Parameters affecting the stability of the digestate from a two-stage anaerobic process treating the Organic Fraction of Municipal Solid Waste

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ABSTRACT

This paper focused on the factors affecting the respiration rate of the digestate taken from a continuous anaerobic two-stage process treating the Organic Fraction of Municipal Solid Waste (OFMSW). The process involved a hydrolytic reactor (HR) that produced a leachate fed to a Submerged Anaerobic Membrane Bioreactor (SAMBR). It was found that a Volatile Solids (VS) removal in the range 40-75% and an operating temperature in the HR between 21 and 35ºC resulted in digestates with similar respiration rates, with all digestates requiring 17 days of aeration before satisfying the British Standard Institution stability threshold of 16 mg CO_2.gVS^{-1}.day^{-1}. Sanitization of the digestate at 65ºC for 7 days allowed a mature digestate to be obtained. At 4 g $VS.L^{-1}.d^{-1}$ and Solid Retention Times (SRT) greater than 70 days, all the digestates emitted CO_2 at a rate lower than 25 mg CO_2 g $VS^{-1}.d^{-1}$ after 3 days of aeration, while at SRT lower than 20 days all the digestates displayed a respiration rate greater than 25 mg CO_2 g $VS^{-1}.d^{-1}$. The compliance criteria for Class I digestate set by the European Commission (EC) and British Standard Institution (BSI) could not be met because of nickel and chromium contamination, which was probably due to attrition of the stainless steel stirrer in the HR.

Keywords: Anaerobic Digestion; Organic Fraction of Municipal Solid Waste; digestate stability; respiration rate.

List of abbreviations

BSI -British Standard Institution

COD -Chemical Oxygen Demand ($mg.L^{-1}$)

dm -dry matter

EC -European Commission

GW -Garden Waste

HM -Heavy Metals

HR -Hydrolytic Reactor

HRT -Hydraulic Retention Time (days)

KW -Kitchen Waste

OFMSW -Organic Fraction of Municipal Solid Waste

OLR -Organic Loading Rate (g $VS.L^{-1}.day^{-1}$)

RMP -Residual Methane Potential (mL CH_4/g VS)

SAMBR -Submerged Anaerobic Membrane Bioreactor

SRT -Solid Retention Time (days)

VFAs -Volatile Fatty Acids ($mg.L^{-1}$)

VS -Volatile Solids

1. Introduction

Anaerobic digestion has been drawing increased interest in recent years as shown by the number of publications as well as the number of full scale plants in Europe. Besides producing an energy rich biogas, anaerobic digestion of the organic fraction of Municipal Solid Waste (OFMSW) also produces digestate than can be used as a fertilizer. Many studies on the anaerobic treatment of the OFMSW found VS removals were typically in the range 50-70% at organic loading rates in the range 4-8 kg $VS.m^{-3}.day^{-1}$ (Vandevivere et al., 2003). The solid and liquid retention time is generally 20-30 days. At lower temperatures, the VS removal can drop to 50 % (Trzcinski et al., 2010).

Mechanical-biological treatment of the OFMSW is now the main strategy to reduce biodegradable MSW in waste in the UK and in Europe (Scaglia and Adani, 2008). It consists of mechanical pre-treatment followed by an aerobic (composting-like process) or anaerobic process so that waste impacts are reduced. These processes have attracted attention because they produce stabilized waste that can be sold as fertilizer or disposed of in landfill, in which case it will have a low impact on the environment (Adani et al., 2004). As a result, the biological stability of digestate after biological processing has become of particular interest recently. Digestate can be defined as the material resulting from the anaerobic digestion of separately collected biowaste (European Commission, 2001). Digestate quality is related to the absence of phytotoxicity and weed seeds, and the presence of both organic and inorganic elements (Baffi et al., 2007).

Composting of organic wastes is a biooxidative process involving the mineralisation and partial humification of the organic matter, leading to a stable, sanitised and humus-like material rich in organic matter and free of offensive odours resulting from the composting process (European Commission, 2001). Furthermore, the final product must be free of phytotoxicity and pathogens and have certain humic properties (Zucconi and de Bertoldi, 1987). If the material contains mainly recalcitrant or humus-like matter, it is not able to sustain microbial activity, and therefore is considered stable. Stability prevents nutrients from being tied up in rapid microbial growth, allowing them to be available for plant needs. The control of parameters such as bulk density, porosity, particle size, nutrient content, C/N ratio, temperature, pH, moisture and oxygen supply have been demonstrated to be key for composting optimization since they determine the optimal conditions for microbial development and organic matter degradation (Bernal et al., 2009). The aerobic respiration rate was selected by many authors as the most suitable parameter to assess aerobic biological activity and hence digestate stability (Hue and Liu, 1995; Wallace et al., 2005). However,

carbon dioxide evolution is the most direct technique of digestate stability because it measures carbon derived directly from the digestate being tested. With respect to the CO_2 production rate the stability threshold established by the British Standards Institution (BSI) (WRAP, 2005) is 16 mg CO_2.g VS^{-1}.day^{-1}, whereas the one set by the European Commission (EC) is 1000 ± 200 mg O_2. kg VS^{-1}.hr^{-1} (European Commission, 2001), which is equivalent to 33 mg CO_2.g VS^{-1}.day^{-1}.

Although many papers discuss anaerobic bioreactor performance and optimisation of biogas production, relatively few papers have focused on the solid by-product. Thus the objective of this paper was to present results on the effect of various parameters on the performance of a continuous lab-scale anaerobic process and their consequences on the digestate respiration rate. The aim was to characterise the digestate in terms of residual methane potential and aerobic respiration rate, and determine the effect of parameters such as the solid retention time (SRT) and temperature of the digester, type of digester (first and second stage), digestate aeration time and incubation temperature, particle size and moisture on the aerobic respiration rate of the digestate. Another aim was to quantify the heavy metal content of a typical OFMSW digestate produced in an anaerobic process and determine the influence of the SRT.

2. Materials and methods

2.1. Feedstock

The simulated OFMSW mixture used in this study consisted of 41% Kitchen Waste (KW), 11% Garden Waste and 48% Paper Waste on a wet basis. The collection, storage and preparation of the OFMSW feedstock can be found elsewhere (Trzcinski and Stuckey, 2009a), while the properties of the feedstock can be found in Trzcinski and Stuckey (2009b). Briefly, the total solids (TS) content of the mixture of waste was adjusted with process liquid

to obtain the OFMSW feedstock at 10% TS. The organic content of the simulated OFMSW feedstock was in the range of 82-86% dry matter, and the COD/VS ratio was found to be 1.2-1.6 g COD.g VS^{-1}.

2.2. Two-stage anaerobic process

The HR (working volume of 10 L) was an acrylic cylinder with a concentric stainless steel mesh inside the cylinder, and was mixed intermittently (15 min ON-15 min OFF). The stainless steel mesh was used to retain the large partially hydrolyzed particles, and thereby separate the coarse solids from the leachate being fed to the SAMBR, or in other words uncouple the SRT and the HRT. The SAMBR was a three litre acrylic plastic reactor with a submerged Kubota polyethylene flat sheet membrane (0.1 m^2 surface area - pore size of 0.4 micron), and was maintained at 35ºC. The SAMBR contained a standing baffle designed to direct the fluid to the upcomer and downcomer regimes. The biomass was continuously mixed using headspace biogas that was pumped through a stainless steel tube diffuser to generate coarse bubbles. The bubbles pushed the sludge flow upward between the membrane module and the reactor wall in the upper section. Details of the HR and SAMBR can be found elsewhere (Trzcinski and Stuckey, 2009a). The HR and SAMBR were connected in series: the leachate was fed continuously to the SAMBR, and the SAMBR permeate was recycled to the HR in order to maintain the moisture and alkalinity of the system, and reduce the water usage (Figure 1).

2.2.1 Effect of HR temperature and SRT on digestate respiration rate

For this experiment, the HR was fed daily with the OFMSW feedstock at a constant organic loading rate (OLR) of 4 g $VS.L^{-1}.day^{-1}$. The digestate was withdrawn daily from the HR and manually dewatered by hand compression through a 500 micron sieve to recover the

remaining process liquid so that the use of fresh water was minimized. The digestate then went through the steps described in Figure 1. In order to investigate the effect of HR temperature on the digestate respiration rate, the HR was operated at 35°C until day 100, and then at 21°C until the end of the experiment.

2.2.2 Effect of OLR and SRT on digestate respiration rate

For this experiment, the HR was maintained at 35ºC throughout the test, while the OLR was increased stepwise to investigate its influence on the aerobic respiration rate. In order to avoid the build up of solids in the reactor the SRT was consequently reduced. Each condition studied lasted for about 20 days, and the residual methane potential (RMP) of the digestate and its heavy metal (HM) content were measured for each condition. The aim was to provide additional information regarding the influence of process operation on digestate quality.

2.3. Aerobic respiration rate

In order to obtain similar and reproducible moistures, the digestates were dried in a fume-cupboard for 1-2 days, and then wetted with tap water so that the moisture content was close to 60%. About 20 g of moistened digestate was kept in an unsealed polyethylene plastic bag and equilibrated for three days at 30°C in order to stimulate the growth of aerobic microbes. The bags were shaken every day to enhance the transfer of oxygen through the pores of the digestate, and ensure that oxygen was not the limiting factor for microbial equilibration. The adjustment of moisture to 60% and the equilibration period of 3 days prior to starting the measurement of evolved carbon dioxide were recommended in several publications (Leege and Thompson, 1997; Llewelyn, 2005; Switzenbaum et al., 2002; Wallace et al., 2005). After three days of equilibration, a known mass of digestate was inserted into a brown bottle, a rubber sleeve containing two pellets of NaOH was placed in the neck of the bottle, and

finally the Oxitop measuring head was screwed on the bottle to keep it air-tight (Figure 1). When biomass is degrading organic matter it consumes oxygen from the headspace and produces carbon dioxide that is precipitated with the pellets of NaOH to form Na_2CO_3. As a result, a vacuum is created in the bottle which is proportional to the oxygen consumed by the bacteria. The OxiTop measuring head measures and stores this pressure for 4 days once the measurement has started. The formula shown below was used with the values from the OxiTop measuring heads (GmbH, 2006):

$$mg\,O_2 / L = \frac{MM_{O_2}}{R \cdot T_m} \cdot \left(\frac{V_t - V}{V} + \alpha \cdot \frac{T_m}{T_0} \right) \cdot \Delta p(O_2)$$

Where MM_{O_2} is the molecular weight of oxygen (32,000 mg.mol^{-1}), R is the gas constant (83.14 L.mbar.mol^{-1}.K^{-1}), T_0 is the reference temperature (273.15 K), T_m is the measuring temperature, V_t is the bottle volume (mL), V is the sample volume (mL), α is the Bunsen oxygen absorption coefficient at T_m and $\Delta p(O_2)$ is the difference of the oxygen partial pressure (mbar). The values were then converted from mg of oxygen to mg of carbon dioxide by multiplying by 1.375 (44/32) considering that one mole of oxygen is converted to one mole of carbon dioxide during the degradation of one mole of carbon:

$$C + O_2 \rightarrow CO_2 + Energy$$

The moisture and volatile solids (VS) content of the digestate was determined for each sample according to Standards Methods (APHA, 1999). Carbon dioxide evolution was monitored over 4 days (Leege and Thompson, 1997; Wallace et al., 2005), and the rate of evolution was calculated as the ratio of total CO_2 evolved (mg) per mass of volatile solids incubated divided by 4 days. Each sample was run in triplicate and the standard deviation was in the range 0.1 to 1.2 mg CO_2.g VS^{-1}.day^{-1}.

2.4. Analytical and statistical methods

The heavy metal (HM) content of the digestate was determined according to Standard Methods (APHA, 1999) after digestion with *Aqua Regia*. The ultimate anaerobic biodegradability of the feedstock and the residual methane potential of the digestate were analyzed by Owen et al.'*s* bioassay method (1979). Each test was carried out in triplicate. Volatile Fatty Acids (VFAs: acetic, propionic, iso- and n-butyric, iso- and n-valeric and n-caproic acids) were measured using a Shimadzu Gas Chromatograph with a flame-ionized detector and a SGE capillary column (12mx0.53mm ID-BP21 0.5µm). The mobile phase was helium, and the injector, column and detector temperature were 200, 80 and 250°C, respectively. The coefficient of variation was 3% for ten identical samples. The heavy metal analysis, methane potential, VFAs analyses were performed in triplicate and the statistical analysis was completed using Microsoft Excel 2007.

3. Results and discussion

3.1. Effect of HR temperature and SRT on digestate respiration rate

Throughout the run, the digestate taken at regular intervals from the HR was assessed in terms of respirometric activity, and the relevant process parameters are summarized in Table 1. It can be seen from Figure 2 that almost all the samples released considerably more CO_2 after 3 days of aeration than the 16 mg CO_2 g $VS^{-1}.d^{-1}$ recommended for a stable digestate (horizontal line) defined by the British Standard BSI PAS 100:2005 (WRAP, 2005). However, the first two samples taken during the start up of the HR were below the limit, but this is thought to be due to the large mass of digestate initially incubated. This can result in stratification where the zones at the bottom are not aerated, while the top layer has good access to oxygen in the headspace. For the subsequent samples, masses of only 2 to 5 g were incubated in the bottles.

The results also show that the operating temperature and the SRT in the HR had no effect on the respiration rate of the digestate (95% confidence level), although it is clear from Table 1 that higher SRT and temperature resulted in higher methane yield and VS removals in the HR. Therefore, the results indicated that lower VS removal in the HR did not correlate with greater respiration rates (R^2 = 0.2). Nonetheless, the respirometric test shows that the digestate taken from the HR operating at 4 g $VS.L^{-1}.d^{-1}$ with 75% VS degradation cannot be used as a fertilizer straight away, and must undergo an aerobic stabilization stage. Depending on the amount of the samples, the identical respirometric test was repeated with the same digestate in the plastic bags but after longer period of aeration, and Figure 2 shows that after 10 days of aeration (open squares) the samples tested (days 80, 85, 92, 99 and 106) were either below or very close to the limit, meaning that for some samples an aeration period of 10 days was not yet sufficient for proper digestate stability.

On the other hand, after 17 days of aeration (star symbol in Figure 1) all the samples tested could be considered as stable according to the BSI definition (95% level). All the samples could not be tested for all the aeration periods considered (3, 7, 10, 17, 25, 30 and 39 days) due to a lack of both sample and Oxitop bottles to carry out all the respiration tests simultaneously. Longer aeration times decreased the respiration rate but at a smaller rate compared to the first two weeks of aeration, which is line with the observations of other researchers (Wallace et al., 2005). This is well illustrated by the digestate withdrawn from the HR on day 80 which displayed a respiration rate of 20 mg CO_2 g $VS^{-1}.d^{-1}$ after 3 days of aeration, but it dropped to half that value after 10 days of aeration. From 17 to 25 and 25 to 39 days of aeration the respiration rate decreased further to 7 and then to 5 mg CO_2 g $VS^{-1}.d^{-1}$. These results also show that even when a high VS removal is obtained in the anaerobic HR, there is still readily degradable substrate available in the digestate for aerobic microbial

growth. After 39 days of aeration, there was considerably less available carbon and the microbial population entered a starvation period.

Abdullahi et al. (2008) also observed that increasing the aerobic post-treatment period can decrease the amount of easily biodegradable components of the waste, and consequently decrease the phytotoxic effects of the resulting soil amendment. The stabilization period of 17 days obtained in this study is similar to the 3 weeks retention time required to stabilise MSW in a full-scale mechanical-biological treatment plant (Adani et al., 2004). Tambone et al. (2009) reported an oxygen uptake rate of 30 mg O_2.g TS^{-1}.20 hr^{-1} (equivalent to 72 mg CO_2.g $VS^{-1}.d^{-1}$) on the digestate from a 30 days HRT anaerobic digester treating cow manure, agro-industrial waste and OFMSW followed by a post-treatment stage of 50 days residence time. However, they used a solid sample resuspended in water for the test which considerably reduces oxygen transfer compared to our solid static method, and this could explain their high respiration rates after such long residence times in their process.

The effect of the SRT was also investigated in the SAMBR because it is important to remove excess sludge in membrane bioreactors to keep a workable flux. It is therefore interesting to compare the stability of the solids withdrawn from the HR and the SAMBR that contained anaerobic bacteria as well as recalcitrant lignocellulosic fibres from the HR. On day 120 of the run it was found that the respiration rates were 16.6 (± 1.9) and 8.2 (± 1.1) mg CO_2.g $VS^{-1}.day^{-1}$ for the digestate from the HR and the SAMBR, respectively. This respirometric test showed that the solids taken from the SAMBR were more degraded than in the HR, leaving less biodegradable material available during the aeration process, and resulting in a significantly (95%) lower CO_2 production, (about 50% less). Furthermore, the initial rate of CO_2 production was slower in the case of the digestate from the SAMBR (data not shown), suggesting that the remaining organics were more recalcitrant and did not support microbial activity as much as on the digestate from the HR that contained more readily available

substrates such as fatty acids. Indeed, the VFA concentration (as chemical oxygen demand) was 3700 mg/L and 80 mg/L in the HR and the SAMBR, respectively. Hence, from these results the digestate from the second stage of our two-stage anaerobic process could be considered as completely stabilised and therefore does not require any further aeration.

3.1.1. Effect of incubation temperature on aerobic respiration rate The effect of the incubation temperature on the digestate respiration rate was investigated. The digestate withdrawn from the HR on day 115 was incubated for 7 days at 65ºC and a control was run at the normal temperature of 30ºC. Sixty-five is the minimum temperature recommended for the sanitisation of composting materials. The sanitisation step serves as the control for human, animal and plant pathogens. The BSI PAS100:2005 document (WRAP, 2005) provides recommendations for how to achieve sanitisation. In addition to a minimum of 65ºC for 7 days, the digestate must also be kept at more than 50% moisture content and the digestate must be mixed/turned at least twice to ensure that the entire batch has been exposed to sanitising conditions.

Table 2 lists the respiration rates of the digestate on day 115 with and without sanitisation, and it can be seen that the sanitisation step considerably decreased the respiration rate. Moreover, after one week of sanitisation the digestate was aerated at 30ºC along with the control kept for 2 weeks at 30ºC in order to determine whether microorganisms could re-colonise the sanitised digestate. It was found that after 1 week aeration at 30ºC the microbial activity of the sanitised digestate was still lower than the control, and moreover, no fungal growth was observed (which was normally the case in non-sanitised samples). In fact, the values obtained corresponded to very mature digestate according to the maturity index established by several authors (Hue and Liu, 1995; Michel et al., 2004; Wang et al., 2004). This indicates that the digestate produced in our HR was a suitable digestate after the

sanitisation step which is required by both the EC (European Commission, 2001) and the BSI (WRAP, 2005).

The last two rows of Table 2 contain the respiration rates of the digestate from day 132 incubated at room temperature and at 30ºC. Based on these results, it cannot be concluded that the respiration rates are significantly different if the digestate is aerated at 20 or 30ºC (at 95%).

3.1.2. Effect of Particle Size

The literature revealed that respirometric tests are usually carried out with digestate with particle sizes in the range 4-15 mm (Hue and Liu, 1995; Popp and Fischer, 1998), and in one research report, the samples were just passed through a 20 mm sieve (WRAP, 2005). In this section the effect of the particle size of the digestate on the respirometric activity was determined. The digestate withdrawn from the HR on day 137 was fractionated according to the particle size of the digestate: less than 0.7 cm, between 0.7 and 1.1 cm and bigger than 1.1 cm. The latter fraction was, however, limited by the diameter of the bottle neck which was 1.5 cm. Each fraction was equilibrated for 3 days with the identical moisture content. It was found that the particle sizes tested did not influence the respiration rate in a significant way (95% confidence). The respiration rates were 24 (±0.9), 19.8 (±1.2) and 22.3 (±1.8) mg CO_2.g VS^{-1}.d^{-1} for the fraction greater than 1.1 cm, the one between 0.7 and 1.1 cm and the one below 0.7 cm, respectively. The initial production rate was similar for the 3 fractions suggesting that there was no lag phase in the CO_2 production or mass transfer limitations in the large particles.

3.2. Effect of OLR and SRT on process performance

The methods used in this experiment are described in section 2.2.2. A VS removal of 71% was achieved during the first 25 days at an OLR of 4 g $VS.L^{-1}.d^{-1}$ and a SRT of 160-180 days (Table 3). Similar VS removals were obtained between days 26 and 46 at the same OLR, but at a SRT of 70-95 days and a HRT of 9-12 days. These VS removals of 70-75% were similar to those in section 3.1 in which the SRT was 66 days and the HRT 20 days. The extent of hydrolysis was considerably reduced at 20 days SRT compared to 70 in the HR which led to a low VS removal of 39%. This low VS removal was attributed to the reduced amount of time given to the substrate to be degraded in the HR. On day 64, the SRT in the HR was further decreased to 6-7 days while the HRT was kept at 4.5 days, but this highlighted even more the importance of SRT on VS removal as the latter dropped further to 26%. Hence, this experiment demonstrated that the SRT cannot be lowered to 20 days as this results in unacceptably low VS removals. An OLR of 10 g $VS.L^{-1}.d^{-1}$ was too high for the HR because the lignocellulosic waste could not be solubilised fast enough. Finally, from day 87 the HR was fed daily with Kitchen Waste (460 g wet $KW.d^{-1}$) and garden waste (18.6 g wet $GW.d^{-1}$), but in such an amount that the OLR was kept constant at 10 g $VS.L^{-1}.d^{-1}$. A SRT of 23 days with an HRT of 1.8 were sufficient to reach 81% VS destruction (Table 3).

3.2.1. Effect of the OLR and SRT on Aerobic Respiration Rate

The digestate taken from the HR was assessed in terms of stability. Figure 3 shows that at 4 g $VS.L^{-1}.d^{-1}$ and a SRT greater than 70 days, all the digestate emitted CO_2 at a rate lower than 25 mg CO_2 g $VS^{-1}.d^{-1}$ after 3 days of aeration. Some digestates were even below the red horizontal line showing the stability threshold of 16 mg CO_2 g $VS^{-1}.d^{-1}$ (WRAP, 2005). In contrast, when the SRT was less than 20 days all the digestates resulted in a respiration rate significantly greater than those obtained at SRTs greater than 70 days (95% confidence). The digestate obtained at SRTs lower than 20 days emitted more than 25 mg CO_2 g $VS^{-1}.d^{-1}$ after

three days of aeration, and more than 30 mg CO_2 g $VS^{-1}.d^{-1}$ when the SRT dropped to 6-7 days. Furthermore, after 3 weeks of aeration all the samples were below the limit of stability regardless of the VS removal in the HR, showing that after 3 weeks of aeration, the digestate will be below the limit even when the VS removal was low in the HR.

Eventually, after day 86 the HR was fed at 10 g $VS.L^{-1}.d^{-1}$ of mainly kitchen waste and the consequences on the digestate were quite surprising. Although the HR achieved more than 80% VS removal in 23 days retention time, which is normal for this type of waste, the digestate was significantly more active in terms of respiration rate. The respiration rates after 3 weeks (triangles in Figure 3) showed very high microbial activity, with values in the range 40-60 mg CO_2 g $VS^{-1}.d^{-1}$. Then after 5 weeks of aeration they decreased to values between 16 and 25 mg CO_2 g $VS^{-1}.d^{-1}$, and thus were still not considered as stable yet. This suggests that there might have been easily degradable substrate remaining after 23 days SRT in the HR, or that some of the feedstock was short-circuiting and appearing in the digestate after less than 23 days retention time. Since the digestate was dewatered to approximately 20% solids, it is likely that the high activity was due to the remaining leachate in the digestate which contained concentrations of total VFAs in the range 7000-9000 mg/L (as COD) after day 90. The impact of the remaining leachate in the digestate was not significant when the substrate was the OFMSW, but it became important when the feedstock was changed to kitchen waste due to the high amount of fatty acids produced. Drennan and DiStefano (2010) have treated food waste and landscape waste in an anaerobic digester operating at 175 days SRT and constant OLR of 2 g $COD.L^{-1}.d^{-1}$ followed by a curing aerobic reactor. These authors observed a decrease in the oxygen uptake rate from 0.91 to 0.37 mg O_2.g $VS^{-1}.hr^{-1}$ (equivalent to 30 and 12 mg CO_2.g $VS^{-1}.d^{-1}$, respectively) within 3 weeks of curing probably because of the very long SRT and low OLR used in their study which resulted in lower VFAs levels (700-900 mg acetic $acid.L^{-1}$) and shorter stabilisation period. This indicates that

aeration periods longer than 3 weeks will be required to reduce the respiration rate of a digestate when food waste are fed at high loading rates and short SRTs, as was the case in our study.

Besides the aerobic respiration rate, the residual methane potential of the digestate is an important factor because emissions of methane and odours are not desirable for a stable digestate. The emission of methane also relates to the presence of easily available substrate such as VFAs that can cause phytotoxicity, and therefore indicates an unstable digestate. The residual anaerobic biodegradability of the digestate withdrawn from the HR is included in Table 3, and it is clear that longer SRTs led to lower residual biodegradabilities. Tambone et al. (2009) reported a residual biogas production of 0.113 L biogas.VS^{-1} after their anaerobic process (30-40 d HRT) followed by a 50 days retention time post-treatment. In our process, we also obtained residual biogas potentials in the range 0.1-0.16 at OLR of 4 g $VS.L^{-1}.d^{-1}$ and SRTs longer than 70 days. As the SRT decreased to 20 days, the residual biogas potential crossed the threshold of 0.25 L.g VS^{-1} set by the BSI (WRAP, 2010). Interestingly, if the RMP obtained from the digestate is compared with the ultimate biodegradability of the feedstock (=244 ml CH_4.g VS^{-1}), the percentage that was biodegraded and which hence does not appear in the RMP of the digestate, matches relatively well with the VS removal obtained by a VS balance on the HR.

The very high RMP obtained from the digestate at the end of the run with kitchen waste as the main substrate suggests that the digestate was contaminated with leachate high in VFAs. The VS removal based on the RMP of the digestate (last row and last column of Table 3) could not be calculated because the digestate RMP was greater than the ultimate biodegradability of KW (=357 ml CH_4.g VS^{-1}). This is clearly impossible and strengthens

the hypothesis laid out in Section 3.2.1 that the digestate taken during that period was contaminated with fresh substrate (short-circuiting) and possibly leachate containing VFAs.

3.2.2. Effect of Moisture Content on Digestate Activity

The method used in this study relies on the fact that the maximum activity of the digestate is expressed at moisture contents between 40 and 60%. However, during the course of the respirometric test, some water evaporates in the bottle kept at 30ºC. In order to investigate the extent of the evaporation during the test, the moisture content of the digestate was measured before and after the respirometric test. The moisture content was 60.7 and 45.4%, respectively, before and after the test, which shows that a 15% loss in moisture occurred during the 4 day test. However, even at the end of the test the moisture content remained in the optimum range. Hence, it can be stated that the moisture content will remain valid through the test inasmuch as the initial moisture is relatively close to the upper value of the range.

In order to gain more insight into the influence of moisture content, additional tests were carried out using the same digestate, but with different moisture contents. Two different samples were used, namely the digestates from the HR on days 74 and 85. The carbon dioxide emission rate is shown in Figure 4, and it can be seen that moisture content in the range 57-73 % gave similar results (95% confidence). In contrast, when the moisture content was less than 40 %, the microbial activity was significantly lower. This is due to the sample being too dry, and some zones of the samples might not be wetted at all resulting in less available carbon for growth and metabolic activity. Digestate samples that have moisture content below 40% may be biologically dormant.

3.3. Effect of the SRT on the Heavy Metal (HM) content of the Digestate

Although several studies have shown that the use of stabilized OFMSW residues from a variety of processes (primarily composting) in agriculture has many benefits to soil, crops and the environment (Prabpai et al., 2009), concerns still persist about their content of potentially hazardous heavy metals (Gajalakshmi and Abbasi, 2008). If excessive loads of pollutants are introduced with the application of low-quality digestate, soil fertility may be adversely affected, ground-water quality threatened, and the food chain contaminated (McBridge, 1995).

Table 4 contains the HM content of the digestate taken from the HR over the same periods considered in Table 3. We expected to find significantly greater HM concentrations at 10 g $VS.L^{-1}.d^{-1}$ than at 4 g $VS.L^{-1}.d^{-1}$ because of greater amounts of OFMSW fed, but this was not observed due to the lower VS degradations at 10 g $VS.L^{-1}.d^{-1}$. It was even found that the Zinc and Copper content decreased as the SRT decreased and as the OLR increased (Table 4). The lower concentration is likely to be the result of dilution caused by the greater fresh water consumption at lower SRT to keep the working volume constant. It was observed that the Zn, Pb, Cd and Cu content were below the compliance criteria of the *Class I* compost that can be used without any specific restriction (European Commission, 2001). However, the Ni content was equal or above 50 mg.kg dm(dry matter)$^{-1}$ in 4 out of 5 samples tested which means that it should be classified as a digestate of *Class II* that can be used in quantity not exceeding 30 tons dm/ha on a three-year average (European Commission, 2001). However, since the Chromium content was above 100 mg.kg dm^{-1} in 4 out of 5 samples tested, the digestates obtained in this study should really be defined as a "*Biowaste*", and should only be used as a component of an artificial soil or in those land applications that are not destined for food or fodder crop production (such as final landfill cover, landscape restoration in old and disused mines, anti-noise barriers, road construction, golf course, etc.) (European Commission, 2001). The criteria given by the BSI are the minimum qualities that

the compost must fulfil for general use such as soil improver, mulch, growing medium, turf dressing, topsoil, etc. Given the values of Nickel and Chromium in this study, the digestate cannot be used as compost as defined by the BSI.

Nonetheless, it is believed that the high Ni and Cr contents are due to the paddle stirrer that broke down during this study. The paddle was made of stainless steel which contains nickel and chromium, and the thickness of the rod was seen to decrease over time. It is believed that some chromium and nickel leached from the stainless steel rod due to continuous attrition, and hence appeared in the digestate. Moreover, a comparison with the HM content of the feedstock reported previously (Trzcinski and Stuckey, 2009b) confirmed that such high values of Ni and Cr in the digestate were impossible given the initial content of the feedstock that was found to be circa 4 mg.kg dm^{-1} for both Nickel and Chromium. This strengthens the hypothesis of an external source of these two elements. The Zn content was found to decrease tenfold compared to the feedstock values, whereas the Cu content remained similar, i.e. circa 50 mg.kg dm^{-1}. In contrast, the Cd content dropped from 2 mg.kg dm^{-1} in the feedstock to 0 in the digestate probably due to a complete precipitation with sulphide ions in the HR.

4. Conclusions

The HR could treat the OFMSW at an OLR of 4 g $VS.L^{-1}.d^{-1}$ with a SRT of 60-70 days and a HRT of 20 days resulting in a VS removal of 75% at 35ºC. At 21ºC, the VS removal dropped to 50%. The aerobic respiration rate of the digestate was not influenced by the VS removal or the temperature in the HR as similar respiration rates between 15 and 30 mg CO_2. g $VS^{-1}.d^{-1}$ were obtained at 35 and 21ºC. In contrast, it was found that the SRT and OLR had a significant effect on the aerobic respiration rate. At 4 g $VS.L^{-1}.d^{-1}$ and SRT greater than 70 days, all the digestates emitted CO_2 at a rate lower than 25 mg CO_2 g $VS^{-1}.d^{-1}$ after 3 days of

aeration. At SRTs lower than 20 days and higher OLR all the digestates displayed a respiration rate greater than 25 mg CO_2 g $VS^{-1}.d^{-1}$. After 3 weeks of aeration all the samples were below the limit of stability, except when the substrate was kitchen waste fed at high OLR which required more time to stabilize. The particle size of the digestate in the range 0.7–1.5 cm was found to have no significant effect on the respirometric activity, while the moisture content of the digestate in the range 57-73% was found to have no significant impact of the respiration rate; below 40% the digestate was biologically dormant, and as a result, displayed a very low respirometric activity. Finally, the heavy metal content of the simulated OFMSW feedstock was determined and results showed that it decreased as the SRT decreased, and as the OLR increased, due to greater fresh water consumption. The digestate did not meet the compliance criteria set by the EC for Class I digestate and the BSI because of Nickel and Chromium contamination which was probably due to attrition of the stainless steel stirrer.

Acknowledgements

This research was supported by a grant from the Department of Environment, Food and Rural Affairs (DEFRA) in the UK.

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Table 4. Heavy metal content of the digestate from the HR and comparison with the compliance criteria for compost and stabilised biowaste established by the European Commission (EC) and the British Standard Institution (BSI). (standard deviation).