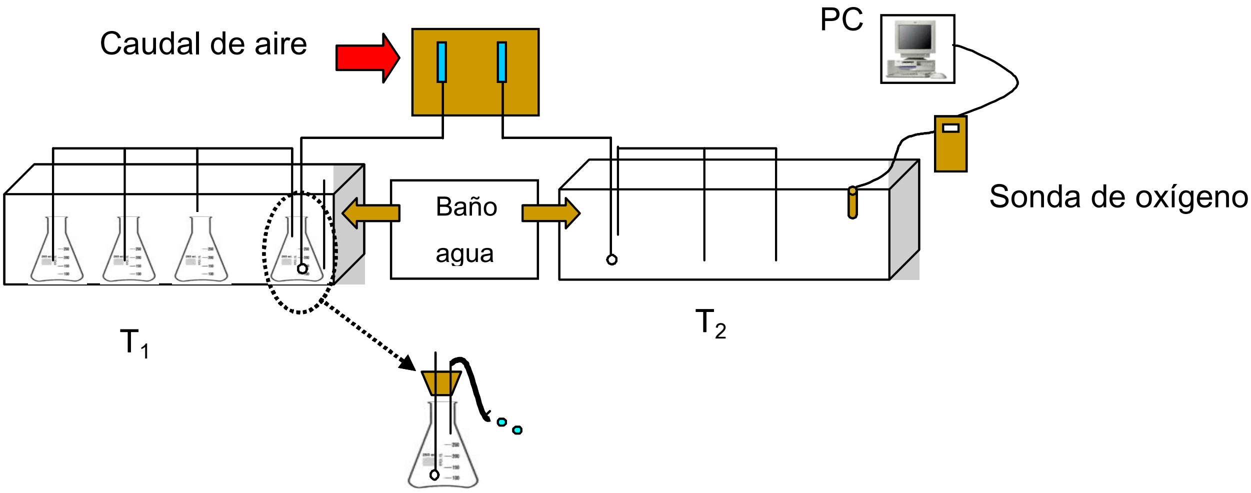
Figure 1. Respirometer setup.

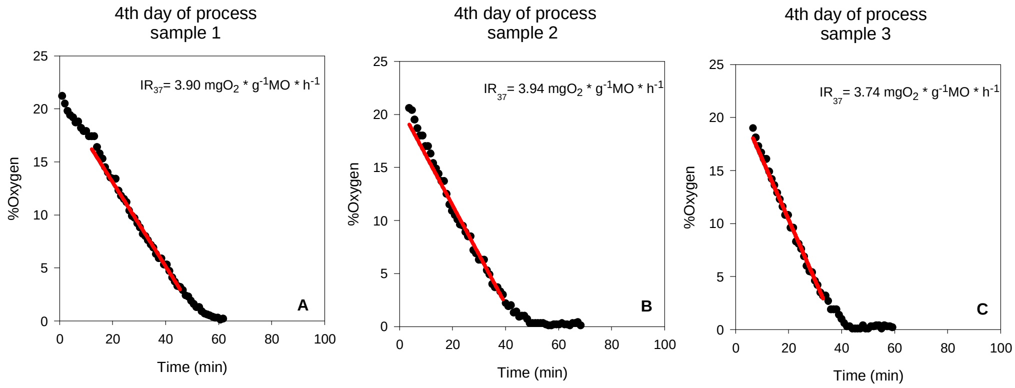
Figure 2. O_2 (%) versus time of three replicates of a OFMSW sample taken the fourth day of composting process. RI values obtained at 37 ºC.

Figure 3. Respirometric index and temperature profiles in the composting of PCS at pilot plant scale

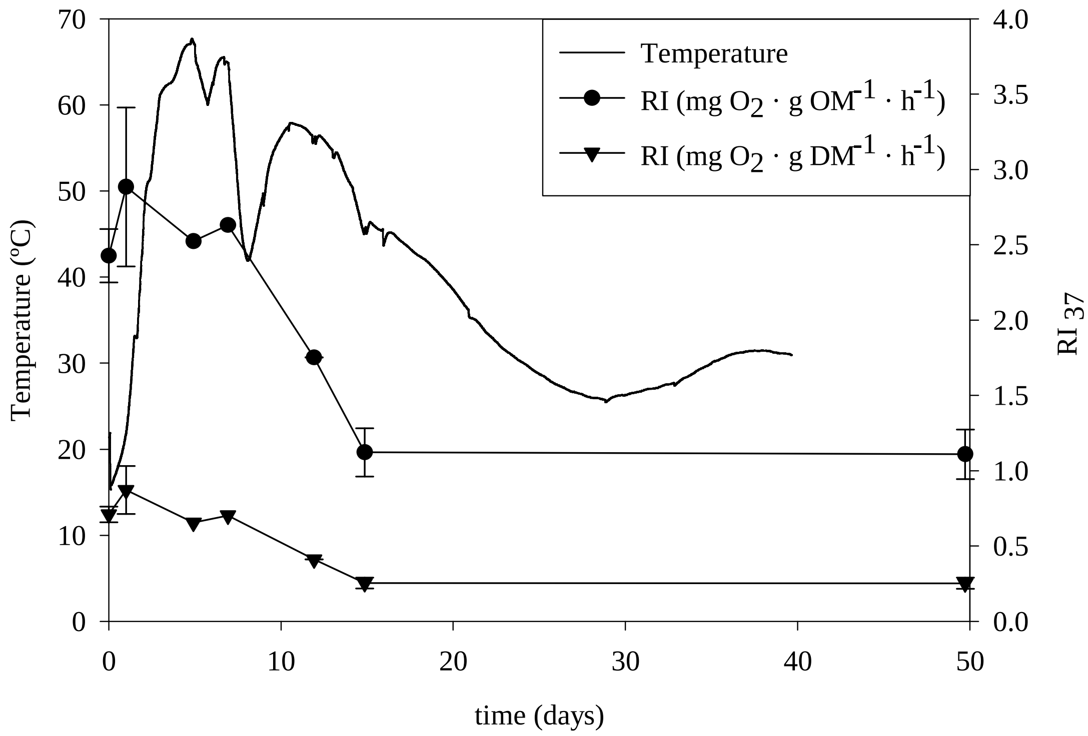
Figure 4. Average temperature profiles and respirometric indexes obtained at 37 ºC (RI_{37}) and at the *in situ* temperature (RI_T) for fresh sludge composting process.

Figure 5. Average temperature profiles and respirometric indexes obtained at 37 ºC (RI_{37}) and at the *in situ* temperature (RI_T) for anaerobically digested sludge composting process.

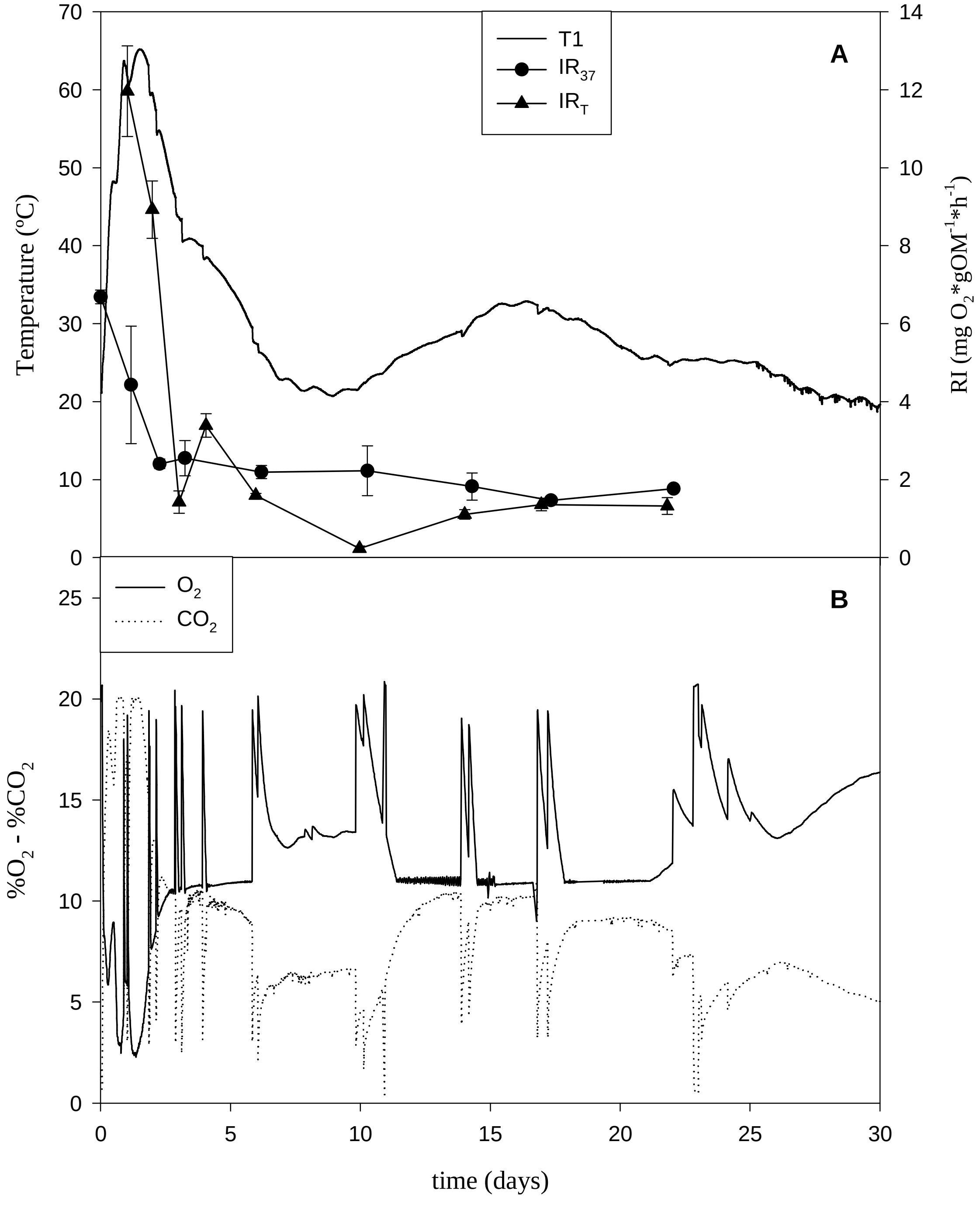

Caudal de aire
PC
Baño
agua
Sonda de oxígeno
T1
T2



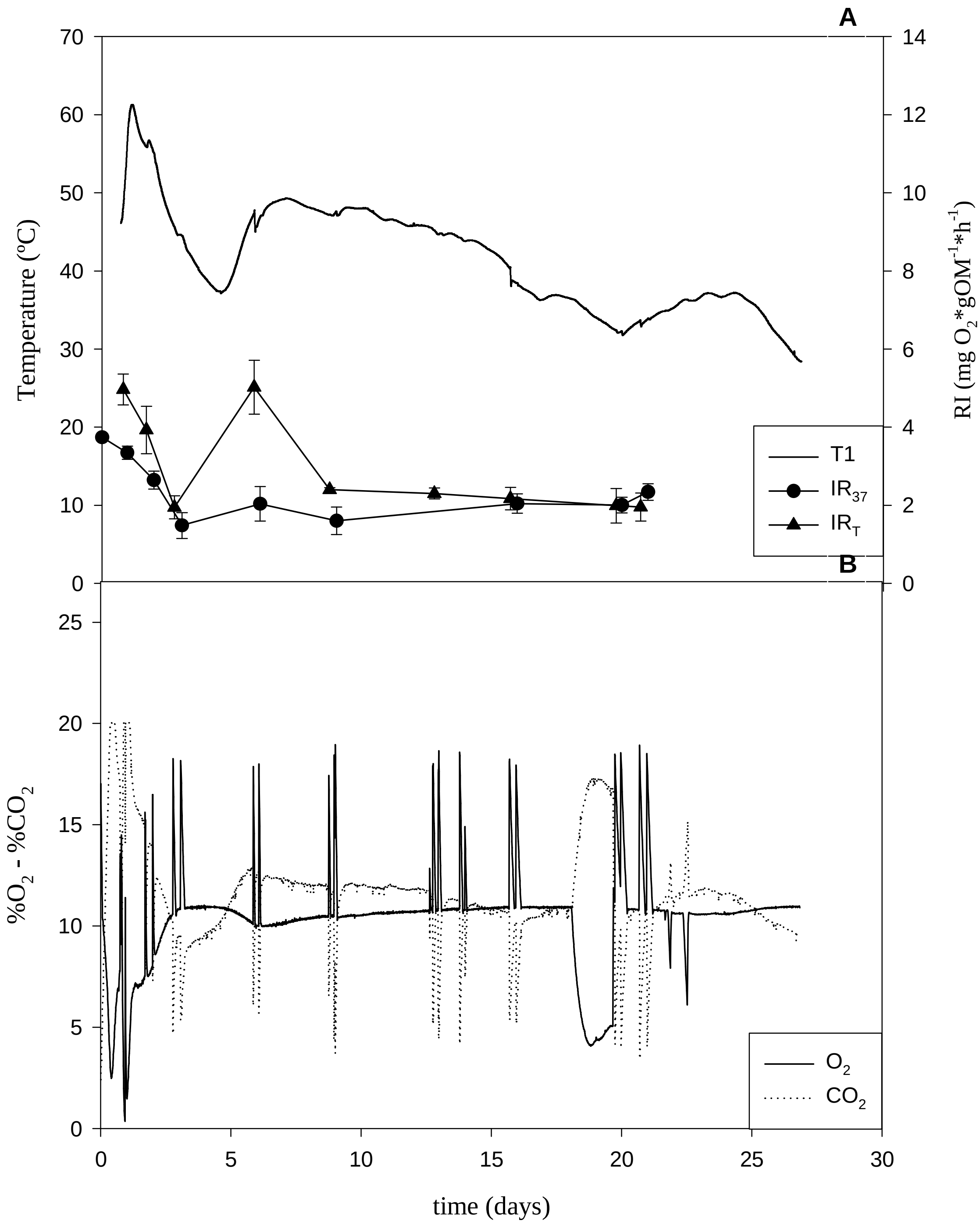
4th day of process
sample 1
IR37= 3.90 mgO2 * g-1MO * h-1
%Oxygen
Time (min)
A
4th day of process
sample 2
IR37= 3.94 mgO2 * g-1MO * h-1
%Oxygen
Time (min)
B
4th day of process
sample 3
IR37= 3.74 mgO2 * g-1MO * h-1
%Oxygen
Time (min)
C



Temperature
RI (mg O2 · g OM-1 · h-1)
RI (mg O2 · g DM-1 · h-1)
Temperature (ºC)
RI 37
time (days)
0
10
20
30
40
50
60
70
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
0
10
20
30
40
50



T1
IR37
IRT
A
Temperature (ºC)
RI (mg O2*gOM-1*h-1)
O2
CO2
B
%O2 - %CO2
time (days)


A
B
Temperature (ºC)
RI (mg O2*gOM-1*h-1)
T1
IR37
IRT
%O2 - %CO2
O2
CO2
time (days)
0
5
10
15
20
25
30